

QUALITY ASSURANCE PROJECT PLAN FOR THE

THE SOUTHERN CALIFORNIA BIGHT

PILOT PROJECT

Prepared by:

Southern California Coastal Water Research Project

Bureau of Sanitation, City of Los Angeles

County Sanitation Districts of Los Angeles County

County Sanitation Districts of Orange County

Metropolitan Wastewater Department, City of San Diego

US Environmental Protection Agency, Region IX

Los Angeles Regional Water Quality Control Board

Santa Ana Regional Water Quality Control Board

San Diego Regional Water Quality Control Board

California State Water Resources Control Board

US Environmental Protection Agency, ORD, EMAP

Santa Monica Bay Restoration Project

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1. INTRODUCTION

Several recently published critiques of current monitoring programs in the Southern California Bight have pointed to the need to coordinate monitoring programs on a regional basis (Thomas 1988, Ford and Conway 1988, 1989, NRC 1990). The National Research Council (NRC) suggested that national programs such as NOAA's Status and Trends or EPA's Environmental Monitoring and Assessment Program (EMAP) could serve as the basis for linking monitoring programs.

EMAP provides a framework for establishing regional monitoring in the Southern California Bight (SCB). Although EMAP was developed to address management questions on national and large regional scales, its probability-based sampling design can be applied to smaller regions like the Southern California Bight. In addition, EMAP's emphasis on interagency participation encourages cooperation among local, state, and federal monitoring programs operating within a region and results in improved data and reduced cost for all participants.

The Southern California Bight Pilot Project (SCBPP) was developed as a pilot project to test EMAP design as a design for reference surveys and as an alternative design for compliance monitoring program. Participants in the SCBPP include EMAP and EPA Region IX; California State Water Resources Control Board; the Los Angeles Regional Water Quality Control Board, the Santa Ana Regional Water Quality Control Board, the San Diego Regional Water Quality Control Board; the City of Los Angeles, Environmental Monitoring Division (CLA,EMD); County Sanitation District of Los Angeles County, County Sanitation District of Orange County; Metropolitan Wastewater Department of the City of San Diego; Southern California Coastal Water Research Project; and the Santa Monica Bay Restoration Project.

2. PROJECT ORGANIZATION

Effective project management is a vital component in the success of any environmental monitoring project. This is especially true when the project requires coordinating the efforts of many diverse groups to produce data that are reliable and comparable.

Overall coordination of the project will be the responsibility of SCCWRP. Dr. Jeffrey N. Cross, Executive Director of SCCWRP, is the Project Manager. The Steering Committee is composed of representatives of the 12 participating agencies and other individuals whose technical and/or programmatic expertise can provide guidance to the project.

Steering Committee

Dr. M. James Allen

Mr. Gordon Anderson

Affiliation

Southern California Coastal Water Research Project

Santa Ana Regional Water Quality Control Board

Dr. Mary Bergen	Southern California Coastal Water Research Project
Ms. Elizabeth Carolan	San Diego Regional Water Quality Control Board
Dr. Jeffrey N. Cross	Southern California Coastal Water Research Project
Dr. John Dorsey	Environmental Monitoring Division, City of Los Angeles
Mr. Terrence Fleming	US EPA Region IX
Ms. Janet Hashimoto	US EPA Region IX
Dr. C. Irwin Haydock	County Sanitation Districts of Orange County
Mr. Michael Lyons	Los Angeles Regional Water Quality Control Board
Mr. John Mitchell	State Stormwater Quality Task Force
Ms. Janet Stull	County Sanitation Districts of Los Angeles County
Dr. Kevin Summers	US EPA Office of Research and Development/EMAP
Ms. Patricia Vainik	Metropolitan Wastewater Department, City of San Diego
Ms. Patricia Velez	Santa Monica Bay Restoration Project
Dr. Stephen Weisberg	Versar, Inc.
Mr. Craig Wilson	California State Water Resources Control Board

Mr. Terrence Fleming is the QA Officer and will be responsible for direction the QA components of the project. He will review the manual, assist with training, conduct proficiency tests and audits and summarize the QA information.

Dr. M. James Allen is the Field Coordinator. He will oversee administration and technical components of field operations. He will coordinate the schedule and logistics of field sampling; determine equipment sharing needs; write procedures manuals; develop sample storage and transfer protocols; develop data sheets and a tracking system; implement training programs; and work with the QA Officer and Information Management Coordinator.

Mr. Richard Gossett is the Laboratory Coordinator. He will oversee the administrative and technical components of laboratory analyses. He will coordinate the schedule and logistics of laboratory analyses; write procedures manuals; develop data sheets and a tracking system, implement training programs and work with the QA Officer and Information Management Officer.

Mr. Robert Hall is the Information Management Officer. He will coordinate the schedule and logistics of data reporting and management; develop data transfer formats and protocols; write procedures manuals; and work with the QA Officer and Data Analysis and Reporting Coordinator.

Drs. Mary Bergen and M. James Allen are the Data Analysis and Reporting Coordinators. They will be responsible for coordinating the various groups addressing the different assessment questions with the data collected

during the pilot. They will also be responsible for collating and editing the various portions of the report written by these groups.

Each of the coordinators is supported by technical representatives from each of the agencies and organizations participating in the SCBPP. The coordinators are responsible for overseeing all technical effort in their project areas, and for soliciting and compiling the comments of all members of their technical support groups. The coordinators will act as liaisons for maintaining communication and consensus among project participants throughout the further development and implementation of the SCBPP.

Field Liaisons

Ann Dalkey	City of Los Angeles, Department of Public Works
Joe Meistrell	County Sanitation District of Los Angeles County
Dave W. Montagne	County Sanitation District of Los Angeles County
George Robertson	County Sanitation District of Orange County
Mike Mengel	County Sanitation District of Orange County
Tim Rothans	Metropolitan Wastewater Department, City of San Diego
Harold Stubbs	Southern California Coastal Water Research Project
Dario Diehl	Southern California Coastal Water Research Project

Affiliation

Laboratory Liaisons

Farhana Mohammed	City of Los Angeles, Environmental Monitoring Division
Aurora Elyada	City of Los Angeles, Environmental Monitoring Division
Dave Montagne	County Sanitation District of Los Angeles County
Elly Gabrielian	County Sanitation District of Los Angeles County
Connie Lillis	County Sanitation District of Los Angeles County
Charles Phillips	Science Applications International, Inc.
Ron Velarde	Metropolitan Wastewater Department, City of San Diego
Steve Meyer	Metropolitan Wastewater Department, City of San Diego
Dave Tsukada	Southern California Coastal Water Research Project
Eddy Zeng	Southern California Coastal Water Research Project

Affiliation

QA/QC Liaisons

Aurora Elayda	Environmental Monitoring Division, City of Los Angeles
Dave Montagne	County Sanitation District of Los Angeles County
Don Cadien	County Sanitation District of Los Angeles County
Elly Gabrielian	County Sanitation District of Los Angeles County
James Cowan	County Sanitation District of Orange County
Steve Meyer	Metropolitan Wastewater Department, City of San Diego

Affiliation

Ron Velarde	Metropolitan Wastewater Department, City of San Diego
Steve Bay	Southern California Coastal Water Research Project

The following QA Specialists were responsible for the preparation of the QA manual:

Field Operations	Harold Stubbs
Water Quality	Richard Santangelo
Chemistry	Richard Gossett
Benthos	Dave Montagne
Fish Trawls	Jim Allen
Sediment Toxicity	Steve Bay

<u>Information Management</u>	<u>Affiliation</u>
Paul Pau	City of Los Angeles, Environmental Monitoring Division
Nick Leonard	City of Los Angeles, Environmental Monitoring Division
Janet Stull	County Sanitation Districts of Los Angeles County
April Ford	County Sanitation Districts of Los Angeles County
Chi-Li Tang	County Sanitation Districts of Los Angeles County
Steve Fanizza	County Sanitation Districts of Orange County
Lori Vereker	Metropolitan Wastewater Department, City of San Diego
Steve Meyer	Metropolitan Wastewater Department, City of San Diego
Michael Lyons	Los Angeles Regional Water Quality Control Board
Gordon Anderson	Santa Ana Regional Water Quality Control Board
Elizabeth Carolan	San Diego Regional Water Quality Control Board
Larry Cooper	Southern California Coastal Water Research Project
Dario Diehl	Southern California Coastal Water Research Project
Rick Packard	EcoAnalysis, Inc.

Such distributed coordination provides a mechanism for ensuring that the interest of all SCBPP participants are recognized and considered; it also creates a forum for constructive resolution of any conflicts that may arise during the course of the project.

3. QUALITY ASSURANCE OBJECTIVES

3.1. OVERVIEW

The primary goal of the QA/QC plan is to ensure that the data generated by the participants is comparable. This is especially important for the SCBPP because of the widely distributed implementation proposed for the project. At least five different organizations (including the POTWs, SCCWRP, and contractors) will collect samples for the SCBPP, and at least six different

organizations will be involved in laboratory processing of samples. The POTWs and SCCWRP, who will be responsible for much of the SCBPP sampling, each have their own processing laboratories; consequently, several different laboratories will perform the same kinds of functions for the project (e.g., chemical analyses and sorting and identifying benthic samples). Encouraging and maintaining consistency in field and laboratory operations and ensuring data comparability, therefore, will be critical to the success of the SCBPP.

Data comparability will be achieved through a combination of common methods (where appropriate) and performance based standards. Where common methods have been agreed upon for the SCBPP, QA/QC measures will be used to assure that methods are applied consistently. Where performance based standards are appropriate, QA/QC measurements will be used as a measure of performance. The appropriate QA/QC procedures for each of the monitoring program components (e.g., field operations, water quality, sediment and tissue chemical analyses, benthic analyses, demersal fish analyses) have been established by the SCBPP Steering Committee.

3.2. GENERAL APPROACH TO QUALITY ASSURANCE

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

Quality control (QC) activities are implemented during the monitoring 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.

Because of the distributed implementation of the SCBPP, the QA program will emphasize quality assurance activities. The abilities of the laboratories that process samples from the current compliance monitoring programs are well established and acceptable for the SCBPP. QA activities, therefore, have focused on developing a common field manual and documenting the

comparability of laboratory methods. Training of field and laboratory personnel is focused on communicating goals and objectives of the pilot project as well as any modifications in methods or procedures that have been made for the pilot project 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).

3.3. 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 the SCBPP. Comparability of reporting units and calculations, data base management processes, and interpretative procedures must be ensured if the overall goals of the SCBPP are to be realized; furthermore, SCBPP data must be generally comparable with EMAP data to facilitate data sharing.

Specific MQOs for precision and accuracy, the most readily quantifiable components of data quality, have been identified for the SCBPP to ensure that the data produced by the many field crews and laboratories involved in the project will be comparable. Accuracy is defined as the difference between the measured value of an indicator and its true or expected value, which represents an estimate of systematic error or net bias (Kirchner 1983, Hunt and Wilson 1986, Taylor 1987). Precision is the degree of mutual agreement among individual measurements and represents an estimate of random error (Kirchner 1983, Hunt and Wilson 1986, Taylor 1987). Together, accuracy and precision provide an estimate of the total error or uncertainty associated with a measured value. Requiring participating field crews and laboratories to achieve standard, quantitative MQOs for accuracy and precision will help to ensure that individual data sets are free of any crew- or laboratory-specific bias and that the degree of random error is consistent across data sets. Accuracy and precision goals for indicators to be measured during the SCBPP are provided in Table 3-1. 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 3.1
Measurement Quality Objectives for SCBPP indicators and data.

