

US EPA ARCHIVE DOCUMENT

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ENVIRONMENTAL MONITORING AND ASSESSMENT PROGRAM  
NEAR COASTAL DEMONSTRATION PROJECT QUALITY ASSURANCE  
PROJECT PLAN

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PROJECT QUALITY ASSURANCE PLAN APPROVAL

This project quality assurance (QA) plan was developed to assure that all environmental data generated for the U.S. Environmental Protection Agency (EPA) Environmental Monitoring and Assessment Program (EMAP) Near Coastal Demonstration Project are scientifically valid, representative, comparable, complete, and of known and acceptable precision and accuracy. The signatures of key project personnel below indicate concurrence with the procedures specified in the plan and a commitment to disseminate the plan and the philosophy of quality to all project personnel.

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This plan is approved for use in the Environmental Monitoring and Assessment Program's Near Coastal Demonstration Project.

NOTICE

This document is a preliminary draft. It has not been formally released by the U.S. Environmental Protection Agency and should not at this stage be construed to represent Agency policy. It is being circulated for comments on its technical merit and policy implications, and is intended for internal Agency use only. Mention of trade names and commercial products does not constitute endorsement or recommendation for use.

## ABSTRACT

This document outlines the integrated quality assurance plan for the Environmental Monitoring and Assessment Program's Near Coastal Demonstration Project. The quality assurance plan is prepared following the guidelines and specifications provided in 1983 by the Quality Assurance Management Staff of the U.S. Environmental Protection Agency Office of Research and Development.

Objectives for five data quality indicators (representativeness, completeness, comparability, precision, and accuracy) are established for the Near Coastal Demonstration Project. The primary purpose of the integrated quality assurance plan is to maximize the probability that data collected over the duration of the project will meet or exceed these objectives, and thus provide scientifically sound interpretations of the data in support of the project goals. Various procedures are specified in the quality assurance plan to: (1) ensure that collection and measurement procedures are standardized among all participants; (2) monitor performance of the measurement systems being used in the Near Coastal Demonstration Project to maintain statistical control and to provide rapid feedback so that corrective measures can be taken before data quality is compromised; (3) allow for the periodic assessment of the performance of these measurement systems and their components; and, (4) to verify and validate that reported data are sufficiently representative, unbiased, and precise so as to be suitable for their intended end use. These activities will provide data users with information regarding the degree of uncertainty associated with the various components of the Near Coastal Demonstration Project data base.

This quality assurance plan has been submitted in partial fulfillment of Contract Number 68-03-3249 to Lockheed Engineering & Sciences Company, Contract Number 68-C8-0066 to Science Applications International Corporation, and Contract Number 7176-849 to Computer Sciences Corporation under the sponsorship of the U.S. Environmental Protection Agency.

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## ACKNOWLEDGMENTS

We would like to thank the following individuals for their timely peer reviews of this document: D. Bender and L. Johnson, TAI, Inc. Cincinnati, Ohio; R. Graves, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio; C.A. Manen, National Oceanic and Atmospheric Administration, Rockville, Maryland; K. Summers, U.S. Environmental Protection Agency, Environmental Research Laboratory, Gulf Breeze, Florida; R. Pruell and S. Schimmel, U.S. Environmental Protection Agency, Environmental Research Laboratory, Narragansett, Rhode Island; F. Holland and S. Weisberg, Versar, Inc., Columbia, Maryland. The assistance provided by R. Graves in the development of measurement quality objectives for analytical chemistry is especially appreciated.

Word processing support provided by A. Tippet and compilation of review comments by J. Aoyama, Lockheed Engineering & Sciences Company, Las Vegas, Nevada is greatly appreciated.

## SECTION 1

### INTRODUCTION

#### 1.1 OVERVIEW

The U.S. Environmental Protection Agency (EPA), in cooperation with other federal and state organizations, has designed the Environmental Monitoring and Assessment Program (EMAP) to monitor indicators of the condition and health of the Nation's ecological resources. Specifically, EMAP is intended to respond to the growing demand for information characterizing the condition of our environment and the type and location of changes in our environment. Simultaneous monitoring of pollutants and environmental indicators will allow for the identification of the likely causes of adverse changes. When EMAP has been fully implemented, the program will answer the following critical questions:

- o What is the current status, extent and geographic distribution of our ecological resources (e.g., estuaries, lakes, streams, forests, grasslands, etc.)?
- o What percentage of resources appear to be adversely affected by pollutants or other anthropogenic environmental stresses?

