Southern California Bight
2003 Regional Marine Monitoring Survey
(Bight'03)

Quality Assurance Manual

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June 2003

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I. INTRODUCTION

The Southern California Bight (SCB; Figure 1-1), 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 1990a). 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 that is expected to increase another 20% by 2010 (NRC 1990b). 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 $31M to monitor the environmental quality of natural resources in the SCB (Schiff et al 2002). 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 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.

The 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.

The 1998 Survey

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

The inclusion of new participants provided several benefits. Cooperative interactions among many organizations with different perspectives and interests, including a combination of regulators and dischargers, ensured that the most appropriate regional questions were being addressed in the study. The additional resources brought by new participants also expanded the number of habitats and indicators that were sampled. Sampling for Bight'98 included all of the areas sampled in 1994, plus a new focus on nearshore habitats (bays, harbors and beaches)

and offshore islands. Bight'98 also coordinated with a Mexican program to characterize the condition of SCB coastal waters south of the US border. The new indicators that were measured included shoreline microbiology, biomarkers and new chemical measures.

The 2003 Survey

The 2003 Southern California Bight Regional Monitoring Project (Bight'03) represents the milestone of creating a periodic, but ongoing regional monitoring program in the SCB. Regional monitoring is recognized as an important component of understanding and managing our coastal resources (SCCWRP 2002). As such, regional assessments conducted every five years, are now written into NPDES permits for many SCB dischargers. The Bight'03 program not only provides the large-scale assessments necessary for both the regulated and regulatory community, but also provides a platform for exploring new questions, testing new technologies, and providing further standardization and improvements in overall monitoring quality.

Like its predecessors, Bight'03will involve nearly 60 organizations (Table I-1), many of them new to the regional monitoring program. Bight'03 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.

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

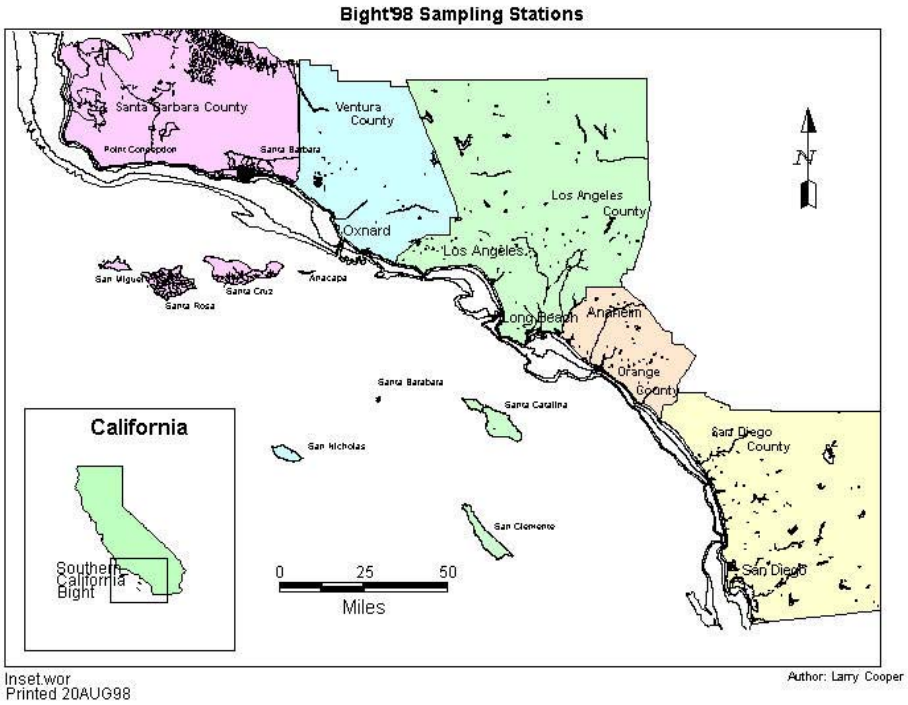

TABLE I-1. Participants in the Bight'03 Regional Monitoring Program. Participants in the coastal ecology component are asterisked.

AES Corporation*
AMEC Incorporated
Aquatic Bioassay and Consulting Laboratories (ABCL)*
Channel Islands National Marine Sanctuary (CINMS)*
Chevron USA Products Company*
City of Long Beach
City of Los Angeles Environmental Monitoring Division (CLAEMD)*
City of Oceanside*
City of Oxnard*
City of San Diego*
City of Santa Barbara
City of Ventura
Encina Wastewater Authority*
Granite Canyon Marine Pollution Studies Lab*
Houston Industries, Inc.*
Instituto de Investigacione, Oceanologicas (UABC)
Jet Propulsion Laboratory
Los Angeles County Dept. of Beaches & Harbors*
Los Angeles County Dept. of Health Services
Los Angeles County Dept. of Public Works*
Los Angeles Regional Water Quality Control Board*
Los Angeles County Sanitation Districts (LACSD)*
Marine Biological Consultants
Marine Ecological Consultants
Marine Corps Base - Camp Pendleton
Minerals Management Service
NES Energy, Inc.*
NRG Energy, Inc.*
Occidental College*
Orange County Environmental Health Division
Orange County Public Facilities and Resources (OCPFRD)*
Orange County Sanitation District (OCSD)*
Port of Los Angeles
Port of Los Angeles*
Port of San Diego*
Reliant Corporation*
San Diego County Dept. of Environmental Health
San Diego Regional Water Quality Control Board (SDRWQCB)*
San Elijo Joint Powers Authority*
Santa Ana Regional Water Quality Control Board*
Santa Barbara Health Care Services

TABLE I-1 (continued). Participants in the Bight'03 Regional Monitoring Program. Participants in the coastal ecology component are asterisked.

Santa Monica Baykeeper
South Orange County Water Authority (SOCWA)*
Southern California Coastal Water Research Project (SCCWRP)*
State Water Resources Control Board (SWRCB)*
Surfrider Foundation
University of California, Irvine
University of California, Riverside*
University of California, San Diego
University of California, Santa Barbara
US EPA Region IX*
US EPA Office of Research and Development*
US Fish and Wildlife Service
US Geological Survey*
Ventura County Watershed Protection Division*

II. QUALITY ASSURANCE OBJECTIVES

A. Overview

The primary goal of the QA/QC plan is to ensure that the data generated in the Bight'03 survey are comparable among particpants. Many different organizations will be participating in the collection and analysis of samples in Bight'03; 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 Coastal Ecology monitoring program components (e.g., field operations, water quality, water, sediment and tissue chemical analyses, benthic analyses, demersal and pelagic fish analyses) have been established by the Bight'03 Steering Committee.