<u>Indicators</u>	<u>Accuracy</u> ¹	<u>Precision</u>	<u>Completeness</u>
<u>Water Quality</u>			
salinity	0.5 ppt	NA	90%
temperature	1°C	NA	90%
dissolved oxygen	0.5 mg/L	NA	90%
Sediment grain size	NA ²	20%	90%
Total organic carbon	15%	20%	90%
<u>Sediment contaminants</u>			
organics	30%	30%	90%
inorganics	20%	30%	90%
Sediment toxicity	NA	NA	90%
<u>Benthic infauna</u>			
sample collection	NA	NA	90%
sorting	5%	NA	90%
counting	10%	NA	90%
identification	10%	NA	90%
biomass	NA	10%	90%
<u>Demersal fish</u>			
sample collection	NA	NA	90%
counting	10%	NA	90%
identification	5%	NA	95%
length	5 mm	NA	90%
biomass	NA	10%	90%
gross pathology	NA	NA	90%
Contaminants in fish	30%	30%	90%

¹percent error

²not applicable

An MQO for completeness was also defined for the SCBPP. 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

in the SCBPP is 90% for each measurement process. The sampling design for the SCBPP 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.

3.4. QUALITY ASSURANCE AND QUALITY CONTROL

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 SCBPP 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 SCBPP 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.

3.5. FIELD MANUAL AND QUALITY ASSURANCE PROJECT PLAN

A Field Operations Manual (SCBPP 1994) has been prepared to standardize data collection efforts in the field. A single laboratory manual would not be appropriate for the SCBPP since each of the participating laboratories have their own internal operating procedures. Comparability of laboratory efforts will be ensured through compliance with the requirements listed in *the Quality Assurance Project Plan* (QAPP) which identifies performance based standards and the appropriate level of QA/QC.

These manuals were prepared by the field, laboratory and QA/QC coordinators who worked with the appropriate personnel from each of the participating agencies to establish the appropriate procedures for the SCBPP. 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.

4. REQUIREMENTS FOR FIELD AND LABORATORY OPERATIONS

4.1. FIELD OPERATIONS

All field operations conducted by SCBPP participants will be planned and executed in accordance with the logistics plan, which appears in the appendix of the *SCBPP Workplan*, and follows guidelines established for the SCBPP by the Steering Committee, the QA Specialists and their liaisons, and the Field Coordination Team.

4.1.1. Field Operations Manual

Following standard protocols for the SCBPP field operations will be critical to the quality assurance for the project. SCBPP participants use similar methods for the existing compliance monitoring programs; however, methods vary slightly between organizations. Using common methods for the SCBPP will eliminate this variability and help to ensure the comparability of data among organizations. Standard field procedures are documented in the *SCBPP Field Operations Manual* (SCBPP 1994). Copies of the manual will be distributed to the field crews prior to the survey. The field operations manual will provide the basis for protocol calibration exercises and a reference for field personnel during sampling activities.

The field manual includes detailed descriptions of collection procedures, criteria for acceptable samples, and conditions under which samples need to be recollected. For instance, methods for collecting benthic sediment samples include specifications for minimum and maximum depth of penetration. Methods for trawling include trawl duration, trawl speed, and type of gear to use. This degree of detail is intended to ensure that all data will be collected in a similar manner by all field crews.

The field manual will also contain standard data forms for the pilot project. Standard data forms will ensure that all groups record the necessary data and use comparable units of measurement. Using standard data forms will facilitate development of data entry protocols and minimize transcription error.

4.1.2. Protocol Calibration Procedures

Proper training of field personnel is a critical aspect of quality assurance. Organizations participating in the SCBPP will provide personnel who have extensive field experience, but not necessarily with the standard methods selected for this project. Instruction for the SCBPP, therefore, will focus on ensuring consistency in data collection among all field personnel. Chief Scientists and boat captains will be instructed on the field procedures to be followed during the survey, and they will instruct their field crews on the proper procedures for the survey. The Field QA Specialist will audit the field procedures of each participating organization prior to the SCBPP survey to ensure that all field crews have learned and understand the standard sampling protocols.

Chief Scientists and boat captains of all organizations participating in the survey will be required to attend a protocol calibration meeting, which will be conducted several weeks before the survey. The goals and objectives of the pilot project and the individual responsibilities of the Chief Scientist and boat captain will be discussed at this meeting. Meeting participants will be instructed on field procedures for the survey, including proper data entry on field data forms. The meeting will emphasize decision-making procedures, including the criteria for accepting the different types of samples that will be collected and determining whether or not a station can be sampled. Lines of communication within the project and field quality assurance/quality control activities will also be discussed. Each agency will receive a workplan and a field operations manual for the SCBPP before the field survey.

The Chief Scientist of each participating organization will review the field operations to be performed during the survey with his/her crew. Chief Scientists will be responsible for ensuring their crews' compliance with the standard sampling protocols. Pre-survey responsibilities include reviewing the SCBPP workplan and field operations manual with the field crews and conducting training, as needed. Field personnel that cannot or will not perform an operation as required by the pilot project should not participate in that operation.

4.1.3. Field Quality Control and Audits

Quality control of measurements made during the sampling period will be accomplished using a variety of QC sample types and procedures, as described in later sections of this document. The Field QA Specialist will conduct a pre-survey audit of each field crew to ensure compliance with the prescribed sampling protocols. A field QA checklist has been developed to provide comparability and consistency in this process. The Field QA Specialist will provide additional instruction when discrepancies are noted during a field QA audit. This instruction will focus on the review of sampling procedures including water column measurements using a CTD, sediment collection and processing, and trawl processing.

4.1.4. Navigation

Navigation is an important aspect of quality assurance for the SCBPP. The ability to accurately locate sampling sites is critical to the success of the survey. Positioning equipment is vessel-specific; however, a minimum of a Loran-C, a radar, and a fathometer will be required for this project.

Equipment calibration is essential for accurate navigation. The boat captain will be responsible for calibrating the navigation equipment and maintaining a navigation log for all sampling stations. The log includes latitude and longitude coordinates, GPS coordinates (if GPS is available), depth measurements for each station, and daily calibration information. The Chief Scientist will be responsible for reviewing the log as part of the daily QC check of

all completed data forms. The Field QA Specialist will check basic navigation and the completeness and accuracy of the navigation logs.

As position data are received at the Field Operations Center at SCCWRP, automatic-range checks will be performed on station latitude and longitude coordinates. The reported station location will be compared to the expected coordinates and flagged for further investigation if the positions differ by more than 300 m. If discrepancies are found, original data sheets will be reviewed and the Chief Scientist will be contacted to provide an explanation.

4.2. LABORATORY OPERATIONS

This section addresses only general laboratory operations, while the sections on each indicator present specific QA/QC requirements and procedures associated with the processing of specific samples. 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 $\pm 2\%$ 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 25°C.
- 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.

4.2.1. Laboratory Personnel, Training, and Safety

Each laboratory participating in the SCBPP pilot has designate an on-site QA Liaison. This individual will serve as the point of contact for SCBPP 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. Laboratories may be required to demonstrate acceptable performance before analysis of samples can proceed, as described for each indicator in subsequent sections. Laboratory operations will be evaluated on a continuous basis through technical systems audits, performance evaluation studies, and by participation in interlaboratory round-robin programs.

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.

4.2.2. Quality Assurance Documentation

All laboratories must have the latest revisions of the *QA Project Plan* (this document) and *Laboratory Methods Manual*. In addition, the following documents and information must be current, and they must be available to all laboratory personnel participating in the pilot project:

- Laboratory QA Plan: Clearly defined policies and protocols specific to a particular laboratory including personnel responsibilities, laboratory acceptance criteria for release of data, and procedures for determining the acceptability of results.
- Laboratory Standard Operating Procedures (SOPs) - Detailed instructions for performing routine laboratory procedures. In contrast to the Laboratory Methods Manual, SOPs offer step-by-step instructions describing exactly how the method is implemented in the laboratory, specific for the particular equipment or instruments on hand.

- Instrument performance study information - Information on instrument baseline noise, calibration standard response, analytical precision and bias data, detection limits, etc. This information usually is recorded in logbooks or laboratory notebooks.
- Control charts - Control charts must be developed and maintained throughout the project for all appropriate analyses and measurements (see section 4.2.5).

4.2.3. Analytical Procedures

Complete and detailed procedures for processing and analysis of samples in the field and laboratory are provided in the *SCBPP Field Operations Manual* (SCBPP, 1994) and the *SCBPP Laboratory Manual* (In preparation) respectively, and will not be repeated here.

4.2.4. Laboratory Performance Audits

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 the *Methods Manual* and *QA Project Plan* and to assist the laboratory where needed. Additionally, technical systems audits may be conducted. Reviews may be conducted at any time during the pilot project. Furthermore, laboratory performance will be assessed on a continuous basis through the use of internal and external performance evaluation (PE) samples. Laboratories are encouraged to participate in intercomparison studies (round-robins).

5. WATER QUALITY MEASUREMENTS

5.1. OVERVIEW

This section presents Southern California Bight Pilot Project QA/QC protocols and requirements for water quality measurements from collection to final validation. Collection and analysis methods are documented in the Southern California Bight Pilot Project Field Operations Manual, Version 7 (SCBPP 1994). All data are generated by the field crews.

Quality control of the water column measurements made with these electronic instruments has several positive aspects: calibration, preventative maintenance, QC checks prior to deployment, and systematic review of the resultant data and QC results. Specifics of field quality control checks will be discussed in the following sections

5.2. EQUIPMENT AND SOFTWARE

All water quality profiling equipment used during the SCBPP shall be standardized according to the that listed in the field manual. Additionally, data collection specifics and software shall be standardized in a like manner.

5.3. INSTRUMENT CALIBRATION

Conductivity sensors shall be factory calibrated at 6 month intervals; temperature sensors shall be factory calibrated at 12 month intervals. A preventative maintenance diagnostic evaluation of the CTD unit and accompanying sensors shall occur on a three year interval. Lab calibrations of pressure, dissolved oxygen, and transmissometer sensors shall follow the procedures in the SCBPP Field Operations Manual. All calibration and maintenance, whether factory or lab, shall be documented.

5.4. TRAINING

Training of all personnel expected to operate the CTD is necessary to assure reliable operation and results. Operators must read and understand relevant sections in the SCBPP field operations manual. All operators will be certified in the use of this instrument during training. This shall be accomplished by demonstrating proficiency to senior staff experienced in CTD usage and documentation of that proficiency and training via checklists and signoff by that senior staff.

5.5. FIELD MEASUREMENTS

A number of factors can adversely effect the performance of the CTD or any or all of the attached sensors. The dissolved oxygen sensor is the most effected, although the conductivity sensor is susceptible as well. The most commonly encountered problems are:

- CTD descent rate too rapid for thermal equilibration of dissolved oxygen sensor; especially pronounced during periods of seasonal stratification of the water column. A descent rate of 0.5-0.75 m/s yields optimal dissolved oxygen results.
- Insufficient surface equilibration time prior to deployment; most Sea-Bird CTD unit pumps have a 45 s delay. A 90 s surface soaking time is recommended prior to deployment in order to activate the pump and thermally equilibrate the sensors; A 3 min soak time is recommended after first power up of the system.
- Pinched tubing in the temperature-conductivity-dissolved oxygen-pump line will yield unreliable flow rates and thus potentially unreliable conductivity and dissolved oxygen results.
- Mud being drawn through the conductivity cell and into the plumbing loop upon CTD contact with the bottom.

Deployment procedures must be constantly monitored and strict adherence to protocols must be followed to mitigate these and other potential problems. Protocols specified in the *SCBPP Field Operations Manual* must be followed to assure data quality and equipment maintenance.