- o Which resources are degrading, where, and at what rate?
- o What are the most likely causes of adverse effects?
- o Are adversely affected ecosystems improving as expected to control and mitigation programs?

To answer these types of questions the near coastal demonstration project has set four major objectives, the various, integrated monitoring networks within EMAP have four major objectives:

- o Provide a quantitative assessment of the regional extent of near coastal environmental problems by assessing pollution exposure and ecological condition.
- o Measure changes in the regional extent of environmental problems for the Nation's near coastal ecosystems.
- o Identify and evaluate associations among the ecological condition of the Nation's near coastal ecosystems and pollutant exposure, as well as other factors known or suspected to affect ecological condition (e.g., climatic conditions, land use patterns).

- o Assess the effectiveness of pollution control actions and environmental policies on regional scales (i.e., large estuaries like Chesapeake Bay, major coastal regions like the mid-Atlantic and Gulf coasts, and nationally).

The Near Coastal component of EMAP will monitor the status and trends in environmental quality of the coastal waters of the United States. This program will complement and eventually merge with the National Oceanic and Atmospheric Administration's (NOAA) existing National Status and Trends Program for Marine Environmental Quality to produce a single, cooperative, coastal and estuarine monitoring program.

The strategy for implementation of the Near Coastal project is a regional, phased approach starting in 1990 in the Virginian Province. This biogeographical province covers an area from Cape Cod, Massachusetts to Cape Henry, Virginia (U.S. EPA, 1989). Additional provinces will be added in future years, eventually resulting in full national implementation of the program.

## 1.2 QUALITY ASSURANCE PROJECT PLAN SPECIFICATIONS

The quality assurance policy of the EPA requires every monitoring and measurement project to have a written and approved

quality assurance plan (Stanley and Verner, 1983). This requirement applies to all environmental monitoring and measurement efforts authorized or supported by the EPA through regulations, grants, contracts, or other means. The quality assurance plan for the project specifies the policies, organization, objectives, and functional activities for the project. The plan also describes the quality assurance and quality control activities and measures that will be implemented to ensure that the data will meet all criteria for data quality established for the project. All project personnel must be familiar with the policies and objectives outlined in this quality assurance plan to assure proper interactions among the various data acquisition and management components of the project. EPA guidance (Stanley and Verner, 1983) states that the 15 items shown in Table 1-1 should be addressed in the QA project plan. Some of these items are extensively addressed in other documents for this project and therefore, as allowed by the guidelines, are only summarized or referenced in this document.

This document contains proposed protocols and designs for the integrated quality assurance program that will be implemented for the project. This plan is intended to be a "living" document and, accordingly, may be revised and/or appended as needs warrant.

TABLE 1-1. SECTIONS IN THIS REPORT AND IN RELATED DOCUMENTS THAT ADDRESS THE 15 SUBJECTS REQUIRED IN A QUALITY ASSURANCE PROJECT PLAN<sup>a</sup>

Quality Assurance Subject	This Report
Title page	Title page
Table of contents	Table of contents
Project description	Section 3
Project organization and responsibility	Section 2
QA objectives	Section 4
Sampling procedures	Section 6
Sample custody	Section 8
Calibration procedures	Section 5,6,7
Analytical procedures	Section 7
Data reduction, validation, and reporting	Section 8,9
Internal QC checks	Section 5
Performance and system audits	Section 5,6,7
Preventive maintenance	Section 6
Corrective action	Section 5
QA reports to management	Section 9

<sup>a</sup> Addressing these 15 QA subjects is specified in Stanley and Verner (1983).