B. General Approach To Quality Assurance

The QA program for Bight'03 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) field collection, processing, and laboratory analytical techniques will be applied consistently and correctly; 2) the number of lost, damaged, and uncollected samples will be minimized; 3) the integrity of the data will be maintained and documented from sample collection to entry into the data record; 4) all data will be comparable; and 5) results can be reproduced.

Quality control (QC) activities are implemented during the data collection phase of the 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'03 have well established monitoring programs. QA activities for Bight'03 have focused on developing a common field manual and documenting the comparability of laboratory methods. Training of field and laboratory personnel is focused on communicating goals and objectives of the survey, as well any modifications in methods or procedures that have been made to ensure data comparability. The

purpose of this training is to verify that all participants will be able to implement the agreed upon procedures in a consistent manner with comparable proficiency. Quantitative measures 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 because of the large number of particpants. 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'03 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- and/or laboratory-specific bias and that the degree of random error is consistent across data sets. Accuracy and precision goals for indicators to be measured during the Bight'03 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.

TABLE 2-1. Measurement Quality Objectives for Bight'03 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%
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%
gross pathology	5%	NA	90%
Contaminants in fish	30%	30%	90%

An MQO for completeness was also defined for Bight'03. 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'03 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'03 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 new Field Operations Manual (2003) has been prepared to standardize data collection efforts in the field. Each participating organization collecting samples in the field has identified a single point of contact for field operations (referred to as the Lead Scientist in the field operations manual).

A single laboratory manual was not developed for the 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 this Quality Assurance Plan (QAP) which identifies performance based standards and the appropriate level of QA/QC. Procedures for benthic analyses appropriate to the Bight'03 Project are detailed in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual (2003)

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'03 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 Bight'03 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'03 survey,
- A series of presurvey methods and taxonomy protocol intercalibration meetings/exercises to ensure that project participants understand the requirements outlined in the field manual, and
- A presurvey audit of new participants, or participants who have experienced a significant turnover in personnel since Bight'98, to demonstrate understanding and capability.

Quality Control measures for the field collection effort include:

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

Field operations manual

Standard field procedures are documented in the Bight'03 Field Operations Manual (2003). 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.

Lead Scientists and Boat Captains will be instructed on the field procedures to be followed during the survey and they, in turn, will instruct their field personnel on the proper

procedures for the survey. The lead scientist of each organization is responsible for distributing the Bight'03 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'03 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.

Lead scientists and boat captains of all organizations participating in the survey will be required to attend a protocol calibration meeting, which will be conducted several weeks before the survey. The goals and objectives of the Bight'03 will be discussed at this meeting as well as the responsibilities of the chief scientist and boat captains during the Bight'03 survey. Each participating organization will be provided with a Workplan, Field Operations Manual and QA/QC Document for Bight'03 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 Lead Scientist of each organization will train their field personnel, as needed, on the field operations to be conducted during the survey. It will be the responsibility of the Lead Scientist of each organization to review the Workplan and Field Operations Manual with their field crews and to ensure that they understand the field procedures and specific field QA/QC requirements that must be followed during the survey. It is also the Lead Scientist's responsibility to train their field crews, as needed, on operations to be performed. Personnel that cannot perform an operation as required by the 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 Auditor prior to sampling for the Bight'03 study. These pre-survey 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 Bight'98 Survey or who have a significant number of staff members that did not participate in the Bight'98 Survey. If resources and time are still available after all of these organizations have been audited, the organizations that participated in the Bight'03 will be subject to a field audit to confirm the capabilities that existed and were documented for the Bight'03.

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

Lead 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 Lead 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. At the very minimum, each vessel will be required to have the following instrumentation: A Differential Global Positioning System (DGPS); a radar; and a fathometer. A field computer for recording station and sampling information is recommended, but not required.

The Boat Captains will be responsible for accurate occupation of the sampling sites and will assist as necessary in maintaining a record of all station occupation and sampling event information. The information required to be recorded for every station occupation and sampling event is described in the Bight'03 Field Operations Manual. The Cruise Leaders are required to assure that all field-collected data are complete and accurate and that station occupation and sampling event data are submitted in electronic form to the Information Management Officer on a weekly basis. These weekly submissions will be reviewed to track the overall sampling progress, identify strata that are at risk of being under-sampled due to unanticipated rates of station abandonment, and to verify that each field team is accurately and completely sampling each station.

B. Laboratory Operations

Several laboratories are participating in Bight'03. Quality assurance and quality control measures are necessary to ensure that the data generated by the participating laboratories are comparable. This section addresses only general laboratory operations. The sections on each indicator (i.e., chemistry, benthic analyses, and toxicity) present specific QA/QC requirements and procedures associated with the processing of specific samples.

The quality assurance measures for Bight'03 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'03 procedures and QA/QC requirements are understood.

- Apresurvey demonstration of laboratory capability

Quality control measures for laboratories participating in Bight'03 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 V of this document. MQOs for benthic analysis are provided in Chapter VI of this document. MQOs for toxicity are provided in Chapter VII of this document.

Documentation of general laboratory practices

All laboratories providing analytical support for chemical or biological analyses must have the appropriate facilities to store and prepare samples, and appropriate instrumentation and staff to provide data of the required quality within the time period dictated by the 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'03 QAP, 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 log books 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'03 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'03 Field Operations Manual (2003). Procedures for benthic analyses are provided in the Infaunal Sample Analysis Laboratory Procedure (SCCWRP, 2003) which is attached as an appendix to this document. Procedures for chemistry, and toxicity analysis 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'03 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'03 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).

IV. MEASUREMENTS OF FISH AND INVERTEBRATE ASSEMBLAGES AND FISH PATHOLOGY

A. Overview

This section presents Bight'03 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'03 Field Operations Manual (2003). The field crews will generate data on species identification, enumeration, biomass, length measurements (fish only), and gross external pathology.

Field crews will conduct a standard 10-min trawl at selected stations (5-min in bays, harbors, and marinas). The Bight'03 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. If appropriate, diseased specimens will be examined by a pathologist.

B. Field Operations

Trawling

Field crews must adhere to prescribed sampling protocols because fish and invertebrate assemblage data (species identification, enumeration, biomass, and length) are significantly influenced by the collection methods. Factors influencing the catch are gear type, 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 Lead 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 field crews new to the survey. 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'03 Field Operations Manual (2003). The quality of fish and invertebrate identification, enumeration, biomass, and length will be ensured through presurvey training, audits, and intercalibration, and in-survey and postsurvey audits.