Specific quality control efforts include:

- Placing the CTD in a water bath on deck or providing some other means to avoid excessive heating of sensors during transit between stations;
- Averaging data during the profile to not more than 24 scans/bin;

- Deploying the CTD at a descent rate not exceeding 1 m/s;
- Prior to each cast, a minimum 3 min soak time at the first station and minimum 90 s soak time at all subsequent stations at the start of a cast. During this equilibration time, the unit shall be lowered to a depth sufficient to purge air from the plumbing line.

5.6. INSTRUMENT DEPLOYMENT CHECKS

Each CTD cast data file shall be reviewed in the field immediately for evidence of problems. This shall include the graphical display or range checks of all parameters of interest (temperature, DO, salinity, transmissivity) versus depth.

5.7. INFORMATION MANAGEMENT

The details of this section are still being worked out and requirements will be specified by the CTD users group and will be conveyed to the information management subcommittee.

5.8. DATA EVALUATION PROCEDURES

CTD data evaluation should consist of the following steps:

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

5.8.1. Data Completeness

The first part of data evaluation is to verify that all required information has been provided in the data package. All CTD data files must be reviewed to assure that all required parameters have been sampled for each station. Each CTD file must be checked to verify that it is associated with the correct station and event. Any CTD file that does not match recorded values should be flagged for investigation.

5.8.2. Data Quality

Data validation, or the process of assessing data quality, can begin after it has been determined that the data package is complete. Each CTD profile must be examined, both manually and via automatic range checks, as part of the validation. Each CTD cast must be visually inspected to identify any unusual patterns or spikes that necessitate further review. Specific parameters which should be checked are:

- Amount of time at the surface should be at least 90 s;
- Stability of dissolved oxygen at the end of the surface soak - readings for the last 30 s prior the downcast should not vary by more than 0.5 mg/L;
- Stability of the salinity values at the beginning and end of the bottom-soak values should not differ by more than 0.3 ppt;
- Unexpected patterns or trends in the downcast (e.g., spikes); and
- Indications that the CTD was lowered into the sediment (large change in oxygen and/or salinity, or a spike in transmissivity values).

In addition to examining the profiles themselves, the following range checks (=control limits) should be conducted (either manually or automatically) on the downcast and bottom soak values:

- Depth acceptable range = 0 -200 m;
- Temperature acceptable range = 10 - 24 °C;
- Salinity acceptable range = 33 - 35 ppt;
- Dissolved Oxygen acceptable range = 3 - 12 mg/L; and
- Light Transmission acceptable range = 20 - 90%.

These are ranges within which our coastal values will likely fall; any value not within these ranges, while not impossible, would be unlikely and should be flagged for investigation. The values and flags should be output in a QA/QC report for each cast.

5.8.3. Data Qualifier Codes

After the above checks are made, a database QA code should be assigned to the cast. These codes describe the acceptability of the different water quality parameters in different sections of the cast.

5.8.4. Final Actions

Upon completion of the above steps, a report summarizing the QA review of the data package should be prepared. 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 written record on data concerns and a documented rationale for why certain data were accepted as estimates or were rejected. The following specific items should be addressed in the QA report:

- Summary of overall data quality, including a description of data that were qualified.
- Summary of all QA data (e.g., field QC checks, calibrations, calibration checks).
- Description of data reporting, including any corrections made for transcription or other reporting errors, and descriptions of data completeness relative to objectives stated in the *QA Project Plan*.

The QA/QC data collected during this pilot project will be used to assess the accuracy and precision of individual measurements, but ultimately to assess the comparability of data generated by multiple laboratories and field crews.

6. MEASUREMENTS OF FISH AND INVERTEBRATE ASSEMBLAGES AND FISH PATHOLOGY

6.1. OVERVIEW

This section presents SCBPP 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 *SCBPP Field Operations Manual* (SCBPP 1994). The field crews will generate data on species identification, enumeration, biomass, length measurements (fish only), and gross external pathology.

Field crews will conduct a “standard” 10-min trawl at selected stations. The *SCBPP 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, counted, weighed, and examined for evidence of gross external pathologies. Demersal invertebrates will be processed in the same way but length will not be measured. Organisms suspected of having pathologies will be fixed in 10% buffered formalin and shipped to SCCWRP. Diseased specimens will be examined by a pathologist.

6.2. QUALITY CONTROL PROCEDURES: FIELD OPERATIONS

6.2.1. 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 be provided with “standard” nets to ensure comparability of gear. The importance of maintaining the trawl duration and speed within established limits should be stressed during the pre-survey protocol calibration meeting. During sampling, crews must record towing speed and trawl duration on the Trawl Cover over Sheet. The Chief Scientist will be responsible for reviewing all trawl data sheets and the boat captain’s log daily and for investigating and correcting any discrepancies.

The Field QA Specialist will monitor adherence to collection methodology during a pre-survey audit of each field crew. During the audit, the Field QA Specialist will ensure that the following trawling procedures are executed correctly: the net is rigged properly, the trawl is deployed and retrieved properly, and the trawl data sheets are accurate and complete. The Field QA Specialist 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%. All of the samples collected (except for repeat trawls for bioaccumulation samples) will be processed, identified, counted, measured (fish only), and weighed.

6.2.2. Species Identification, 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 *SCBPP Field Operations Manual* (SCBPP 1994). The quality of fish and invertebrate identification, enumeration, biomass, and length measurements will be ensured principally through QA/QC audits and demersal fish and invertebrate identification workshops at the Trawl QA Meeting for the Chief Field Taxonomists prior to sampling. The Chief Scientist or Chief Field Taxonomist will be responsible for reviewing the standard sampling procedures with his/her field crew and conducting training as needed. The Trawl QA Specialist(s) will confirm fish and invertebrate identification, enumeration, biomass, and length measurements during pre-survey field audits.

During field QA audits, the QA Specialists 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 procedures in the *SCBPP Field Operations Manual*. The QA Specialists will check the identifications of at least 25% of the species collected during the day and will note the number examined and the number identified incorrectly. The QA Specialists will check the length and weights, and counts and weights, of at least 10 randomly selected species per visit. The QA Specialists will also check the identification of all pathologies during the audit.

A voucher collection of organisms collected in SCBPP trawls will be developed during the survey; the collection will be housed at SCCWRP for one year after the project and archived in a museum. In addition, each organization will be encouraged to develop its own voucher collection. Prior to the survey, each field crew will be given a list of fish and invertebrate species. Each crew will be required to provide at least one representative of every species identified in the field. Qualified taxonomists will verify the species identifications and provide immediate feedback to the field crews, especially when errors are found. All erroneous identifications for a given field crew will be corrected in the database. Extra voucher specimens will be saved to provide a reference collection during training for subsequent years.

To maintain a consistent level of field crew performance, the SCBPP program has established an overall accuracy objective of 95% (i.e., <5% errors) for all fish and invertebrate identifications, and 90% for enumeration, biomass, and length measurements (fish only) in a given sampling season. If this is not met, corrective actions will include increased emphasis on training and more rigorous testing of field crews during the survey.

6.3. QUALITY CONTROL PROCEDURES: GROSS EXTERNAL PATHOLOGY

The field crew must examine all demersal fish and epibenthic invertebrates collected in standard trawls 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 training workshops for Chief

Field Taxonomists and through QA/QC audits during the survey. The Chief Scientist or Chief Field Taxonomist will be responsible for transmitting this knowledge to his/her field crew. 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 SCBPP 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 the proper field identification of pathologies, all definitive examinations will be conducted by a qualified pathologist. Upon receipt of a sample at the pathology laboratory, pathologist will examine the organisms and provide the Trawl QA Specialist with the results.

Each laboratory should also maintain a reference collection of preserved specimens or photographs that represent every type of pathological condition identified in the SCBPP fish and invertebrates. Each of these examples should be verified by an external pathologist experienced with the species in question. The reference collection will be used to verify the diagnoses made in future years to ensure intralaboratory consistency. The reference collections will also be compared with those of other laboratories to ensure interlaboratory consistency. A reference collection will also be developed for future training purposes.

7. ANALYSIS OF CHEMICAL CONTAMINANTS IN SEDIMENTS AND TISSUES

7.1. OVERVIEW

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

The SCBPP measures a variety of organic and inorganic contaminants in marine sediment and fish tissue samples (Table 7.1). SCBPP requires that laboratories demonstrate comparability continuously through strict adherence to common QA/QC procedures, routine analysis of Certified Reference Materials, and regular participation in interlaboratory comparison exercises (round-robins). This is a "performance-based" approach for quality assurance of low-level contaminant analyses, involving continuous laboratory evaluation through the use of accuracy-

based materials (e.g., CRMs), laboratory fortified sample matrices, laboratory reagent blanks, calibration standards, and laboratory replicates. No single analytical

method has been approved officially for low-level (i.e., low ppb) analysis of organic and inorganic contaminants in marine sediments and fish tissue. Under the SCBPP performance-based chemistry QA program, laboratories are not required to use a single, standard analytical method for each type of analysis, but rather are free to choose the best or most feasible method within the constraints of cost and equipment provided that the resulting data would be of known and documented quality.

Table 7.1
Constituents that will be measured in marine sediments and fish tissues
by laboratories participating in the Southern California Bight Pilot Project.

<u>CONSTITUENT</u>	<u>SEDIMENT</u>	<u>TISSUE</u>	<u>CONSTITUENT</u>	<u>SEDIMENT</u>	<u>TISSUE</u>
Acenaphthene	Yes	No	Aldrin	Yes	Yes
Acenaphthylene	Yes	No	α -Chlordane	Yes	Yes
Anthracene	Yes	No	Dieldrin	Yes	Yes
Benz[a]anthracene	Yes	No	Endosulfan	Yes	Yes
Benzo[b]fluoranthene	Yes	No	Endrin	Yes	Yes
Benzo[k]fluoranthene	Yes	No	Heptachlor	Yes	Yes
Benzo[ghi]perylene	Yes	No	Heptachlor epoxide	Yes	Yes
Benzo[a]pyrene	Yes	No	Heptachlor benzene	Yes	Yes
Chrysene	Yes	No	Lindane	Yes	Yes
Dibenz[ah]anthracene	Yes	No	Mirex	Yes	Yes
Fluoranthene	Yes	No	Trans-nonachlor	Yes	Yes
Fluorene	Yes	No	Antimony	Yes	No
Indeno(1,2,3-cd)pyrene	Yes	No	Arsenic	Yes	No
Naphthalene	Yes	No	Cadmium	Yes	No
Phenanthrene	Yes	No	Chromium	Yes	No
Pyrene	Yes	No	Copper	Yes	No
Aroclors ¹	Yes	No	Lead	Yes	No
PCB Congeners ²	Yes ³	Yes	Mercury	Yes	No
2,4'-DDT	Yes	Yes	Nickel	Yes	No
4,4'-DDT	Yes	Yes	Selenium	Yes	No
2,4'-DDE	Yes	Yes	Silver	Yes	No
4,4'-DDE	Yes	Yes	Zinc	Yes	No
2,4'-DDD	Yes	Yes	Total organic carbon	Yes	No
4,4'-DDD	Yes	Yes	Lipids	No	Yes
			Sediment grain size	Yes	--

¹Aroclors 1242, 1254, 1260

²Congeners 8, 18, 28, 29, 44, 50, 52, 66, 77, 87, 101, 104, 105, 118, 126, 128, 138, 153, 154, 170, 180, 187, 188, 195, 201, 206, 209

³Congeners will be measured on a subset of sediment samples.