SECTION 2  
PROJECT ORGANIZATION

2.1 MANAGEMENT STRUCTURE

For the Near Coastal Demonstration Project, expertise in specific research and monitoring areas will be provided by several EPA laboratories and their contracting organizations. The Environmental Research Laboratory in Narragansett, Rhode Island (ERL-NARR) has been designated as the principal laboratory for the demonstration project, and will therefore provide oversight and implementation support for all activities for the Demonstration Project. The Environmental Monitoring Systems Laboratory in Cincinnati, Ohio (EMSL-CIN) will provide technical support for quality assurance activities and analysis of chemical contaminants in sediment and tissue samples. The Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV) will provide quality assurance and logistics support. The Environmental Research Laboratory in Gulf Breeze, Florida (ERL-GB) has been designated as the principal laboratory for the statistical design of the Near Coastal Demonstration Project. Figure 2-1 illustrates the management structure for the 1990 Virginian Province Near Coastal Demonstration Project. All key personnel involved in the Near Coastal Demonstration Project are listed in Table 2-1.

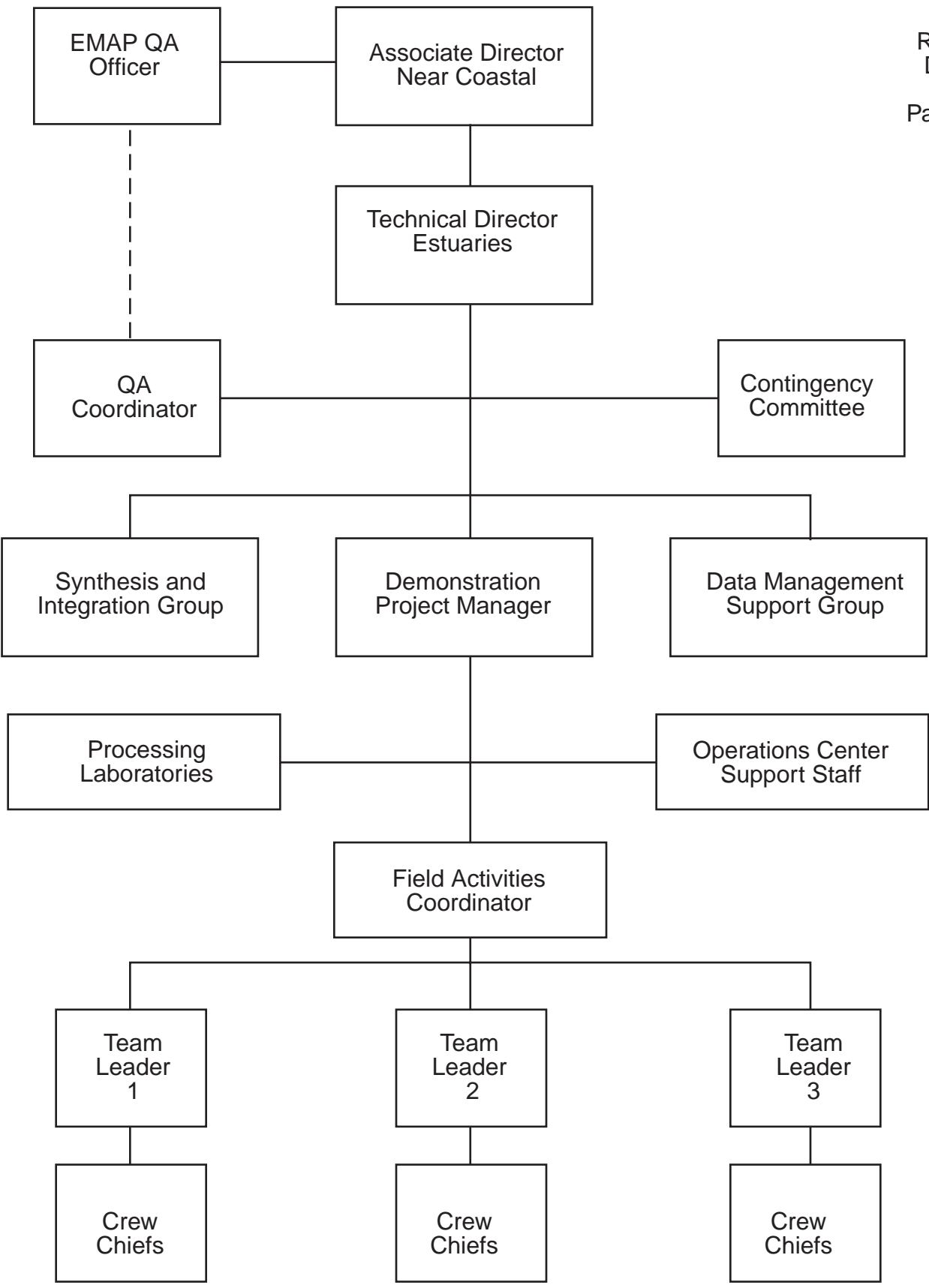


Figure 2-1. Management structure for the 1990 Virginian Province Demonstration Project (taken from Holland, et al., in preparation).

Table 2-1. List of Key Personnel, Affiliations, and Responsibilities within the EMAP Near Coastal Demonstration Project

<u>NAME</u>	<u>ORGANIZATION</u>	<u>RESPONSIBILITY</u>
R. Linthurst	U.S. EPA-DC	EMAP Director
J. Messer	U.S. EPA-RTP	Deputy Director
J. Paul	U.S. EPA-NARR	NC Associate Director
F. Holland	Versar	NC Acting Technical Director
K. Summers	U.S. EPA-GB	NC Design Lead
S. Schimmel	U.S. EPA-NARR	NC Demo Project Lead
R. Valente	SAIC	Project QA Officer
R. Pruell	U.S. EPA-NARR	Analytical Chemistry Support
B. Graves	U.S. EPA-CIN	EMAP QA Coordinator
B. Thomas	U.S. EPA-CIN	Contaminant Analysis Support
D. Heggem	U.S. EPA-LV	QA Support
J. Scott	SAIC	Toxicology/Sampling
C. Strobel	SAIC	Logistics Lead
S. Weisberg	Versar	Technical Support
J. Rosen	CSC	Data Base Management Lead
J. Baker	LESC	Logistics Support
J. Pollard	LESC	QA Support
R. Slagle	LESC	Data Base Management Support
K. Peres	LESC	QA Support
T. Chiang	LESC	QA Support
C. Manen	NOAA	NOAA QA Liaison

## SECTION 3

### PROJECT DESCRIPTION

#### 3.1 PURPOSE

The objectives of the 1990 Near Coastal Demonstration Project are to:

- o Obtain estimates of the variability associated with Near Coastal indicators which will allow establishment of program level data quality objectives (DQOs).
- o Evaluate the utility, sensitivity, and applicability of the EMAP Near Coastal indicators on a regional scale.