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

During the survey, each Cruise Leader will check to make sure that the scales are calibrated at the start of each day, that the appropriate identification aids and processing equipment are on board, and that processing follows the protocol described in the Bight'03 Field Operations Manual. In addition, each Cruise Leader 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'03 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 one voucher specimen of each species identified in the field. Prior to the survey, each field crew will be given a list of fish and invertebrate species likely to be encountered in the survey to facilitate tracking of specimens collected. A voucher collection of organisms collected in the Bight'03 trawls will be developed during the survey. The collection will be housed at SCCWRP along with the Bight'03 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'03 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, some definitive examinations may be required and 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'03 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'03 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

There are many aspects to assuring the quality of chemical measurements. This section presents Bight '03 QA/QC protocols and requirements covering a wide range of activities, from sample collection and laboratory analysis, to the final validation of the resultant data. Guidance for much of this section is based on USEPA SW846 and protocols developed for the EMAP-E Virginian Province, as well as those developed over many years by the National Oceanic and Atmospheric Administration's (NOAA) National Status and Trends (NS&T) Program. The protocols described herein are applicable to low parts-per-billion analyses of both marine sediment and fish samples unless, otherwise noted.

The Bight '03 survey will measure a variety of organic and inorganic contaminants in marine sediment and whole fish samples (Table 5.1). In addition, the Bight '03 survey requires that the participating analytical laboratories demonstrate comparability continuously through strict adherence to common QA/QC procedures, routine analysis of Certified Reference Materials (CRMs), and regular participation in interlaboratory comparison exercises (round-robin analyses). The QA/QC program has adopted a "performance-based" approach to achieving quality assurance of low-level contaminants. Laboratories are not required to use the same analytical methods for each type of analysis. Instead, Teach laboratory is free to choose the best, or most feasible method available within the constraints of cost and equipment, and provided that the resulting data meets all of the specified QA/QC criteria for accuracy, precision and sensitivity..

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

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

B. Sample Collection, Preservation and Holding Time

Field personnel must strictly adhere to Bight '03 protocols to insure 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 that 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)

- Conditions for sample collection, preservation and holding times should be followed (Table 5.2).

C. Laboratory Operations

Overview

The Bight '03 survey will involve the distribution of sediment and tissue chemistry samples among several different laboratories. Each participating laboratory will analyze samples using existing methodology and report results only for the constituents listed in Table 5.1.

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

The performance-based Bight '03 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 '03 program are summarized in Tables 5.3-5.6. The following sections discuss general aspects of the QA/QC elements.

Prior to the analysis of samples, each laboratory should calculate nominal MDLs for each analyte; establish an initial calibration curve for all analytes; and demonstrate acceptable performance on a known or blind accuracy-based material. Following a successful first phase,

the laboratory must demonstrate its continued capabilities by participating in an on-going series of interlaboratory comparison exercises; repeated analysis of certified reference materials (CRMs); laboratory control standards; and analysis of laboratory method blanks and spiked samples. These steps are detailed in the following sections.

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

To accomplish the objectives of the Bight '03 study, three criteria must be met for any analytical methods used:

- Sufficient sensitivity must be obtained to achieve the required data reporting objectives for any target analytes (Table 5.7). 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 the other laboratories. Laboratories analyzing the Bight '03 samples must participate in the on-going intercalibration exercises. The acceptable performance for any given laboratory is that the concentrations of any measurable constituents must be within three standard deviations of the average measured concentrations reported by all of the laboratories that analyzed for that constituent (precision criteria). Alternatively, the results must be within specified limits agreed upon by the intercalibration groups.

- Analyses of certified reference materials must yield values within the specified range of the certified values (Tables 5.3-5.6). However, due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy will only apply to analytes having certified values that are >10 times the MDL established by the laboratory (accuracy criteria).

The on-going intercalibration exercises are used to provide an initial check on the performance of the participating laboratories against these criteria. Any laboratory that fails to meet these criteria should repeat analyses of the intercalibration samples before commencing analyses of actual Bight '03 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 '03 study are summarized in Tables 5.3-5.6. 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 5.3-5.6).

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

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

Initial documentation of method detection limits

In the Bight '03 program, the MDL will be used to demonstrate the capability of a laboratory to reach the sensitivity required to measure a specific constituent and demonstrate acceptable precision. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition: *"The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte."* The calculated MDL is a function of method precision at low analyte concentrations. 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. The MDLs should be determined in both fish tissue and sediment, using "clean" sample matrices in order to minimize the interference by other compounds in a sample on the estimation of detection limits for the 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 target analyte and each analytical method employed. Briefly, at least seven replicates of each representative matrix should be spiked at a concentration between one and five times the estimated detection limit (except for certain trace metals; see below for details), or at RL as a default. The amount of sample (i.e., mass of sediment or tissue) used in calculating the MDL should match, as closely as possible, the amount of sample typically used. The mean and standard deviation of the replicates are used to

compute the MDL by multiplying the standard deviation by the Student t value for the 99% confidence interval (for n=7, t=3.143).

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

Reporting levels

In the Bight '03 program, RLs are used to report concentrations of target analytes (Table 5.7). The Bight '03 Chemistry Committee has defined RLs as the lowest concentration of any specific calibration range. The RL is therefore the lowest quantitative value that 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 '03 Reporting Levels.

Trace Metals. The maximum acceptable MDLs are set at one-fifth of the effects range low (ERL) NOAA sediment quality guideline, for those analytes for which an ERL has been developed. For the purpose of this study, reporting levels (RLs) are used interchangeably with maximum acceptable MDLs. In the case of analytes for which no ERL has been established, the RL will be set by the individual laboratories at the lowest reasonable level with consideration of the analyte, the matrix, and the analytical methods used.

The RL for the whole fish samples will be set at three standard deviations above the calculated MDL for each respective analyte, with the understanding that this value will vary somewhat among laboratories. The reason for this are two-fold: 1) there are no fish concentration guidelines for metals similar to those established for sediments, and thus there is no basis for the establishment of an *a priori* target RL; and 2) the data from the fish metals analyses will be used primarily in the mass balance assessment component of the Bight '03 survey, and thus there is an impetus to push RLs as low as reasonably possible to account for as much of the mass of each trace element as possible within the fish "compartment" of the Southern California Bight. Although there are some guidelines established for fish and shellfish to protect humans and animals from risks due to consumption, these concentrations are in the parts-per-million range, and thus at least an order of magnitude above current analytical detection limits.