7.2. QUALITY CONTROL PROCEDURES: SAMPLE COLLECTION, PRESERVATION, AND HOLDING

Field personnel must strictly adhere to SCBPP protocols to insure the collection of representative, uncontaminated sediment and fish tissue chemistry samples. These sample collection protocols are described in detail in the SCBPP Field Operations Manual. Briefly, the key aspects of quality control associated with chemistry sample collection are as follows:

- Field personnel must be thoroughly trained in the proper use of sample collection gear and must be able to distinguish acceptable versus unacceptable sediment grab samples or fish trawls in accordance with pre-established criteria;
- Field personnel must be thoroughly trained to recognize and avoid potential sources of sample contamination (e.g., engine exhaust, winch wires, deck surfaces, ice used for cooling);
- Samplers and utensils which come in direct contact with the sample should be made of non-contaminating materials (e.g., glass, high-quality stainless steel and/or Teflon®) and should be thoroughly cleaned between sampling stations;
- Sample containers should be of the recommended type (Table 7.2) and must be free of contaminants (i.e., carefully pre-cleaned); and
- Conditions for sample collection, preservation and holding times should be followed (Table 7.2).

Table 7.2
Summary of chemistry sample collection and holding time
conditions for the SCBPP

<u>Sediment</u> <u>Parameter</u>	<u>Container</u> <u>Type</u>	<u>Sample size</u> <u>Size (g)</u>	<u>Preservation</u> <u>Requirements</u>	<u>Maximum</u> <u>Holding Time</u>
Sediment grain size	plastic or glass (4 oz)	100 (80% full)	cool (4°C)	28 days
Total organic carbon	plastic or glass (4 oz)	100 (80% full)	frozen (-20°C)	6 months
Trace metals	plastic or glass (8 oz)	200 (80% full)	frozen (-20°C)	6 months
Trace organics	glass (8 oz)	200 (80% full)	frozen (-20°C)	6 months
<u>Tissue</u> <u>Parameter</u>	<u>Container</u> <u>Type</u>	<u>Sample size</u> <u>Size</u>	<u>Preservation</u> <u>Requirements</u>	<u>Maximum</u> <u>Holding Time</u>
Trace organics	water tight plastic bags	whole fish	frozen (-20°C)	1 year

7.3. QUALITY CONTROL PROCEDURES: LABORATORY OPERATIONS

7.3.1. Overview

The SCBPP will involve the distribution of chemistry samples to several different laboratories. Each participating laboratory will analyze samples using existing methodology and report results only for the constituents that match those listed in Table 7.1.

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

The performance-based SCBPP QA program for analytical chemistry laboratories consists of two basic elements:

- initial demonstration of laboratory capability (e.g., performance evaluation);
- ongoing demonstration of capability.

Prior to the analysis of samples, each laboratory must demonstrate proficiency by: submitting written protocols for the analytical methods that will be used for sample analysis to the Lab Coordinator for review; calculating method detection limits for each analyte; establishing an initial calibration curve for all analytes; and demonstrating 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; calibration checks; and analysis of laboratory reagent blanks and fortified samples. These steps are detailed in the following sections.

The results for the various QA/QC samples should be reviewed by laboratory personnel immediately following the analysis of each sample batch. These results should then be used to determine when warning and control limit criteria have not been met and corrective actions must be taken, before processing a subsequent sample batch. When warning limit criteria have not been met, the laboratory is not obligated to halt analyses, but the analyst(s) is advised to investigate the cause. When control limit criteria are not met, specific corrective actions are required before the analyses may proceed. Warning and control limit criteria and recommended frequency of analysis for each QA/QC element or sample type required in the SCBPP program also are summarized in Table 7.3.

Table 7.3
Summary of the data quality requirements for the
SCBPP chemistry measurements

<u>MEASUREMENT</u>	<u>FREQUENCY</u>	<u>CONTROL LIMIT</u>
<u>Instrument calibration</u>		
Trace metals	1-3/batch	15% on average for initial calibration curve, 25% for each analyte
Trace organics	1-3/batch	15% on average for initial calibration curve, 25% for each analyte
Total organic carbon	1-3/batch	15% on average for initial calibration curve, 25% for each analyte
<u>CRM or LCM</u>		
Trace metals	1/batch	20% of certified value for analytes >10 times MDL; precision should be <30% RSD of analytes >10 times MDL for all batches combined
Trace organics	1/batch	30% of certified value for analytes >10 times MDL; precision should be <30% RSD of analytes >10 times MDL for all batches combined
Total organic carbon	1/batch	15% of certified value; precision should be <20% RSD for all batches combined
<u>Lab reagent blanks</u>		
Trace metals	1/batch	No analyte should be detected at >3 times its MDL
Trace organics	1/batch	No analyte should be detected at >3 times its MDL
Total organic carbon	1/batch	No analyte should be detected at >3 times its MDL
<u>Recovery surrogates</u>		
Trace metals	1/sample	30-150%
Trace organics	1/sample	30-150%
Total organic carbon	NA	NA
<u>Internal standards</u>		
Trace metals	1/sample	Lab develop their own
Trace organics	1/sample	Lab develop their own
Total organic carbon	NA	NA
<u>Matrix spikes/MS duplicate</u> (Note: spike should contain all target analytes at 10 times MDL)		
Trace metals	1/batch	Recovery should be 50-120% for 80% of the analytes; relative difference <30%
Trace organics	1/batch	Recovery should be 50-120% for 80% of the analytes relative difference <30%
Total organic carbon	1/batch	Recovery should be 50-120% for 80% of the analytes relative difference <20%
<u>Duplicates</u>		
Trace metals	1/batch	Relative difference <30%
Trace organics	NA	NA
Total organic carbon	1/batch	Relative difference <30%

7.3.2. Instrument Calibration

Equipment should be calibrated prior to the analysis of each sample batch, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended control limit criteria (Table 7.3). All calibration standards should be traceable to a recognized organization for the preparation and certification of QA/QC materials (e.g., National Institute of Standards and Technology, US EPA, etc.). Calibration curves must be established for each constituent and a minimum of three analytical standards of increasing concentration, covering the range of expected sample concentrations. The calibration curve should be well characterized and must be established prior to the analysis of samples. Only data which results from quantification within the demonstrated working calibration range may be reported by the laboratory (i.e., quantification based on extrapolation is not acceptable). Samples outside the calibration range should be diluted or concentrated, as appropriate, and reanalyzed.

7.3.3. Initial Documentation of Method Detection Limits

In the SCBPP program, the Method Detection Limit (MDL) will be used to define the analytical limit of detectability. 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." Confidence in the apparent analyte concentration increases as the analyte signal increases above the MDL.

Each SCBPP analytical laboratory must calculate and report an MDL for each analyte of interest in each matrix of interest (sediment or tissue) prior to the analysis of field samples for a given year. Each laboratory is required to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate MDLs for each analytical method employed. Briefly, seven replicates of each representative matrix should be spiked at a concentration between one and five times the estimated detection limit. The samples are then processed completely through the entire analytical method. The matrix and the amount of sample (i.e., weight of sediment or tissue) used in calculating the MDL should match as closely as possible the matrix of the actual field samples and the amount of sample typically used. Calculate the variance and standard deviation of the replicates and compute the MDL by multiplying the standard deviation by the t value for the 99% confidence interval (for $n=7$, $t=3.143$). Each laboratory must periodically (at least once each year) evaluate its MDLs for the analytical methods used and the sample matrices typically encountered.

7.3.4. Initial Demonstration of Ability: Blind Analysis of a Representative Sample

A representative sample matrix which is not compromised, homogeneous and contains the analytes of interest at concentrations of interest will be provided to

each analytical laboratory new to the SCBPP program; this sample will be used to evaluate laboratory performance prior to the analysis of field samples. The sample used for this initial demonstration of laboratory capability typically will be distributed blind (*i.e.*, the laboratory will not know the concentrations of the analytes of interest) as part of the interlaboratory comparison exercises. Based on results that have typically been attained by experienced NS&T laboratories, a new laboratory's performance generally will be considered acceptable if its submitted values are within $\pm 30\%$ (for organic analyses) and $\pm 20\%$ (for inorganic analyses) of the known concentration of each analyte of interest in the sample. These criteria apply only for analyte concentrations equal to or greater than 10 times the MDL established by the laboratory. If the results for the initial analysis fail to meet these criteria, the laboratory will be required to repeat the analysis until the performance criteria are met, prior to the analysis of real samples.

7.3.5. Continuing Demonstration of Ability

7.3.5.1. Routine Analysis of Certified Reference Materials or Laboratory Control 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). Certified Reference Materials 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 agency using two independent measurement techniques for verification. In addition, the certifying agency 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 agency. A list of reference materials commonly used by EMAP-E laboratories is presented in Table 7.4.

7.3.5.2. Laboratory Control Material (LCM)

A Laboratory Control Material is similar to a Certified Reference Material in that it is a homogeneous matrix that closely matches the samples being analyzed. A "true" LCM is prepared (*i.e.*, collected, homogenized and stored in a stable condition) strictly for use in-house by a single laboratory. Alternately, the material may be prepared by a central laboratory and distributed to others (so-called regional or program control materials). Unlike CRMs, concentrations of the analytes of interest in LCMs are not certified but are based upon a statistically valid number of replicate analyses by one or several laboratories. In practice, this material can be used to assess the precision (*i.e.*, consistency) of a single laboratory, as well as to determine the degree of comparability among different laboratories. If available, LCMs may be preferred for routine (*i.e.*, day to day) analysis because CRMs are relatively expensive. However, CRMs still must be analyzed at regular intervals (*e.g.*, monthly or quarterly) to provide a check on accuracy.

Table 7.4
Certified Reference Materials commonly used by EMAP-E laboratories.
SRMs are available from NIST (301/975-6776); all other reference materials
are available from NRC (613/993-2359).

Calibration solutions

SRM 1491	Aromatic hydrocarbons in hexane/toluene
SRM 1492	Chlorinated pesticides in hexane
SRM 1493	Chlorinated biphenyl congeners in 2,2,4-trimethylpentane
SRM 2260	Aromatic hydrocarbons in toluene
SRM 2261	Chlorinated pesticides in hexane
SRM 2262	Chlorinated biphenyl congeners in 2,2,4-trimethylpentane

Environmental matrices (organics)

SRM 1941a	Organics in marine sediment
SRM 1974	Organics in mussel tissue (<i>Mytilus edulis</i>)

Environmental matrices (inorganics)

SRM 1646	Estuarine sediment
MESS-1	Estuarine sediment
BEST-1	Marine sediment
BCSS-1	Marine sediment
PACS-1	Harbor sediment
DOLT-1	Dogfish liver
DORM-1	Dogfish muscle
SRM 1566a	Oyster tissue

7.3.5.3. Routine Analysis of CRMs or LCMs

Routine analysis of Certified Reference Materials or, when available, Laboratory Control Materials, is a vital aspect of the "performance-based" SCBPP QA philosophy. At least one CRM or LCM must be analyzed along with each batch of 25 or fewer samples (Table 7.3). For CRMs, both the certified and non-certified concentrations of the target analytes should be known to the analyst(s) and should be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs or LCMs (Table 7.3). If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM or LCM, the data for the entire batch of samples is suspect.

Calculations and instruments should be checked; the CRM or LCM may have to be reanalyzed (i.e., reinjected) to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample

processing. The results of the CRM or LCM analysis should never be used by the laboratory to "correct" the data for a given sample batch.

7.3.5.4. Precision criteria

Each laboratory is expected to maintain control charts for use by analysts in monitoring the overall precision of the CRM or LCM analyses. Upper and lower control chart limits (e.g., warning limits and control limits) should be updated at regular intervals; control limits based on 99% confidence intervals around the mean are recommended. Following the analysis of all samples in a given year, an RSD (relative standard deviation or coefficient of variation) will be calculated for each analyte of interest in the CRM. Based on typical results obtained by experienced analysts, an overall RSD of <30% will be considered acceptable precision for each analyte having a CRM concentration >10 times the laboratory's MDL. Failure to meet this goal will result in a thorough review of the laboratory's control charting procedures and analytical methodology to determine if improvements in precision are possible.