- o Determine the effectiveness of the EMAP network design for quantifying the extent and magnitude of pollution problems.
- o Demonstrate the usefulness of results for purposes of planning, prioritization, and determining the effectiveness of existing pollutant control actions.
- o Develop methods for indicators that can be transferred to other regions and other agencies.

- o Identify and resolve logistical issues associated with implementing the network design.

Information gained from the 1990 demonstration project will also be used to refine the overall EMAP design. The demonstration project itself will serve as a model for the implementation of EMAP projects for other ecosystem types and in other regions.

The strategy for accomplishing the above objectives will be to field test the proposed Near Coastal indicators and the network design through the demonstration project in the Virginian Province estuaries. Estuaries were selected as the target ecosystem because their natural circulation patterns concentrate and retain pollutants. Estuaries are spawning and nursery grounds for many species of living resources, and the estuarine watersheds receive a great proportion of the pollutants discharged in the waterways of the U.S. The Virginian Province was chosen because: (1) known pollution impacts are particularly severe; (2) unacceptable levels of contaminants are known to occur in the water, sediments, and biota; and (3) the vitality of many living resources are threatened (U.S. EPA, 1989).

SECTION 4  
QUALITY ASSURANCE OBJECTIVES

4.1 DATA QUALITY OBJECTIVES

To address the project objectives, the conclusions of the project must be based on scientifically sound interpretations of the data base. To achieve this end, and as required by EPA for all monitoring and measurement programs, objectives must be established for data quality based on the proposed uses of the data (Stanley and Verner, 1985). The primary purpose of the quality assurance program is to maximize the probability that the resulting data will meet or exceed the data quality objectives (DQOs) specified for the project. Data quality objectives established for the EMAP Near Coastal project, however, are based on control of the measurement system because error bounds cannot, at present, be established for end use of indicator response data. As a consequence, management decisions balancing the cost of higher quality data against program objectives are not presently possible. As data are accumulated on indicators and the error rates associated with them are established, end use DQOs can be established and quality assurance systems implemented to assure acceptable data quality to meet preestablished program objectives.