Trace Organics. The RLs for the polynuclear aromatic hydrocarbons (PAHs), chlorinated pesticides, and polychlorinated biphenyls (PCBs) in sediments are set based on the combination of the ERL values and historical data. Fish tissue RLs for the chlorinated hydrocarbon analytes are based on tissue residue guidelines for protection of wildlife, as recommended by Environment Canada.

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 seven replicates of clean matrix spiked at or near the RL should be $\leq$ 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 section 5.3.10).

Calibration verification

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

For continuing trace metal measurements, the continuing calibration verification (CCV) verifies that the instrument stays in calibration throughout the analysis. The CCV is prepared in the same acid matrix as the calibration standard. It is analyzed after every ten samples and at the end of the run. The CCV can come from any source that is near the mid-range of the calibration and must be within the ranges specified in Table 5.3.

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 analysis of the calibration verification standard is within the specified control limits (Tables 5.3-5.6), the analyst(s) should identify and eliminate the source(s) causing the failure and perform another calibration verification. If problem persists, preventive maintenance or corrective actions must be performed. Another calibration verification standard is then analyzed, and the results should be assessed using the calibration verification criteria (Tables 5.3-5.6). If the calibration verification criteria are not met, a new initial calibration must be performed. No sample analysis should begin until 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 5.3-5.6) are based on the laboratory's maximum acceptable method detection limits (trace metals) or reporting levels (trace organics and TOC) as documented prior to the analysis of samples. For trace metals, it is preferable that the level of any analyte in the method blank be below the MDL, Alternatively, the concentration of any target analyte must be less than 5% of the ERL for those constituents that have an ERL established, or less than 5% of the concentration of the analyte in the sample for those analytes without established ERL values. . A reagent blank concentration equal to or greater than three times the MDL for one or more of the analytes of interest requires definitive corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis. For trace organics, if the method blank contains any analyte with a measured concentration greater than RL, all samples for that batch should be re-analyzed if the analyte is detected in samples. Concentrations lower than RL should be reported, but not used to correct concentrations in the field samples.

Sample duplicates

Analysis of sample duplicates is used to assess the precision of an analytical method in quantifying target analytes and not required for all methods. The relative percent difference (RPD) between the sample and sample duplicate results is calculated as follows:

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

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

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

Matrix spikes and matrix spike duplicates

A laboratory spiked sample matrix (commonly called a matrix spike or MS) and a laboratory spiked sample matrix duplicate (commonly called a matrix spike duplicate or MSD) will be used both to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest and to provide an estimate of analytical precision. A minimum of one MS should be

analyzed for 10% of samples. The matrix spike solution should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be between 10 and 100 times the MDL for that analyte, as previously calculated by the laboratory. If the unspiked sample contains more than this amount, then the sample should be spiked with one to five times the preexisting concentration in the sample.

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

For trace metals, the spike control limits are presented in Table 5.3 for all elements except iron and aluminum due to their high concentrations. If the percent recovery for any analyte in the MS or MSD is lower than the control limits, the raw data quantitation reports should be reviewed. If the reason for a low percent recovery value is not identified, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interference and further instrument response checks may not be warranted, especially if the low recovery occurs in both the MS and MSD, and the other QC samples in the batch indicate that the analysis was "in control". An explanation for low percent recovery values for MS/MSD results should be given in the cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response must be included.

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 5.3-5.6 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 '03 study is presented in Table 5.8.

A laboratory control material (LCM) may be used in addition to, but not as a replacement for, CRMs.. A LCM is similar to a CRM in that it is a homogeneous matrix that closely matches the samples being analyzed. For the Bight '03 study, two sediment materials from the Palos Verdes Shelf (PV7C and MRS032803) were used as LCMs in addition to the required CRMs for the initial interlaboratory calibration study. Although the concentrations of the target analytes in these materials are not certified, they can be used to assess the precision (i.e., consistency) of a single laboratory, and to determine the degree of comparability among different laboratories. In practice, LCMs may be preferred for routine (i.e., day to day) analysis because CRMs are relatively expensive. Moreover, as-collected (i.e., wet) LCMs from the study area are more representative of the types of samples that will be delivered to the laboratories during the actual study. However, for the Bight '03 study the specified CRMs must be analyzed with every sample batch to provide a check on analytical performance.

Routine analysis of CRMs and LCMs is a vital aspect of the "performance-based" Bight '03 QA philosophy. For the organic analyses, one CRM (NIST1944) and must be analyzed along with each batch of samples (Tables 5.3-5.6 and 5.9). For the metals analyses, two CRMs (540 and 016-050) must be analyzed with each batch of samples. However, only one of these CRM (540) will be used for determination data acceptability criteria. For CRMs, both the certified and non-certified concentrations of the target analytes should be known to the analyst(s) and should be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs and LCMs (Tables 5.3-5.6 and 5.9).

If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM, the data for the entire batch of samples is suspect. Calculations and instruments should be checked; the CRM may have to be reanalyzed (i.e., re-injected) 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 -5.5. Each laboratory should set its own control limit criteria based on the experience and best professional judgment of the analyst(s). It is the responsibility of the analyst(s) to demonstrate that the analytical process is always "in control" (i.e., highly variable surrogate recoveries are not acceptable for repeat analyses of the same certified reference material and for the matrix spike/matrix spike duplicate).

Internal standards (organics)

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

Whole fish sample processing

Whole fish samples will be processed as in the Bight'98 survey, by homogenization in a blender with an equal mass of ultra-pure water (~18 megaohom and organic free). In the Bight'03 survey, fish samples will be analyzed for trace metal constituents as well as for orgaincs, and thus every effort should be made to avoid both contamination by both organic and inorganic species. Therefore samples will be processed in a glass blender cup,fitted with new blades assemblies. The stainless steel blades are coated with TiN in an effort to minimize trace metal contamination. However, other wetted parts are not coated and may introduce metal contaminatio to the samples. Refernce fish sample "blanks" (e.g. Orange Roughy) may be used to monitor trace metal contamination from processing. In addition, blades assemblies will be continuously inspected for signs of corrosion or abbrasive wear, and will be replaced as necessary to minimize the potential for contamination.

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. The analytical laboratory must be notified of any additional information or corrective actions deemed necessary after the data evaluation. Following satisfactory resolution of all "corrective action" issues, the final action is to notify the laboratory in writing that the submitted results have been officially accepted as complete. It may be necessary or desirable for a team of individuals (e.g., 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. In addition, if holding times have been exceeded, options for reanalysis may be limited. The following steps are to be followed and documented in evaluating Bight '03 chemistry data:

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

Checking data completeness

The first part of data evaluation is to verify that all required information has been provided in the data package. For the Bight'03 survey, 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 '03 chemistry database to verify that the correct format has been supplied.