7.3.5.5. Accuracy criteria

The "absolute" accuracy of an analytical method can be assessed using CRMs only when certified values are provided for the analytes of interest. However, the concentrations of many analytes of interest to SCBPP are provided only as non-certified values in some of the more commonly used CRMs. Therefore, control limit criteria are based on "relative accuracy", which is evaluated for each analysis of the CRM or LCM by comparison of a given laboratory's values relative to the "true" or "accepted" values in the LCM or CRM. In the case of CRMs, this includes both certified and noncertified values and encompasses the 95% interval for each value as described in Table 7.3.

Based on typical results attained by experienced analysts in the past, accuracy control limit criteria have been established both for individual compounds and combined groups of compounds (Table 7.3). There are two combined groups of compounds for the purpose of evaluating relative accuracy for organic analyses: PAHs and PCBs/pesticides. The laboratory's value should be within $\pm 30\%$ of the true value on average for each combined group of organic compounds, and the laboratory's value should be within $\pm 35\%$ of either the upper or lower 95% confidence limit for at least 70% of the individual compounds in each group. For inorganic analyses, the laboratory's value should be within $\pm 20\%$ of either the upper or lower 95% confidence limit for each analyte of interest in the CRM. Due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy only apply to analytes having CRM true values that are >10 times the MDL established by the laboratory.

7.3.6. Continuing Calibration Checks

The initial instrument calibration performed prior to the analysis of each batch of samples is checked through the analysis of calibration check samples (i.e.,

calibration standard solutions) inserted as part of the sample stream. Calibration standard solutions used for the continuing calibration checks should contain all the analytes of interest. It is recommended that analysis of the calibration check solution should occur somewhere in the middle and at the end of each sample batch. Analysts should use best professional judgment to determine if a different frequency of calibration checks are necessary or desirable.

If the control limit for analysis of the calibration check standard is not met (Table 7.3), the initial calibration will have to be repeated. If possible, the samples analyzed before the calibration check sample that failed the control limit criteria should be reanalyzed following the recalibration. The laboratory should begin by reanalyzing the last sample analyzed before the calibration standard which failed. If the relative percent difference (RPD) between the results of this reanalysis and the original analysis exceeds 30%, the instrument is assumed to have been out of control during the original analysis. If possible, reanalysis of samples should progress in reverse order until it is determined that there is less than 30% RPD between initial and reanalysis results. Only the re-analysis results should be reported by the laboratory. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control check) is suspect. In this case, the laboratory should prepare a narrative explanation to accompany the submitted data.

7.3.7. Laboratory Reagent Blank

Laboratory reagent blanks (also called method blanks or 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 reagent blank should be processed through the entire analytical procedure in a manner identical to the samples. Warning and control limits for blanks (Table 7.3) are based on the laboratory's method detection limits as documented prior to the analysis of samples. A reagent blank concentration between the MDL and three times the MDL for one or more of the analytes of interest should serve as a warning limit requiring further investigation based on the best professional judgment of the analyst(s). 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.

7.3.8. Recovery Surrogates

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 recovery surrogate**. 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.

Control limit criteria for surrogate recoveries are provided in Table 7.3. Each laboratory should set its own warning 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).

7.3.9. Internal Standards

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

7.3.10. Matrix Spike and Matrix Spike Duplicate

A laboratory fortified sample matrix (commonly called a matrix spike or MS) and a laboratory fortified 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 5% of the total number of samples submitted to the laboratory in a given year should be selected at random for analysis as matrix spikes/matrix spike duplicates. Each MS/MSD sample is first homogenized and then split into three subsamples. Two of these subsamples are fortified with the matrix spike solution and the third subsample is analyzed as is to provide a background concentration for each analyte of interest. The matrix spike solution should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be at least 10 times the MDL for that analyte, as previously calculated by the laboratory.

Recovery data for the fortified compounds ultimately will provide a basis for determining the prevalence of matrix effects in the sediment samples analyzed during the project. If the percent recovery for any analyte in the MS or MSD is less than 50%, the recommended warning limit, the chromatograms and raw data

quantitation reports should be reviewed. If an reason for a low percent recovery value is not discovered, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interferences 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 relative percent difference (RPD) between the MS and MSD results should be <30 for each analyte of interest (Table 7.3). The RPD 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.

If results for any analytes do not meet the RPD <30% control limit criteria, calculations and instruments should be checked. A repeat analysis may be required to confirm the results. Results which repeatedly fail to meet the control limit criteria indicate poor laboratory precision. In this case, the laboratory is obligated to halt the analysis of samples and eliminate the source of the imprecision before proceeding.

7.4. QUALITY CONTROL PROCEDURES: OTHER SEDIMENT PARAMETERS

The SCBPP laboratories will measure total organic carbon (TOC) and sediment grain size in addition to "conventional" contaminants. The laboratory QA/QC requirements for these sediment measurements are presented in the following sections.

7.4.1. Total Organic Carbon

As a check on precision, the laboratory should analyze at least one total organic carbon (TOC) sample in duplicate for each batch of 25 or fewer samples. Based on typical results attained by experienced analysts, the relative percent difference (RPD) between the two duplicate measurements should be <20%. If this control limit is exceeded, analysis of subsequent sample batches should stop until the source of the discrepancy is determined and the system corrected.

At least one certified reference material (CRM) or, if available, one laboratory control material (LCM) should be analyzed along with each batch of 25 or fewer TOC samples. Any one of the marine sediment CRMs distributed by the National Research Council of Canada's Marine Analytical Chemistry Standards Program (e.g., BCSS-1, MESS-1, and PACS-1; Table 7.4) have

certified concentrations of total carbon and are recommended for this use. Prior to analysis of actual samples, it is recommended that the laboratory perform several total organic carbon analyses using a laboratory control material or one of the aforementioned CRMs to establish a control chart (the values obtained by the laboratory for total organic carbon should be slightly less than the certified value for total carbon in the CRM). The control chart should then be used to assess laboratory precision for subsequent analyses of the LCM or CRM with each sample batch.

In addition, a method blank should be analyzed with each sample batch. Total organic carbon concentrations should be reported as mg/g (ppm) dry weight of the unacidified sediment sample. Data reported for each sample batch should include QA/QC sample results (duplicates, CRMs or LCMs, and method blanks). Any factors that may have influenced data quality should be discussed in a cover letter accompanying the submitted data.

7.4.2. Sediment Grain Size

As a check on precision, the laboratory should analyze at least one sediment grain size sample in duplicate for each batch of 25 or fewer samples. Based on typical results attained by experienced analysts, the relative percent difference (RPD) between the two duplicate measurements should be <20%. If this control limit is exceeded, analysis of subsequent sample batches should stop until the source of the discrepancy is determined and the system corrected.

7.5. QUALITY CONTROL PROCEDURES: INFORMATION MANAGEMENT

7.5.1. Sample Tracking

The SCBPP information management personnel have developed a comprehensive system for labeling sample containers, recording sampling information in the field, and tracking sample shipments. A complete description of this system is provided in the *SCBPP Information Management Plan*. Each analytical laboratory must designate a sample custodian who is authorized to check the condition of and sign for incoming field samples, obtain documents of shipment, and verify sample custody records. This individual is required, upon receipt of samples, to record and transmit all tracking information to the Information Management Center (SCCWRP). Laboratory personnel should be aware of the required sample holding times and conditions (Table 7.2), and the laboratory must have clearly defined and documented custody procedures for sample handling, storage, and disbursement.

7.5.2. Data Reporting Requirements

Laboratory personnel must verify that the measurement process was "in control" (i.e., all specified QA/QC requirements were met) for each batch of samples before proceeding with the analysis of a subsequent batch. In addition, each laboratory must establish a system for detecting and eliminating

transcription and/or calculation errors prior to reporting data. It is recommended that an individual not involved directly in sample processing be designated as laboratory QA Officer to perform these verification checks independent of day-to-day laboratory operations.

Only data that has met QA requirements should be submitted by the laboratory. When QA requirements have not been met, the samples should be reanalyzed and only the results of the reanalysis should be submitted, provided they are acceptable. Each data package should consist of the following:

7.5.2.1. Cover letter

A cover letter, both hard copy and in electronic file format, should provide a brief description of the procedures and instrumentation used (including the procedure(s) used to calculate MDLs), as well as a narrative explanation of analytical problems (if any) or failure(s) to meet quality control limits.

7.5.2.2. Hard copy of tabulated results

Tabulated results in hard copy should include sample size, wet weight, dry weight, and concentrations of the analytes of interest (reported in units identified to three significant figures unless otherwise justified). Concentration units should be ng/g dry weight for sediment and ng/g wet weight for tissue. The results should be checked for accuracy and the report signed by the laboratory manager or designee.

7.5.2.3. Computer file of tabulated results

Tabulated results in computer-readable form (e.g., diskette) should be included in the same shipment as the hard copy, but packaged in a diskette mailer to prevent damage. There are two acceptable formats for computer-readable data, descriptions of which are available from the SCBPP Information Management Officer (Robert Hall): 1) ASCII text files in a format specified by the SCBPP Information Management Officer, or 2) any format agreed upon by the submitting laboratory and the SCBPP Information Management Officer. If data are not delivered in one of these formats, the data package will be considered incomplete and will not be accepted.

7.5.2.4. Method detection limits

Tabulated method detection limits achieved for the samples should be included in the data package.

7.5.2.5. QA/QC results

Results for all QA/QC samples (e.g., CRMs, calibration check samples, blanks, matrix spike/matrix spike duplicates, etc.) must be submitted by the laboratory as part of the data package for each batch of samples analyzed. The

laboratory must provide a "batch number" as a way to link samples from a given batch or analytical set with their accompanying QA/QC samples.

7.5.2.6. Data qualifier codes

Laboratories are responsible for assigning only two data qualifier codes or "flags" to the submitted data. If an analyte is not detected, the laboratory should report the result as *ND*. The detection limit (MDL) is reported as a separate variable.

7.5.2.7. Factors affecting data quality

There may be a limited number of situations where sample re-analysis is not possible or practical (i.e., minor violation of a single control limit criterion). The laboratory is expected to provide a detailed explanation of any factors affecting data quality or interpretation; this explanation should be in the form of a cover letter, both on paper and in electronic file format (i.e., text file) accompanying each data package submitted. Depending on the nature of the narrative explanations, the SCBPP program will develop a limited list of codes for qualifying data in the database.

7.5.3. Data Evaluation Procedures

It is the responsibility of the Project Manager or his designee to acknowledge initial receipt of the data package(s), verify that the four data evaluation steps (see below) are completed, notify the analytical laboratory of any additional information or corrective actions deemed necessary after the data evaluation, and, following satisfactory resolution of all "corrective action" issues, take final action by notifying the laboratory in writing that the submitted results have been officially accepted as complete. It may be necessary or desirable for a team of individuals (e.g., the QA Coordinator, Lab Coordinator and/or staff analytical chemists) to assist the Project Manager in technical evaluation of the submitted data packages. While the Project Manager has ultimate responsibility for maintaining official contact with the analytical laboratory and verifying that the data evaluation process is completed, it is the responsibility of the QA Coordinator to closely monitor and formally document each step in the process as it is completed. This documentation should be in the form of a data evaluation tracking form or checklist that is filled in as each step is completed. This checklist should be supplemented with detailed memos to the project file outlining any concerns with data omissions, analysis problems, or descriptions of questionable data identified by the laboratory.