The data quality objectives presented for accuracy, precision, and completeness (Table 4-1) can be more accurately termed "measurement quality objectives" (MQOs). These objectives are based on the likely magnitude of error generated through the measurement process. The MQOs for the Near Coastal project were established by obtaining estimates of the most likely data quality that is achievable based on either the instrument manufacturer's specifications or historical data. Scientists familiar with each particular data type provided estimates of likely measurement error for a given measurement process. These MQOs are then used as quality control criteria both in field and laboratory measurement processes to set the bounds of acceptable measurement error.

DQOs or MQOs are usually established for five aspects of data quality: representativeness, completeness, comparability, accuracy, and precision (Stanley and Verner, 1985). In addition, recommended detection limits are established. These terms are defined below with general guidelines for establishing DQOs for each QA parameter.

Table 4-1. Measurement Quality Objectives for EMAP Near Coastal Indicators and Associated Data

Indicator/Data Type	Maximum Allowable Accuracy (Bias) Goal	Maximum Allowable Precision Goal	Completeness Goal
Sediment contaminant concentration			
Organics	30%	30%	90%
Inorganics	15%	15%	90%
Sediment toxicity	NA	NA	90%
Benthic species composition and biomass			
Sample collection	NA	NA	90%
Sorting	10%	NA	90%
Counting	10%	NA	90%
Taxonomic identification	10%	NA	90%
Biomass	NA	10%	90%
Sediment characteristics			
Grain size (most abundant size class)	NA	10%	90%
Total organic carbon	10%	10%	90%
Percent water	NA	10%	90%
Acid volatile sulfides	10%	10%	90%
Dissolved oxygen concentration	0.5 mg/L	10%	90%
Salinity	1 ppt	10%	90%
Depth	0.5 m	10%	90%



Table 4-1. (Continued)

Indicator/Data Type	Maximum Allowable Accuracy (Bias) Goal	Maximum Allowable Precision Goal	Completeness Goal
Fluorometry	NA	10%	90%
Transmissometry	NA	10%	90%
pH	0.2 pH units	NA	90%
Temperature	0.5 °C	NA	90%
Contaminants in fish and bivalve tissue			
Organics	30%	30%	90%
Inorganics	15%	15%	90%
Gross pathology of fish	NA	10%	90%
Fish community composition			
Sample collection	NA	NA	75%
Counting	10%	NA	90%
Taxonomic identification	10%	NA	90%
Length determinations	± 5 mm	NA	90%
Relative abundance of large burrowing bivalves			
Sample collection	NA	NA	75%
Counting	10%	NA	90%
Taxonomic identification	10%	NA	90%
Histopathology of fish	NA	NA	NA
Apparent RPD depth	± 5 mm	NA	90%
Water column toxicity	NA	40% ( <u>Champia</u> )	90%
	NA	50% ( <u>Arbacia</u> )	

#### 4.2 REPRESENTATIVENESS

Representativeness is defined as "the degree to which the data accurately and precisely represent a characteristic of a population parameter, variation of a property, a process characteristic, or an operational condition" (Stanley and Verner, 1985). Representativeness applies to the location of sampling or monitoring sites, to the collection of samples or field measurements, to the analysis of those samples, and to the types of samples being used to evaluate various aspects of data quality. The location of sampling sites and the design of the sampling program in the Near Coastal Demonstration Project provide the primary focus for defining representative population estimates from the Virginian Province near coastal estuarine environment. The proposed sampling design combines the strengths of systematic and random sampling with an understanding of estuarine systems, to collect data that will provide unbiased estimates of the status of the Nation's estuarine resources. Field protocols are documented in the Near Coastal field methods manual (Strobel et al., in preparation) for future reference and protocol standardization, as are laboratory measurement protocols in the Laboratory Methods Manual (Graves et al., in preparation). The types of QA documentation samples (i.e., performance

evaluation material) used to assess the quality of chemical data will be as representative as possible of the natural samples collected during the project with respect to both composition and concentration.