- Once the data have been entered into the appropriate Bight'03 database, automated checks should be performed 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.

Assessing data quality

Data validation, or the process of assessing data quality, can begin after Bight'03 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'03 laboratories are expected to submit data that has been tabulated and checked thoroughly for accuracy; the raw data reports needed to perform these checks (e.g., chromatograms, original quantitation reports) are not submitted as part of the data package. The laboratory is required to maintain this raw data in an orderly manner and to have these records available for review by Bight'03 personnel upon request. The first-step validation checks performed by Bight'03 personnel will be limited to the following:

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

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

3) A check to verify that the reported concentrations for each analyte fall within "environmentally-realistic" ranges, determined from previous studies and expert judgment. In addition, past studies indicate that the different compounds in each class of chemicals being measured on Bight'03 (e.g., PAHs, PCBs, DDTs and other chlorinated pesticides) typically occur in the environment in more or less fixed ratios to one another. For example, the DDT breakdown products p,p-DDD and p,p-DDE typically occur at higher concentrations than p,p-DDT in marine sediments in off Southern California. If anomalous departures from expected relative concentrations are found, it may indicate a problem in the measurement or data reduction, which in turn warrants further investigation.

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

1) Results for QA/QC samples should be tabulated, summarized and evaluated. A set of summary tables should be prepared from the database showing the percent recovery values and relative percent difference values (where applicable) for the CRMs, LCMs and matrix spike/matrix spike duplicate samples. The tables should indicate the percent recovery values for each individual batch of samples, as well as the average, standard deviation, coefficient of variation, and range for all batches combined.

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

3) The summary results, particularly those for the CRMs and/or LCMs should be evaluated by comparing them against the QA/QC warning and control limit criteria for accuracy, precision, and blank contamination specified in Table 5.3.

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

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

Assigning data qualifier codes

Data qualifier codes are notations used by laboratories and data reviewers to briefly describe, or qualify, data and the systems producing data. Bight '03 data reviewers will assign data qualifier codes in situations where there are violations of control limit criteria. The most typical situation is when a laboratory fails to meet the accuracy control limit criteria for a particular analyte in a CRM or matrix spike sample. In these situations, the 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 '03 that data which are qualified as estimates because of minor violation of a control limit in a QA/QC sample are still usable for most assessment and reporting purposes. However, it is important to note that all QA/QC data will be readily available in the database along with the results data, so that interested data users can make their own estimation of data quality.

Taking final action

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

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

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

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

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

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

The chemistry QA results will be presented in the appropriate Bight '03 technical reports,and will also become a permanent part of the database documentation (i.e., meta data). The QA/QC data collected by the Bight '03 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.

E. Summary Of QA/QC Requirements For Analysis Of Chemical Contaminants In Sediments And Whole Fish

The Bight '03 QA/QC requirements for chemical analysis are performance-based. Key quality assurance (QA) measures include:

- MQOs for accuracy, precision and completeness (Table 2.1)
- Specifications for sample collection and holding times (Table 5.2)
- Control limit criteria and recommended frequency of analyses for each QA/QC sample type required in Bight '03 (Table 5.3-5.7)
- Target reporting levels for each analyte (Table 5.7).
- Pre-survey demonstration and documentation of performance by all participating laboratories that they can meet the detection level and precision objectives for each of the target analytes.

- Requirement of participation in pre-survey interlaboratory calibration exercise to assess comparability of results with other participating laboratories.
- Requirement of pre-survey analysis of certified reference materials to assess the Bight ‘03 accuracy criteria.

The required quality control (QC) measure for the Bight ’03 chemical analyses is:

- Evaluation of the results from QA/QC samples by each laboratory after completion of each analytical sample batch.

Table 5.1
Bight '03 Marine Monitoring Survey Target Analyte List
For Sediments and Fish Tissue†

Trace Metals	PAHs†	PCBs	Pesticides
Aluminum		PCB-18	4,4'-DDT
Antimony	Low Molecular Weight	PCB-28	2,4'-DDT
Arsenic		PCB-37	4,4'-DDD
Barium	Acenaphthene	PCB-44	2,4'-DDD
Beryllium	Acenaphthylene	PCB-49	4,4'-DDE
Cadmium	Anthracene	PCB-52	2,4'-DDE
Chromium	Biphenyl	PCB-66	α-Chlordane
Copper	Fluorene	PCB-70	γ-Chlordane
Iron	2-Methylnapthalene	PCB-74	lipid
Lead	1-Methylphenanthrene	PCB-77	
Mercury	Naphthalene	PCB-81	
Nickel	1-Methylnapthalene	PCB-87	
Selenium	2,6-Dimethylnaphthalene	PCB-99	
Silver	1,6,7-Trimethyl-naphthalene	PCB-101	
Zinc	Phenanthrene	PCB-105	
		PCB-110	
		PCB-114	
		PCB-118	
		PCB-119	
		PCB-123	
	High Molecular Weight	PCB-126	
		PCB-128	
	Benz[a]anthracene	PCB-138	
	Benzo[a]pyrene	PCB-149	
	Benzo[b]fluoranthene	PCB-151	
	Benzo[e]pyrene	PCB-153	
	Benzo[g,h,i]perylene	PCB-156	
	Benzo[k]fluoranthene	PCB-157	
	Chrysene	PCB-158	
	Dibenz[a,h]anthracene	PCB-167	
	Fluoranthene	PCB-168	
	Indeno(1,2,3-c,d) pyrene	PCB-169	
	Perylene	PCB-170	
	Pyrene	PCB-177	
		PCB-180	
		PCB-183	
		PCB-187	
		PCB-189	
		PCB-194	
		PCB-201	
		PCB-206	

† Al, Fe and PAH analyses to be performed for sediment only, not fish tissue

Table 5.2
Summary of chemistry sample collection and holding time conditions for the Bight '03

Parameter	Container Type	Container Size (mL)	Preservation Requirements	Maximum Holding Time
Sediment grain size	plastic or glass	125 (80% full)	cold (4ºC)	6 months
Sediment total organic carbon	glass	2500 (80% full)	frozen (-20ºC)	6 months
Sediment trace metals	glass or plastic	250 (80% full)	frozen (-20ºC)	1year
Sediment trace organics	glass	250 (80% full)	frozen (-20ºC)	1 year
Fish trace organics and trace metals	aluminum foil wrapped in plastic bags	whole fish	frozen (-20ºC)	1 year[1]
Fish puree (organics)	glass	250 (80% full)	frozen (-20ºC)	1 year
Fish puree (trace metals)	glass/plastic	250 (80% full)	frozen (-20ºC)	1 year[1]

[1] In all cases the maximum holding time for mercury is 6 months.