Evaluation of the data package should begin as soon as possible following its receipt, since delays increase the chance that information may be misplaced or forgotten and (if holding times have been exceeded) can sometimes limit options for reanalysis. The following steps are to be followed and documented in evaluating SCBPP chemistry data:

- 1) Checking data completeness (verification)

- 2) Assessing data quality (validation)
- 3) Assigning data qualifier codes
- 4) Taking final actions

7.5.3.1. Checking Data Completeness

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

- 1) 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.
- 2) The electronic data file(s) should be parsed and entered into the SCBPP database to verify that the correct format has been supplied.
- 3) Once the data have been entered into the SCBPP database, automated checks should be run to verify that results have been reported for all expected samples and all analytes.

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

7.5.3.2. Assessing Data Quality

Data validation, or the process of assessing data quality, can begin after SCBPP 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, SCBPP laboratories are expected to submit data that has been tabulated and checked 100% for accuracy; the raw data reports needed to perform these checks (e.g., chromatograms, original quantitation reports) are not submitted as part of the data package. The laboratory is required to maintain this raw data in an orderly manner and to have these records available for review by SCBPP personnel upon request. The first-step validation checks performed by SCBPP 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 SCBPP (e.g., PAHs, PCBs, DDTs and other chlorinated pesticides) typically occur in the environment in more or less fixed ratios to one another. For example, the DDT breakdown products p,p-DDD and p,p-DDE typically occur at higher concentrations than p,p-DDT in marine sediments in off Southern California. If anomalous departures from expected ratios are found, it may indicate a problem in the measurement or data reduction, which requires further investigation.

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

- 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 laboratory control material(s) and matrix spike/matrix spike duplicate samples. The tables should indicate the percent recovery values for each individual batch of samples, as well as the average, standard deviation, coefficient of variation, and range for all batches combined.
- 2) Similar summary tables should be prepared for the laboratory reagent blank QA/QC samples.
- 3) The summary results, particularly those for the laboratory control material (i.e., Certified Reference Material), should be evaluated by comparing them against the QA/QC warning and control limit criteria for accuracy, precision, and blank contamination specified in Table 7.3.
- 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:

- 1) The laboratory's cover letter (narrative explanation) should be consulted to determine if the problems were satisfactorily addressed.

- 2) 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).

7.5.3.3 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. SCBPP data reviewers will assign data qualifier codes in situations where there are violations of control limit criteria. The most typical situation is when a laboratory fails to meet the accuracy control limit criteria for a particular analyte in a Certified Reference Material or matrix spike sample. In these situations, the QA reviewer should verify that the laboratory did meet the control limit criteria for precision. If the lack of accuracy is found to be consistent (i.e., control limit criteria for precision were met), then it is likely that the laboratory experienced a true bias for that particular analyte. In these situations, all reported values for that particular analyte will be qualified with a code that has the following meaning: "The reported concentration is considered an estimate because control limits for this analyte were exceeded in one or more quality control samples."

Because some degree of expert judgment and subjectivity typically is necessary to evaluate chemistry QA/QC results and assign data qualifier codes, data validation will be conducted only by qualified personnel. It is the philosophy of the SCBPP 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.

7.5.3.4. 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 Certified Reference Materials and matrix spike/matrix spike duplicates).

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

8. MACROBENTHIC COMMUNITY ASSESSMENT

8.1. OVERVIEW

This section provides the SCBPP QA/QC protocols and requirements for the production of biological data, from sample collection through taxonomic analysis, that will be used in the assessment of benthic infaunal communities. Single benthic samples are collected at each station in the survey. Each sample is screened and fixed in the field, returned to one of four participating laboratories, and analyzed for species composition, abundance, and major taxa biomass. The data produced by each laboratory will be aggregated into a single data set and made available for data analysis and interpretation.

8.2. QUALITY ASSURANCE AND CONTROL PROCEDURES: SAMPLE COLLECTION, PRESERVATION, AND HOLDING

Sediment samples for benthic infaunal analysis will be collected at each station using a SCCWRP-modified 0.1 m² Van Veen grab (Stubbs *et al.* 1987). The participation of several different vessels and field sampling teams in the SCBPP requires that uniform procedures be followed in the field to ensure high quality samples and consistent results. Field personnel will be provided with the *SCBPP Field Operations Manual* (SCBPP 1994) and instruction on sampling procedures, application of sample acceptance criteria, sample processing, and use of field data forms. All personnel are expected to understand and properly carry out all steps in the collection, screening, relaxation, and fixation of infaunal samples, and the subsampling and handling of sediment chemistry and

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

A Measurement Quality Objective (MQO) of 90% has been established for completeness of the field collection of benthic samples. This completeness goal was established in an attempt to derive the maximum statistical power of the sampling design. The MQO was not set at 100% in recognition that the randomized selection of sampling sites employed in the SCBPP is likely to result in the selection of some sites where Van Veen grab sampling will be difficult or impossible. Nevertheless, field crews are expected to strive to meet or exceed this MQO. To this end, site acceptability criteria and relocation procedures are provided in Section 7, and sample acceptability criteria and minimum sampling effort are stipulated in Section 9 of the SCBPP Field Operations Manual. As many as nine attempts at a site must be made to meet the site acceptability criteria. Once a site has been accepted, a minimum sampling effort of four attempts to collect an acceptable sample is required at each station.

Sample acceptability criteria have been established (SCBPP 1994) based on sample condition and depth of penetration of the grab. An acceptable grab is characterized by an even surface with minimal disturbance and little or no leakage of overlying water, and a penetration depth of at least 5 cm, if the target depth of 8 cm cannot be achieved. Samples not meeting these criteria are rejected.

In the laboratories, samples will be stored in a safe and secure manner protected from environmental extremes. Exposure to temperatures above 30°C should be avoided so as to retard evaporative loss. Do not refrigerate samples containing formaldehyde as paraformaldehyde will be formed at lower temperatures. Samples are to be transferred from fixative (borate-buffered 10% formalin) to preservative (70% ethanol) after 72 hr (but within two weeks) of collection. When transferring, thoroughly wash the fixative from the sample, using a 0.5 mm (or smaller) mesh screen to avoid specimen loss. Stored samples must be periodically inspected to assure that the closure is tight and the preservative level adequate. If evaporative loss of preservative is evident, top-off the sample using 100% ethanol.

8.3. QUALITY ASSURANCE AND CONTROL PROCEDURES: LABORATORY OPERATIONS

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

8.3.1. Sample Sorting

Quality control of sorting is essential to assure the value of all the subsequent steps in the sample analysis process. Sample material is sorted into six taxa lots: annelids, mollusks, arthropods, ~~ophiurans~~, **ophiuroids**, miscellaneous echinoderms, and “other phyla”. A standard sorting form is used for tracking the sample. It includes the name of the **laboratory and** technician responsible, time required for sorting, **number of taxa lots and sample containers, and** comments. ~~and re-sorting results.~~ Re-sorting of samples is employed for quality control of sorting. Each laboratory participating in the SCBPP has an existing re-sorting protocol for this purpose. All share a minimum re-sorting effort of 10% of the material sorted with a minimum acceptable removal efficiency of 95%, the equivalent of an accuracy MQO of 5%. Two approaches are used for re-sorting. In one, a 10% aliquot of every sample processed by a sorter is resorted. In the other, 10% of the samples processed by a sorter are completely resorted. In both cases, all re-sorting is conducted by an experienced sorter other than the original sorter. For the SCBPP, 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:

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

If sorting efficiency is greater than 95%, no action is required. Sorting efficiencies below 95% will require re-sorting of all samples sorted by that technician and continuous monitoring of that technician until efficiency is improved. **Actions taken are to be described on the Quality Control Report section of the Sorting form and the report signed by the responsible supervisor.** Organisms found in the resort should be added to the original data sheet and, if of significant biomass, included in the sample biomass estimation. Once all quality control criteria for sample sorting have been met, the sample debris may be discarded.

8.3.2. Biomass Estimation

Calibration checks of the balances used for biomass should be performed using a standard set of reference weights. Wet-weight biomass is measured for six taxa lots: annelids, mollusks, arthropods, ophiurans, miscellaneous echinoderms, and "other phyla". Weights are **measured to the nearest 0.01 gram and** reported to the nearest 0.1 gram, as recommended in EPA's 301(h) guidance document (Tetra Tech 1986). **Both measured and reported values are recorded on the Biomass form.** Procedures are provided in the Infaunal Sample Analysis Laboratory Manual. Results are reported on standardized biomass forms, along with technician's name **and any** comments. ~~and re-weighing results.~~

Quality control of Biomass estimation is provided by re-weighing of the taxa lots from 10% of the samples processed by each laboratory. **Only taxa lots for which reported biomass is >0.1 gram are re-weighed.** If samples are weighed by more than one technician, then 10 % of each technician's samples are re-weighed. The re-weighing process is conducted by a person other than the original technician. Weighing efficiency is calculated **based upon the measured net weight** using the following formula:

$$\frac{\text{Original final measured net weight}}{\text{Reweighed final measured net weight}} \times 100$$

An MQO of 10% has been established for the precision of biomass estimation. If precision is between 95% and 105%, no action is necessary. If precision is between either 90% to 95% or 105% to 110%, the sample has met the MQO, but technician should be provided corrective instruction. If the weighing precision falls below 90% or above 110%, the sample has failed and **the previous five samples weighed by that technician must be re-weighed. If any of these samples fail, then** all samples weighed by that technician must be re-weighed. Corrections to the original data sheet should only be made in those cases where precision is less than 90% or greater than 110%. Results of the QC re-weighing **and any actions taken** are reported on the biomass forms. ~~Biomass Quality Control Report and signed by the responsible supervisor.~~

8.3.3. Quality Assurance of Taxonomic Analysis

The goal of taxonomic analysis for the SCBPP is species level identification of all macrobenthic organisms collected and an accurate count of each species. This task is complicated by the participation of four 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 the SCBPP to provide an important element of quality assurance for this aspect of the project. SCAMIT is a regional

organization of taxonomists, many of whom are primarily involved in infaunal monitoring studies of wastewater impacts within the southern California Bight. SCAMIT was founded in 1982 with the goals of promoting the study of marine invertebrate taxonomy and developing a regionally standardized taxonomy for use in environmental monitoring studies. Activities center on cooperation and communication among the region's taxonomists, sharing of expertise, and monthly workshops. Results of the workshops and other information is communicated to the membership through a monthly newsletter. Participation in SCAMIT's activities is an element common to the existing monitoring QA/QC procedures of all four laboratories participating in the SCBPP.

SCAMIT's cooperation includes the provision of standards for nomenclature use and a mechanism for mutual assistance and exchange of information among the taxonomists involved in the SCBPP. The taxonomic nomenclature used in the SCBPP follows the SCAMIT hierarchical species listing (SCAMIT 1994). This list represents a consensus for standard usage of taxa names in POTW monitoring programs in the Bight. In addition, SCAMIT protocols for the use of open nomenclature (SCAMIT 1986) are followed. Taxonomists from the participating laboratories are required to participate in special SCAMIT/SCBPP workshops prior to the sampling period that focus on the taxonomy of groups requiring particular review to promote uniform treatment in the upcoming survey. Pre-survey workshops consider nemertea, platyhelminths, and other groups. The workshops provide training, pooling of regional resources, and designation of the local expert(s) to be called upon for assistance during sample analysis.

After sample analysis has begun, SCAMIT/SCBPP workshops continue at least monthly to address taxonomic problems arising during analysis of the SCBPP samples. A process for integrating these workshops into the sample analysis process is described in the Infaunal Sample Analysis Laboratory Manual (Figure 8.1). Protocols for the erection and documentation of provisional species names, based largely upon SCAMIT recommendations (SCAMIT 1986), are provided in the SCBPP Laboratory Manual. These protocols are intended to assure that adequate documentation is created for any provisional name erected and that the information is quickly and efficiently communicated to all participating taxonomists.