#### 4.3 COMPLETENESS

Completeness is defined as "a measure of the amount of data collected from a measurement process compared to the amount that was expected to be obtained under the conditions of measurement" (Stanley and Verner, 1985). An aspect of completeness that can be expressed for all data types is the amount of valid data (i.e., not associated with some criteria of potential unacceptability) collected. A criteria ranging from 75 to 90 percent valid data from a given measurement process is suggested as being reasonable for the Near Coastal Demonstration Project. As data are compiled for the various indicators, more realistic criteria for completeness can be developed. The suggested criteria for each data type to be collected is presented in Table 4-1.

#### 4.4 COMPARABILITY

Comparability is defined as "the confidence with which one data set can be compared to another" (Stanley and Verner, 1985). Comparability of reporting units and calculations, data base management processes, and interpretative procedures must be assured if the overall goals of EMAP are to be realized. The EMAP Near Coastal Demonstration Project will generate a high level of documentation for the above topics to ensure that future EMAP efforts can be made comparable. For example, both field and laboratory methods are described in full detail in manuals which will be made available to all field personnel and analytical laboratories. Field crews will undergo intensive training in a single month-long session prior to the start of field work. Finally, the sampling design for the Demonstration Project has been made flexible enough to allow for analytical adjustments, when necessary, to insure data comparability.

#### 4.5 ACCURACY (BIAS), PRECISION, AND TOTAL ERROR

The term "accuracy", which is used synonymously with the term bias in this plan, is defined as the difference between a measured value and the true or expected value, and represents an

estimate of systematic error or net bias (Kirchner, 1983; Hunt and Wilson, 1986; Taylor, 1987). Precision is defined as the degree of mutual agreement among individual measurements, and represents an estimate of random error (Kirchner, 1983; Hunt and Wilson, 1986; Taylor, 1987). Collectively, accuracy and precision can provide an estimate of the total error or uncertainty associated with an individual measured value. Measurement quality objectives for the various indicators are expressed separately as maximum allowable accuracy (i.e., bias) and precision goals (Table 4-1). Accuracy and precision goals may not be definable for all parameters due to the nature of the measurement type. For example, accuracy measurements are not possible for toxicity testing, sample collection activities, and fish pathology identifications because "true" or expected values do not exist for these measurement parameters (see Table 4-1).

In order to evaluate the MQOs for accuracy and precision, various QA/QC samples will be collected and analyzed for most data collection activities. Table 4-2 presents the types of samples to be used for quality assurance/quality control for each of the various data acquisition activities except sediment and fish tissue contaminant analyses. The frequency of QA/QC measurements and the types of QA data resulting from these samples or processes are also presented in Table 4-2. Because

several different types of QA/QC samples are required for the complex analyses of chemical contaminants in sediment and tissue samples, they are presented and discussed separately in Section 5.1 along with presentation of warning and control limits for the various QC sample types.

Table 4-2. Quality Assurance Sample Types, Types of Data Generated, and Measurement Quality Variables Except for all Analytical Variables

Variable	QA Sample Type or Measurement Procedure	Frequency of Use	Data Generated for Measurement Quality Definition
Sediment toxicity	Replicate tests.	Each experiment.	Variance of replicated toxicity results.
Benthic Species Composition and Biomass			
Sorting	Resort of complete sample including debris.	10% of each tech's work.	Number animals resorted.
Sample counting and ID	Recount and ID of sorted animals.	10%, of each tech's work.	Number of count and ID errors.
Biomass	Duplicate weights.	10%	Duplicate results.
Sediment Characteristics	Splits of a sample.	10% of samples.	Duplicate results.
Dissolved Oxygen Concentration	Air-saturated sea water and/or side-by-side collection/measurements with Winkler determinations.	One at each sampling location.	Replicated difference from expected.