Table 5.3
Summary of the data quality requirements for the
Bight '03 trace metal measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Method Blank	1/batch	<MDL or <5% of the measured concentration in samples
Certified Reference Materials ERA Soil #540 RTC CRM016-050	1/batch	See Table 5.8
ICP-AES		
Calibration	Initial setup	Minimum 1 blank and one calibration standard
Interference check	1/run	±20% true value
Initial calibration verification (ICV)	2 points/batch	±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value. Should all spiked sample recoveries be outside 30% of true value, add a post-digestion spike to the unspiked sample and analyze. If all spike recoveries are outside 30% of true value, note matrix caused poor spike recovery. If all spike recoveries are within 30% of true value, repeat digestion. Spike duplicate results must have an RPD $\leq$ 20% if MSD is analyzed.
Spiked blank	1/batch	±25% true value
Duplicate sample or matrix spike sample	10%	Statistical process control analyses (within 3σ)
ICP-MS		
Tuning solution	4 at start of run	RPD < 5%
Calibration	Initial setup	Minimum 1 blank and three calibration standards
Initial calibration verification (ICV)	1 points/batch	±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Calibration Blank	10%	<MDL. If > MDL, run two more times, the average must be <MDL. If average > MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value; ≤ 30% RPD for over 10 times MDL. If ≥ 30% RPD and

Table 5.3
Summary of the data quality requirements for the
Bight '03 trace metal measurements (Cont.)

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

Table 5.4
Summary of the data quality requirements for measurements of polycyclic aromatic hydrocarbons[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 only as part of the on-going QA/QC efforts
Certified reference material	1/batch	Within ± 40% of specified value for 80% of the analytes[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 the AVERAGE of the results from the participating labs ± 3 standard deviations.

Table 5.5
Summary of the 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 evaluation only as part of the on-going QA/QC efforts (performed on clean sediment or tissue)
Certified reference material	1/batch	Within ±40% of the true value for 80% of the analytes[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 the AVERAGE of the results from the participating labs ± 3 standard deviations.

Table 5.6
Summary of the 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 used for
the Southern California Bight Pilot Project.

	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	α-Chlordane	1	10
Iron	[a]	NA	γ-Chlordane	1	10
Lead	9,300	NA	Total organic carbon	[a]	NA
Mercury	30	NA	Lipid	NA	[a]
Nickel	4,200	NA	Sediment grain size	[a]	NA
Selenium	1,000	NA			
Silver	200	NA			
Zinc	30,000	NA			
Acenaphthene	50	NA			
Acenaphthylene	50	NA			
Anthracene	50	NA			
Benzo[a]anthracene	50	NA			
Benzo[a]pyrene	50	NA			
Benzo[b]fluoranthene	50	NA			
Benzo[e]pyrene	50	NA			
Benzo[g,h,i]perylene	100	NA			
Benzo[k]fluoranthene	50	NA			
Biphenyl	50	NA			
Chrysene	50	NA			
Dibenz[a,h]anthracene	100	NA			
Fluoranthene	50	NA			
Fluorene	50	NA			
Indeno(1,2,3-c,d)pyrene	100	NA			
Naphthalene	50	NA			
Perylene	50	NA			
Phenanthrene	50	NA			
Pyrene	50	NA			
2,6-Dimethylnaphthalene	50	NA			
1-Methylnapthalene	50	NA			
1-Methylphenanthrene	50	NA			
2-Methylnapthalene	50	NA			
1,6,7-Trimethylnaphthalene	50	NA			

[a]Report 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.

Table 5.8
Certified reference materials recommended by the Bight '03 Chemistry Technical Committee.

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)	
CRM 1944 (NIST)	PAHs and chlorinated hydrocarbons in marine sediment
LCMs (Field Sediments)	PAHs and Chlorinated hydrocarbons in two marine sedimetns for the Palos Verdes shelf, PV7C and MRS032803; used for pre-survey laboratory intercalibration only; accepatance ranges are determined by the Bight'03 chemistry committee.
CARP-2 (NRC Canada)	Chlorinated hydrocarbons in whole fish
Environmental matrix (Trace Metals)	
CRM-016-050 (RTC)	Metals in stream sediment (for performance evaluation only, not acceptance)
540 (ERA)	Priority Pollutant Soil Certified Standard (used for acceptance criteria)
DORM-2	Metals in Fish Muscle Tissue
Environmental matrix (total organic carbon)	
PACS-2 (NRC Canada)	TOC in marine sediment

VI. MACROBENTHIC COMMUNITY ASSESSMENT

A. Overview

This section provides the Bight'03 QA/QC protocols and requirements for production of biological data about macrobenthic (infaunal) communities, from sample collection through taxonomic analysis. Field and laboratory manuals describing acceptable Bight'03 procedures have been prepared and distributed. Single benthic samples are collected at each station in the survey. Each sample is screened and fixed in the field, returned to one of the participating laboratories, and analyzed for species composition and abundance. The data produced by each laboratory will be aggregated into a single data set and made available for data analysis and interpretation.

B. Field Operations

Sediment samples for macrobenthic infaunal analysis will be collected at each station using a SCCWRP-modified 0.1 m^2 Van Veen grab (Stubbs et al. 1987). The participation of several different vessels and field sampling teams in Bight'03 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 (2003) and instruction on sampling procedures, application of sample acceptance criteria, sample processing, and the collection of required sampling event information. All personnel are expected to understand and properly carry out all steps in the collection, screening, relaxation, and fixation of infaunal samples, and the subsampling and handling of sediment chemistry and toxicity samples.

As described in the Field Operations Manual (2003), pre-survey field audits will be used to establish the capability of field sampling teams. During the field audits, the auditor will provide corrective instruction as necessary. Field audits will also be conducted during the Bight'03 survey to assure that sampling is conducted in a uniform manner and field crews record all required information.

A Measurement Quality Objective (MQO) of 90% has been established for completeness of the field collection of benthic samples. This completeness goal was established in an attempt to derive the maximum statistical power of the sampling design. The MQO was not set at 100% in recognition that the randomized selection of sampling sites employed in the Bight'03 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 are provided in the Field Operations Manual.

Sample acceptability criteria have been established in the Field Operations Manual (2003) based on sample condition and depth of penetration of the grab. An acceptable grab is characterized by an even surface with minimal disturbance and little or no leakage of overlying water, and a penetration depth of at least 5 cm, if the target depth of 8 cm cannot be achieved. Samples not meeting these criteria are rejected.