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

Figure 8.1. Summary of SCBPP Infaunal Sample Taxonomic Analysis

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

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

The ultimate repository of the SCBPP voucher collection and sample material has not yet been identified. This decision will have to balance the need to have the vouchers & sample material properly cared for; and the need to have the material easily available for subsequent review or re-analysis. Taxonomists involved in subsequent regional monitoring efforts will want access to the pilot project sample material. This access makes it possible for the taxonomist to re-identify taxa lots as appropriate to maintain the integrity of the original survey (see SCAMIT Comments & Recommendations to the Monitoring Sub-Committee of the Southern California Bight Review Committee, Jan 1988). SCCWRP's central role in the project as well as its central location makes it the logical repository of the sample material. This would require SCCWRP to make a long-term commitment to the maintenance of such collections, including curatorial care and management of future access. If this commitment cannot be met then other alternatives (e.g., natural history museums) will have to be explored.

8.3.4. Quality Control of Taxonomic Analysis

While the quality of taxonomic analysis in the SCBPP relies heavily on the measures described above, quality control is also provided by the re-identification of 10% of the samples processed by each laboratory. Re-identification will be conducted at a participating laboratory other than that which originally analyzed the samples. Samples for re-identification are selected randomly from each lab's assigned set of samples and randomly re-

distributed to the other three laboratories. The taxonomists conducting the re-identification do not have access to the original results.

The results are returned to the originating lab where the two sets of results are compared and a standardized report of discrepancies is prepared. The two laboratories attempt to reconcile discrepancies. In the process, apparent error is discriminated from actual error and the number of each type of error recorded. Apparent errors are cases where the discrepancy is a result of a difference in the level of the identification, rather than a misidentification. For example, the discrepancy between a report of *Tubulanus* sp. and *Tubulanus frenatus* does not represent an error, but rather a decision by one taxonomist to identify the specimen only to genus level. This decision may be based on the taxonomist's judgment that the specimen's condition is too poor for a species identification, or may reflect his or her lack of expertise in this particular group of organisms. In the latter case, the difference in treatment provides a indication where assistance from other taxonomists involved in the SCBPP is needed. Nomenclature differences are also examples of apparent error. Examples of real error are misidentifications and miscounts. In addition to characterizing analytical accuracy, this process provides information for the SCAMIT/SCBPP synoptic review of the data compiled from the four laboratories at the end of the survey. Significant discrepancies in count are resolved by a third count.

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

$$\frac{\text{Number of Organisms in resolved count} - \text{Number of errors}}{\text{Number of Organisms in resolved count}} \times 100$$

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

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

Each contributing laboratory must maintain an identification and enumeration accuracy of 90% or greater. If accuracy falls below this level, the taxa lot(s) contributing most to the error are singled out. These taxa lots in the preceding or next five samples analyzed by that laboratory (or taxonomist) must

be re-analyzed. If the errors are found to be systematic, those taxa lots in all samples processed by that laboratory (or taxonomist) must be re-analyzed. The calculated accuracy is reported on the Quality Control Accuracy Report, as well as any actions required. The completed report is signed by the responsible supervisor.

8.4. QUALITY CONTROL PROCEDURES: INFORMATION MANAGEMENT

8.4.1. Sample Tracking

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

8.4.2. Record Keeping and Reporting

Each laboratory must be responsible for maintaining thorough and complete records through all stages of the sample analysis and QC procedures. Each laboratory will employ its own bench sheet for taxonomic analysis. For the SCBPP, certain standard forms of notation are employed with the taxonomist's bench sheet that assure that all labs collect the required formation in a uniform fashion. Standardized forms are used for sorting, biomass estimation, and all QC checks. Each participating laboratory will retain its taxonomic bench sheets and voucher sheets. Biomass records, and all QC reports are to be submitted to the with the analytical results. Copies of all these documents are to be retained by the individual laboratories. Copies of all quality control reports are to be provided to the Quality Assurance Coordinator.

The laboratory supervisor is responsible for assuring that all steps in the process of analyzing infaunal samples follow SCBPP 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.

9. SEDIMENT TOXICITY TESTING

9.1. OVERVIEW

Measurement of sediment toxicity during the SCBPP will be conducted on a small scale designed to address a limited number of questions. Sediment samples from 78 stations will be collected and tested using a 10-day amphipod (*Ampelisca abdita*) survival test and a 72-hr sea urchin (*Strongylocentrotus purpuratus*) embryo development. Quality control procedures are described in this section for sample collection, testing facilities, test organisms, test

conditions, instrument calibration, use of reference toxicants, and data reporting.

9.2. QUALITY ASSURANCE PROCEDURES: LABORATORY PERFORMANCE

An anticipated use of sediment toxicity data from the SCBPP will be to provide comparisons with similar results from the small bays and estuaries pilot project. Information on the comparability of toxicity data from the laboratories participating in each project is needed.

Split samples of field sediments with varying levels of toxicity will be exchange between laboratories and analyzed using the amphipod and sea urchin toxicity tests proposed for each project. The sites used for this study will be selected based on previous data and logistics considerations. Five replicates of each sample will be tested. Concurrent reference toxicant tests will also be run. The results produced by each laboratory will be analyzed to indicate the magnitude and precision of response for each sample.

The exercise will provide a demonstration that the proposed tests can be conducted satisfactorily and will also indicate the degree of similarity in toxicity results from each laboratory. This information will be used to determine the degree of confidence that should be used when comparing data between pilot projects.

9.3. QUALITY CONTROL PROCEDURES: SAMPLE COLLECTION

The protocols used for sediment collection are described in the SCBPP Field Operations Manual. Surface sediment (top 2 cm) will be collected from a Van Veen grab using a plastic scoop and placed in a polyethylene jar. Sediment samples will be stored in the dark at 5°C for a maximum of one month before use. Sediment from 2-4 grabs at a site will be composited and thoroughly homogenized before allocation into replicate test containers or use in different tests. Samples will not be sieved prior to toxicity testing.

9.4. QUALITY CONTROL PROCEDURES: AMPHIPOD TOXICITY TEST

An *Ampelisca abdita* survival test will be conducted according to a laboratory SOP based on ASTM (1991) and EMAP guidelines. This test consists of a 10-day exposure to a 4 cm layer of sediment. Tests will be conducted jointly by SCCWRP and another laboratory. Each lab must be able to document its ability to obtain satisfactory control survival and reference toxicant response before testing the SCBPP samples.

9.4.1. Data Reproducibility

Two independent laboratories will conduct the test in order to guard against complete loss of data due to poor laboratory performance. Each laboratory will analyze a subset of 50% of the stations selected from throughout the study area. Data interpretation will be complicated if each laboratory does not produce data of comparable sensitivity and precision. Approximately 20%

of the stations analyzed by each laboratory will be duplicates of a sample analyzed by the other lab. The duplicate sample analyses will provide an indication of the data reproducibility between laboratories. The data will be examined to determine if variations between labs are random or indicate a consistent bias.

9.4.2. Quality of Test Organisms

Amphipods used in the tests will be obtained from a west coast supplier with a proven track record of providing good quality animals. The species identification of each batch of animals will be verified through consultation with a qualified taxonomist. Individuals used in the test should appear to be healthy (i.e., active with no damaged appendages). Test animals should be held in control sediment for at least 2 days but no more than 14 days before use.

A reference toxicant test must be conducted on every batch of amphipods in order to document their sensitivity. This test will consist of a 96-hr exposure to five different concentrations of cadmium dissolved in seawater. The concentration range tested will be specified in the SOP and should produce a reasonably precise estimate of the LC50. Reference toxicant concentrations will be verified by analysis of the stock solution or one of the test concentrations. The resulting LC50 will be compared to a control chart based on previous data.

Reference test LC50 values that fall outside of the control chart 95% confidence interval limits will prompt a review of the test methods in an effort to identify and correct the source of the altered sensitivity. The toxicity test will not be repeated due to the difficulty of obtaining additional sample and potential artifacts introduced by extended sediment storage. The results of the next reference toxicant test will be examined to see if the LC50 is still out of control. If it is, then a detailed review of the test methods and additional reference toxicant tests will be conducted. Sediment testing will not resume until acceptable reference toxicant results are obtained. If the one month holding time criterion is exceeded, the data will be flagged.

9.4.3. Test Conditions

Water quality (pH, DO, salinity, ammonia) of the overlying water will be measured for each treatment at the beginning and end of the test. Instruments will be calibrated and standardized daily according to the procedures of the manufacturers. Analyses will follow SOPs based on standardized methods (APHA 1989) and include measurement of replicate and reference samples. Temperature will be measured continuously. Deviations of water quality parameters from those listed in the SOP will be noted and evaluated for their potential effect on the data.

9.4.4. Test Acceptability

Amphipod survival in the control (collection site sediment) will be the principal measure of test acceptability. Average control survival must be >85%

in order for the test to be valid. The test will be repeated if the control survival is unacceptable. The test may be considered invalid if the water quality measurements deviate substantially from the ranges specified in the SOP. Appropriate comments will be attached to the data set describing the water quality deviations and their impact on the data.

9.5. QC PROCEDURES: SEA URCHIN DEVELOPMENT TEST

The sea urchin development test using *Strongylocentrotus purpuratus* will be conducted according to a laboratory SOP based on procedures described by Dinnel and Stober (1985) and Long *et al.* (1990). This test consists of a 72-hr embryo exposure to samples of 100, 50, and 25% interstitial water diluted with seawater.

9.5.1. Quality of Test Organisms

Sea urchins used to provide gametes will be obtained from northern Santa Monica Bay, a relatively uncontaminated area known to contain sea urchins of good quality. These animals will be acclimated to laboratory conditions (recirculating seawater culture) and a subsample examined to verify gamete quality. Sea urchins for this study will be collected in April and held in the laboratory until used for testing. Previous culture experience indicates that good quality gametes will be available from lab cultured sea urchins through September.

Gametes obtained from each sea urchin will be examined using a microscope. Acceptable quality will be indicated by motile sperm and eggs of uniformly mature appearance. Batches of gametes failing to meet the quality criteria will not be used in the test.

A control (laboratory seawater) and reference toxicant will be included in each toxicity test to document test performance. The reference toxicant test will consist of a 72-hr exposure of embryos to five concentrations of copper chloride dissolved in seawater. Toxicant concentrations will be selected to provide a reasonably precise estimate of the EC50 and will be specified in the SOP. The resulting EC50 will be calculated and compared to a control chart based on previous data. Toxicant concentrations will be verified by chemical analysis of the stock or one of the test solutions.

Reference test EC50 values that fall outside of the control chart 95% confidence interval limits will prompt a review of the test methods in an effort to identify and correct the source of the altered sensitivity. The toxicity test will not be repeated due to the difficulty of obtaining additional sample and potential artifacts introduced by extended sediment storage. The results of the next reference toxicant test will be examined to see if the EC50 is still outside of the desired range. If it is, then a detailed review of the test methods and additional reference toxicant tests will be conducted. Testing will not resume until acceptable reference toxicant results are obtained.

9.5.2. Test Conditions

Water quality parameters (pH, DO, salinity, ammonia, sulfide) will be measured for each interstitial water sample at the beginning of the test. Temperature during the test will also be measured. Deviations of water quality parameters from those listed in the SOP will be noted and evaluated for their potential effect on the data.

9.5.3. Test Acceptability

There must be at least 70% normal development in the laboratory control for the test to be considered valid. The test will be repeated if this criterion is not met. The test may be considered invalid if the water quality measurements deviate substantially from the ranges specified in the SOP. Appropriate comments will be attached to the data set describing the water quality deviations and their impact on the data.