C. Laboratory Operations

The laboratory analysis of infaunal samples for the Bight'03 involves three processes: sample treatment and storage, sample sorting, and organism identification and enumeration. Quality assurance in the form of procedures and standardized reporting requirements are provided in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual (2003) for all three processes. For the most challenging process, organism identification, additional quality control and quality assessment steps are included in order to foster comparability among the taxonomic data sets produced by the participating laboratories. The quality assessment steps for taxonomic analysis are discussed separately below.

Sample treatment and storage

In the laboratories, samples will be stored in a 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 low 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, then top-off the sample using 100% ethanol.

Sample sorting

Sorting must be accurate and complete to assure the value of all the subsequent steps in the sample analysis process. As organisms are removed from the organic and inorganic residues (debris) that compose the sample, they are sorted into broad taxonomic categories for subsequent taxonomic analysis. A standard sorting form is used for tracking the sample. It includes the name of the laboratory and technician responsible, time required for sorting, number of taxa lots and sample containers, 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 alternative approaches are used for re-sorting: the Aliquot method, or the Whole Sample method. In the first method, a 10% aliquot of every sample processed by a sorter is resorted. In the second, 10% of the samples processed by a sorter are completely resorted. In both cases, an experienced sorter other than the original sorter conducts all re-sorting. For Bight'03, 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. Upon completion of all quality control and assessment steps for the survey, the Benthic Committee Chairperson (or designee) will notify each participating laboratory that the sample debris may be discarded.

Taxonomic analysis

The goal of taxonomic analysis for Bight'03 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'03 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, the sharing of expertise, and on participation in monthly workshops. Results of the workshops and other information are communicated to the membership through a monthly newsletter.

SCAMIT's cooperation includes the provision of standards for nomenclature and a mechanism for mutual assistance and exchange of information among the taxonomists involved in Bight'03. The taxonomic nomenclature used in Bight'03 follows the SCAMIT hierarchical species listing (SCAMIT 2001). This list represents a consensus for standard usage of taxon 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'03 workshops prior to the sampling period that 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 local expert(s) to be called upon for assistance during sample analysis.

In order to assure that the data produced by the Bight'03 survey meet the standards set during the previous two regional surveys, it is essential that all participating taxonomists have the expertise and experience necessary to produce data of comparable quality. Qualification criteria have been established to assure that the taxonomists participating in the Bight'03 are capable of meeting that standard. Each organization will provide a list of taxonomists and their

specialty areas. Agencies or their contractors employing taxonomists who did not perform analysis of infaunal samples for the SCBPP or Bight'98 are required to assure that their taxonomists meet the qualifying criteria prior to participation in the Bight'03 Macrobenthic survey. The two criteria are:

- Candidate taxonomists who will be working under the direct oversight and guidance of an experienced taxonomist who analyzed samples in either the SCBPP or Bight'98 are considered to meet the standard for Bight'03.

- Candidate taxonomists who will be not be working under the direct supervision and guidance of an experienced taxonomist who analyzed samples in either the SCBPP or Bight'98 must complete and pass a qualification exercise prior to acceptance as a taxonomist for Bight'03.

In summary, the exercise referred to above is based upon that used as quality control and assessment in the SCBPP and Bight'98 Surveys (Montagne & Bergen 1997, Ranasinghe et al. 2003). The candidate taxonomist will analyze two lots of specimens from samples previously analyzed by SCBPP/Bight'98 taxonomists. The results of the analysis are compared to those of the original taxonomist and the discrepancies classified. Each discrepancy found to be the result of error on the part of the candidate taxonomist conducting the re-analysis is tallied. The effect upon the number of taxa, organism count, and the accuracy of identification will be determined and a percent error of analysis calculated. The candidate taxonomist must be able to meet the measurement quality objective (MQO) of 10% for each of the parameters. Based upon this assessment, the Benthic Committee will provide a report to the Bight'03 Coastal Ecology Planning Committee recommending the acceptance or rejection of the candidate taxonomist.

After sample analysis has begun, SCAMIT/Bight'03 workshops will continue at least monthly to address taxonomic problems arising during analysis of the Bight'03 samples. Protocols for the erection and documentation of provisional species names, based largely upon SCAMIT recommendations (SCAMIT 1986), are provided in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual (2003). 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'03 workshops will culminate in a synoptic data review (SDR) of the data set compiled from all laboratories, and investigation of possible inconsistencies revealed in that process (including examination of voucher specimens or sample lots as needed for resolution). The SDR also draws upon the results of the quality control re-analysis of 10% of the samples analyzed by each laboratory (described below).

While the SCAMIT/Bight'03 workshops are the primary means for exchange of information and assistance, the taxonomists participating in analysis of Bight'03 samples should maintain frequent interaction throughout the process. Timely and frequent communication among the taxonomists analyzing the samples will improve the data produced in the survey. An e-mail list-server will be established that will facilitate this communication. All taxonomists involved in the Bight'03 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.

Appropriate uses of the list server are informing the other members of unusual or newly encountered species, the erection of in-house provisionals, and requests for information or assistance.

The creation and maintenance of voucher collections is an essential element of the QA/QC process. A voucher collection is an invaluable tool during the course of the study, when access to voucher specimens greatly assists the taxonomists in avoiding inconsistent identifications. Upon completion of the study, voucher collections provide other workers the means to determine the identity of species as understood by the original taxonomist. Each participating laboratory must create a voucher collection of all species identified in Bight'03 samples analyzed in that laboratory. Procedures for the creation, maintenance and documentation of the voucher collections are provided in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual (2003). These collections are separate from the laboratories' existing voucher collections and will be the source of material from which is drawn a common Bight'03 voucher collection upon completion of the survey. These collections provide material for review during SCAMIT/Bight'03 workshops and the synoptic review of the data upon completion of analysis.

The ultimate repository of the Bight'03 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 against 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 project sample material. The ideal facility for the repository will be located within the region and will make a long-term commitment to the maintenance of such collections, including curatorial care and management of future access.

Quality assessment of taxonomic analysis

While the quality of taxonomic analysis in Bight'03 relies heavily on the measures described above, quality control and assessment is also provided by the re-identification of 10% of the samples processed by each team of taxonomists. Re-identification will be conducted by a team other than that which originally analyzed the samples. Samples for re-identification are selected randomly from each team's assigned set of samples and randomly re-distributed to the other teams. The taxonomists conducting the re-identification do not have access to the original results. The results of the re-analysis are compared to the original results and a standardized comparative report of discrepancies is prepared. The taxonomists responsible for the original and re-analytical results reconcile the discrepancies. In the process, errors made by the original taxonomists are classified and the number of each type of error recorded. Examples of errors are misidentifications, miscounts, overlooked specimens, or misapplication of counting rules. Errors are discriminated from discrepancies resulting from differences in levels of identification. For example, the discrepancy between a report of *Photis* sp and *Photis lacia* does not represent an error, but rather a decision by one taxonomist to identify the specimen only to genus level. This decision may be based on the taxonomist's judgment that the specimen's condition is too poor for species identification, or may reflect his or her lack of expertise in this particular group of organisms. In the latter case, the difference in treatment provides an indication where assistance from other taxonomists involved in the Bight'03 is needed. Nomenclature differences are also

examples of discrepancies that are not classified as error. In addition to assessing analytical accuracy and the survey data quality relative to the MQOs, this process provides information for the SDR performed at the end of the survey as the last step in compiling a final survey data set.

Based upon the results of data quality assessment for the SCBPP and Bight'98, 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. Accuracy is calculated as the percent error in the original results. Percent error will be calculated for three aspects of sample analysis; number of taxa discriminated ($\%Err_{\#\ Tax}$), total organism count ($\%Err_{\#\ Orgs}$), and identification accuracy ($\%Err_{ID}$).

The error rates are calculated as follows:

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

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

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

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 results of this assessment process will provide a measure of the quality of Bight'03 infaunal data, and add to the baseline for selection of MQOs in future regional surveys based upon the this model.

D. Information Management

Record keeping and reporting

Each laboratory is responsible for maintaining thorough and complete records through all stages of the sample analysis and QC procedures. Each laboratory will employ its own bench sheet for taxonomic analysis. For the Bight'03 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 Committee Chairperson (or designee) upon completion of sample analysis. To insure against loss of documents, copies of all these documents are to be retained by the individual laboratories.

The laboratory supervisor is responsible for assuring that all steps in the process of analyzing infaunal samples follow Bight'03 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. Analytical results are to be transmitted to the Information Management officer in electronic data files that conform to Bight'03 data submission formats and standards as described in the Information Management Plan. It is the submitting laboratory's responsibility to see that these standards are met.

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'03 Survey. Only one toxicity test will be used for assessment of the sediment. The toxicity of whole sediment will be measured using an amphipod (*Eohaustorius estuarius*) survival test.

B. Laboratory Capability

Prior to participating in the Bight 03 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.

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

D. Sample 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 press sieved through a 2 mm screen and 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 one of the exposure treatments (e.g., the median test concentration). 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 and end of the 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.

Interference by ammonia

The presence of high concentrations of ammonia in pore water may be a confounding factor for sediment toxicity tests with *Eohaustorius*. Laboratories will be required to measure the concentration of un-ionized ammonia in the pore water from each station, prior to the start of toxicity testing. If the pore water concentration exceeds the limit of 0.8 mg/L un-ionized ammonia for any station within a batch, the laboratory will be required to perform a similtaneous ammonia reference toxicant test along with that batch. The results of the ammonia reference toxicant testing will be compared to the concentrations of ammonia in the test samples to determine if the levels are high enough to account for any observed toxicity in the sediment samples.

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.

A sediment holding time of no more than two weeks has been established in order to minimize the potential alteration of the sediment toxicity due to storage; this time period is not a criterion for judging test acceptability. Tests on samples that are stored from greater than two weeks up to four weeks will also be considered valid, but a data qualifier will be attached to the record to indicate that the desired storage time was been exceeded. Samples stored for more than four weeks before the start of toxicity testing will be considered unacceptable for testing and the data will not be included in the project database.

VIII. LITERATURE CITED

American Public Health Association, American Water Works Association, and Water Pollution Control Federation. 1989. Standard Methods for the Examination of Water and Wastewater, 17th ed. American Public Health Association, Wash, DC

EPA. 1994. Methods for assessing the toxicity of sediment-assiciated contaminants with estuarine and marine amphipods. United States Environmental Protection Organization, Office of Research and Development, EPA/600/R-94/025.

Hunt, D.T.E., and A.L. Wilson. 1986. The Chemical Analysis of Water: General Principles and Techniques. 2nd ed. Royal Society of Chemistry, London, England 683 pp.

Montagne, D.E. and M. Bergen. 1997. Quality Control and Assessment of Infaunal Identification and Enumeration: The SCBPP Experience. pp 147-154. In: Weisberg, S.B., C. Francisco, D. Hallock, (eds.). Southern California Coastal Water Research Project Annual Report 1996. Westminster, CA.

NRC (National Research Council). 1990a. Managing Troubled Waters. The Role of Marine Environmental Monitoring. National Research Council. National Academy Press, Washington, D.C.

NRC. 1990b. Monitoring Southern California's Coastal Waters. National Research Council. National Academy Press, Washington, D.C.

Ranasinghe, J.A., D.E. Montagne, R.W. Smith, T.K. Mikel, S.B. Weisberg, D. Cadien, R. Velarde and A. Dalkey. 2003. Southern California Bight 1998 Regional Monitoring program: VII. Benthic macrofauna. Southern California Coastal Water Research Project. Westminster, CA. 91 p + 9 Appendices.

SCAMIT (Southern California Association of Marine Invertebrate Taxonomists). 1986. Protocols and Recommendations for the Use of Open Nomenclature in SCAMIT Newsletter, May 1986, Vol. 5 No. 2.

SCAMIT. 2001. A Taxonomic Listing of Soft Bottom Macro- and Megainvertebrates from Monitoring Programs in the Southern California Bight. Edition 4. Southern California Association of Marine Invertebrate Taxonomists, San Pedro. 192 pp.

Schiff, K., S. Weisberg, and V. Raco-Rands. 2002. Inventory of ocean monitoring in the Southern California Bight. *Environmental Management* 29:871-876

Southern California Bight Pilot Project. 1994. Southern California Bight Pilot Project Field Operations Manual. Southern California Coastal Water Research Project, Westminster.

Stanley, T.W. and S.S. Verner. 1985. The US Environmental Protection Organization's quality assurance program. Pp 12-19 In: J.K. Taylor and T.W. Stanley (eds.). Quality Assurance for Environmental Measurements, ASTM STP 867. American Society for Testing and Materials, Philadelphia, PA.

Stubbs, Harold H., Dario W. Diehl, and G. Patrick Hershelmann. 1987. A Van Veen Sampling Method. Technical Report 276. Southern California Coastal Water Research Project, Long Beach. 4 pp.

Taylor, J.K. 1987. Quality Assurance of Chemical Measurements. Lewis Publishers, Inc., Chelsea, MI. 328 pp.