9.6. QUALITY CONTROL PROCEDURES: INFORMATION MANAGEMENT

9.6.1. Sample Tracking

The toxicity laboratory will designate a sample custodian, responsible for documenting the receipt of and inspecting test samples. This person will also be responsible for assuring that established sample storage procedures are followed and transmitting required tracking information to the Information Management Officer.

9.6.2. Record Keeping and Reporting

Records of the test organisms (e.g., species, source, date of collection), equipment calibration, and test conditions will be recorded in a laboratory notebook designated for the SCBPP. Test results will be recorded on standardized data sheets and stored in the laboratory notebook. Output from computer analyses will be stored in the notebook as well on computer diskette.

The toxicity laboratory will be responsible for preparing data reports for transmission to the Information Management Officer in the proper form. Computer files and summary data sheets will be checked for transcription or calculation errors by someone other than the person originally entering the information.

10. INFORMATION MANAGEMENT

10.1. SYSTEM DESCRIPTION

The Information Management System (IMS) developed for the SCBPP is designed to perform the following functions:

- Document sampling activities and standard methods,
- Support program logistics, sample tracking and shipments,
- Process and organize both field and laboratory data,
- Perform range checks on selected numerical data,

- Facilitate the dissemination of information, and
- Provide interaction with SCBPP participants and the EMAP Central Information System.

10.2. QUALITY ASSURANCE AND QUALITY CONTROL

Two types of problems that must be resolved by QA/QC protocols for information and data management are: 1) correction or removal of erroneous individual values and 2) inconsistencies that damage the integrity of the data base. The SCBPP will provide a foundation for the management and quality assurance of all data collected and reported during the life of the project.

10.2.1. Standardization

A systematic numbering system has been developed for unique identification of individual samples, sampling events, stations, shipments, equipment, and diskettes. The sample numbering system will contain codes that will allow the computer system to distinguish among several different sample types. Each sample will be identified and tracked with a unique 10-digit log number. Field 1 identifies the Pilot Project (P). Fields 2 and 3 represent the agency collecting the data (i.e., SC for SCCWRP, SD for San Diego, OC for Orange County, LA for Los Angeles County, and HY for Hyperion). Fields 4 and 5 identify the type of sample analysis (WQ for water quality, BE for benthic analysis, GS for grain size analysis, TO for total organic carbon and nitrogen, MT for sediment metals, OR for organics, FT for fish trawls, and ST for sediment toxicity; fish species codes for tissue bioaccumulation are LS for longfin sanddab, PS for Pacific sanddab, SS for speckled sanddab, HT for hornyhead turbot, CS for California scorpionfish, DS for Dover sole, WC for white croaker, and ES for English sole). Fields 6 through 9 are the station numbers (0009 to 2011). Field 10 is the replicate (0 is the first sample, 1 is the first replicate). This system is flexible enough to allow changes during the life of the project while maintaining a structure that easily identifies the sample type.

A field computer system has been developed for the SCBPP; it includes all field data sheets; its use is optional. A clearly-written instruction manual will be developed for training field personnel and to allow easy reference in the field. Hard copies of all field data sheets are mandatory these can be either hand-written or print outs from the field computer system.

10.2.2. Data Entry, Transcription, and Transfer

In addition to paper data sheets, all data collected by field crews will be recorded on a series of electronic forms. There is a one-to-one correspondence between the electronic forms (or records) and the paper forms. Data entered in each field of the electronic forms can be checked automatically by the software, which will then provide a warning when data do not fall in an expected range.

Following the initial entry of data into the field computer system, it is printed onto hard copy and checked 100% against the original paper data sheets. This check is performed by the field crew chief, who may correct transcription errors and ultimately is responsible for assigning an acceptance code to the entered data. Once the data have been checked and accepted by the crew chief, the field personnel no longer have the ability to make changes.

At the end of CTD sampling period the original field data sheets and a diskette of the data are mailed (or hand carried) to SCCWRP. SCCWRP personnel will forward the data to Region IX. At the end of the sampling period, the original field data sheets for benthic, sediment, and fish trawl sampling activities, and a diskette of the data, are mailed (or hand carried) to SCCWRP for compilation before forwarding on to Region IX. NOTE: Each participating agency should maintain a copy of the field data sheets.

After all data sheets have been received from SCCWRP the SCBPP IMO will perform a 100% manual check of the data sheets against the submitted electronic data before archiving on the Region IX computer. Any erroneous data values identified in this check or in the previously-generated reports are changed to correct values, with authorization from the SCBPP QA Coordinator. In addition, suspicious data is flagged for further investigation. Whenever a change to the data is necessary, the IMO is required to enter a computerized data change form indicating the data sheet, variable, and reason for change. This information is written to a file that is used in compiling error rate statistics for data entry. When satisfied that the data are 100% correct, the IMO will assign an acceptance code.

10.2.3. Automated Data Verification

Erroneous numeric data will be identified using automatic range checks and filtering algorithms as part of the ODES submission process. When data fall outside of an acceptable range, they will be flagged in a report for review by the SCBPP Project Manager, the SCBPP Quality Assurance Coordinator (QAC), or their designee (QA Specialist). This report will detail the files processed and the status of the QA checks. The report will be generated both on disk and in hard copy for permanent filing. The SCBPP Project Manager or Quality Assurance Coordinator will review the report and release data that have passed the QA check for addition to the data base. All identified errors must be corrected before flagged files can be added to a data base. If the data check ranges are not reasonable, the values can be changed by a written request that includes a justification for the change.

Data base entries in the form of codes should be compared to lists of valid values (e.g., look-up tables) established by experts for specific data types. These lists of valid codes will be stored in a central data base for easy access by users. When a code cannot be verified in the appropriate look-up table, the

observation should be flagged in a written report for appropriate corrective action (e.g., update of the look-up table or removal of the erroneous code).

10.2.4. Sample Tracking

Real-time tracking of all sample shipments will be performed at the SCBPP Field Operations Center (SCCWRP). The tracking of sample shipments from the field crews to the analytical laboratories is extremely important in order to minimize loss of samples by the field crews, shipping carrier, or receiving laboratory or as a result of improper packaging. Shipment tracking is performed by: 1) the transfer of shipment and receipt information via daily/weekly telephone calls from the field crews, and receiving labs, and 2) the comparison of electronic shipment and receipt files transmitted to SCCWRP.

All shipments sent to the analytical laboratories by the field crews will be tracked by SCCWRP personnel using the ten-digit log number. All field samples collected are to be associated with a shipment number, copies of field data sheets and chain-of-custody forms, whether they are shipped using a carrier (i.e., UPS or Federal Express) or hand carried to a laboratory by a crew member. The association of field samples with the shipment numbers will make it possible to track numerous individual samples through a single number.

Field crews are required to inform SCCWRP personnel each week via telephone or fax of field and shipping activities. All shipment numbers, shipment dates, sample types, destinations, and carrier identification numbers listed during the telephone call will be carefully recorded by SCCWRP personnel on a phone log.

If verbal confirmation of receipt of a package is not received within three days of the shipment date, the SCCWRP personnel will place a telephone call to the analytical laboratory to confirm that the shipment was not received. If the shipment has not been received, the field coordinator would contact the carrier to begin a trace of the shipment.

The SCCWRP personnel will account for each sample by examining the raw shipped and receipt files, by reviewing the field data sheets, and by contacting the analytical laboratories.

10.2.5. Reporting

Following analysis of the samples, the summary data packages transmitted from the laboratories will include results, QA/QC information, and accompanying text. If the laboratory has assigned internal identification numbers to the samples, the results should include the original sample number and the internal number used by the laboratory. Specific data reporting requirements associated with each indicator are discussed in the corresponding section of this plan. Analytical laboratories are responsible for permanent

archiving of all raw data used in generating results for a minimum period of seven years.

10.2.6. Redundancy (Backups)

All files in the SCBPP IMS will be backed up regularly. At least one copy of the entire system will be maintained off-site to enable the information management team to reconstruct the data base in the event that one system is destroyed or incapacitated. In the field, all information will be recorded both on paper data sheets as well as in the computer (for those members using the field computer system). All information saved to the hard drive will also be copied to a diskette simultaneously. In addition, at the end of each day the field computers will be "equalized" to assure that the information contained on both are identical. At this point all data will be contained on the hard drives of both field computers and on a diskette. At Region IX, incremental backups to removable disk will be performed on all files which have changed on a daily basis. In addition, backups of all SCBPP directories and intermediate files will be performed on a weekly and monthly basis to provide a backup in the event of a complete loss of the Region IX GIS facility.

All original data files will be saved on-line for at least two years, after which the files will be permanently archived. Archiving of data will be on a non-volatile medium such as an optical "WORM" disk, and one copy of this will be kept off-site. All original files, especially those containing the raw field data, will be protected so that they can be read only (i.e., write and delete privileges will be removed from these files).

10.3. DOCUMENTATION AND RELEASE OF DATA

Comprehensive documentation of information relevant to users of the SCBPP IMS will be maintained and updated as necessary. Most of this documentation will be accessible on-line, in data bases which describe and interact with the system. The documentation will include a data base dictionary, access control, and data base directories (including directory structures), code tables, and continuously-updated information on field sampling events, sample tracking, and data availability.

A limited number of personnel will be authorized to make changes to the SCBPP data base. All changes will be carefully documented and controlled by the IMO. Data bases which are accessible to outside authorized users will be available in "read only" form. Access to data by unauthorized users will be limited through the use of standard UNIX security procedures. Information on access rights to all SCBPP directories, files, and data bases will be provided to all potential users.

The release of data from the SCBPP IMS will occur on a graduated schedule. Different classes of users will be given access to the data only after it

has passed a specified level of quality assurance review. Each group will use the data on a restricted basis, under explicit agreements with the Pilot Project Committee. The following four groups are defined for access to data:

- I. The SCBPP participants, including the information management team, the data analysis and reporting coordinators and liaisons, the field and laboratory coordinators, the Project Manager, QA Coordinator, and field crew chiefs.
- II. EMAP-Estuaries ERL-Narragansett personnel, ERL-Gulf Breeze personnel, NOAA EMAP-E personnel, and EMAP quality assurance personnel.
- III. EMAP data users - All other tasks groups within EPA, NOAA, and other federal, state, and municipal agencies.
- IV. General Public - University personnel and the research community.

Prior to release at level IV (general public), all files will be checked and/or modified to assure that values contain the appropriate number of significant figures. The purpose is to assure that the data released do not imply greater accuracy than was realized. This will be especially important in files where data were summarized. In such cases additional figures beyond the decimal point may have been added by the statistical program during averaging or other manipulations. It will be the responsibility of the Quality Assurance Coordinator to determine the appropriate number of significant figures for each measurement.

Requests for premature release of SCBPP data will be submitted to the Information Management Team through the SCBPP Project Manager. The SCBPP Information Manager and the Quality Assurance Coordinator, in consultation with the SCBPP Manager, will determine if the data can be released. The final authority on the release of all data is the SCBPP Project Manager (Jeffrey Cross).

11. QUALITY ASSURANCE REPORTS TO MANAGEMENT

A quality assurance report will be prepared by the QA Coordinator in association with the QA liaisons and QA specialists. This report will summarize the measurement error estimates for the various data types using the QA/QC sample data. Precision, accuracy, comparability, completeness, and representativeness of the data will be addressed in this document.

The report which will be submitted to the SCBPP pilot committee will also provide an evaluation of the QA/QC plan developed for the pilot project. It will describe the effectiveness of the QA/QC to meet the project objectives, spelling out what worked and what didn't work. The evaluation should contain recommendations as to how to improve the process for future surveys.

The QA Coordinator will report regularly to the Project Manager on an informal basis, through e-mail, conference calls, and/or direct contact. One of the primary responsibilities of the QA Coordinator is to keep the Project Manager informed of any issue or problem which might have a negative effect on the data collected.

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