(continued)

Table 4-2. (Continued)

Variable	QA Sample Type or Measurement Procedure	Frequency of Use	Data Generated for Measurement Quality Definition
Salinity	Known check sample in mid-range of calibration.	One at each sampling location.	Replicate difference from expected.
Temperature	Thermometer check of instrument.	One at each sampling location.	Replicated difference from expected.
Depth	Check bottom depth against depth finder on boat	One at each sampling location.	Replicated difference from actual.
Fluorometry	Chlorophyll surface grab filtered and frozen	One at each sampling location.	Check for maximum allowable difference between insitu and grab samples.
Water Clarity	QC check with standard.	One at every sampling location.	Percent difference from standard.
pH	QC check with buffer.	One at each sampling location.	Percent difference from standard.
Gross pathology of fish	Duplicate counts.	10% of trawls.	Replicated difference between determinations.

(continued)





## SECTION 5

### QUALITY ASSURANCE/QUALITY CONTROL PROTOCOLS, CRITERIA, AND CORRECTIVE ACTION

Complete and detailed protocols for field and laboratory measurements can be found in Strobel, et al. (in preparation) and Graves, et al. (in preparation), respectively. Critical features of the QA/QC procedures to be followed are presented in the following sections.

#### 5.1 CHEMICAL ANALYSIS OF SEDIMENT AND TISSUE SAMPLES

For analysis of the parts-per-billion levels of organic and inorganic contaminants in estuarine sediments and tissue (fish and bivalve), no procedure has been officially approved by the regulatory agencies. The recommended analytical methods for the purposes of this project are the standard analytical procedures followed by NOAA (MacLeod, et al., 1985 and Krahn, et al., 1988), and the methods for the Puget Sound Estuary Program (TetraTech, 1986a and 1986b). These procedures have been in effect both for the National Status and Trends Program and for the Puget Sound Estuary Program conducted by multiple agencies, including EPA and NOAA. The Puget Sound Estuary Program does not specify one single method but requires all laboratories

participating in the Status and Trends Program to participate in the NOAA performance-based program. The primary and reference laboratories to be used in the demonstration project will participate in the NOAA program and will be required to initiate corrective action if their performance falls below minimal standards specified for that program (see Table 5-1).

As discussed earlier, the data quality objectives for this project were developed with the understanding that the data will not be used for litigation purposes. Therefore, some of the requirements set by the EPA Contract Laboratory Program for legal and contracting purposes need not be applied to EMAP. In addition, it should also be pointed out that as long as proper QA/QC requirements are implemented and comparable performance on standard materials is demonstrated, multiple procedures for the analysis of the different compound classes used by different laboratories should yield comparable results. Based on this assumption, the QA/QC requirements for the analysis of contaminants in sediments and tissue will provide special emphasis on a performance-based program, which will include performance on matrix spike recoveries, laboratory



blank values, calibration standards, laboratory control materials, and intercomparison/performance evaluation studies using standard reference material. In addition surrogate spike recoveries will be used to correct data for matrix effects. The conceptual basis for use of these quality control samples is presented below. The frequency of use and recommended warning and control limits for these samples is listed in Table 5-1.

#### 5.1.1 QA/QC Requirements

Prior to the analysis of samples, each analytical laboratory must demonstrate its capability. This will be accomplished by providing laboratory documentation of both initial instrument calibration and the performance of the proposed methods through the analysis of standard reference materials (i.e., test materials of known composition). The results of this analysis must be within the specifications listed in Table 5-1 for control limits. Warning limits presented in Table 5-1 are numerical criteria that serve as flags to data reviewers and data users. When a warning limit is exceeded, the laboratory is not obligated to halt analyses, but the reported data may be qualified during subsequent QA/QC review. Control limits are numerical data criteria that, when exceeded, require specific corrective action by the laboratory before the analyses may proceed.

The guidance provided in the following sections is based largely on the protocol developed for the Puget Sound Estuary Program (TetraTech, 1986a and 1986b); it is applicable to low parts-per-billion analyses of both sediment and tissue samples unless specifically noted.

QA/QC requirements are the foundation of this protocol because they provide information necessary to assess the comparability of data generated by different laboratories and different analytical procedures.

Data for all QA/QC variables must be submitted by the laboratory as part of the data package. Program managers and project coordinators must verify that requested QA/QC data are included in the data package as supporting information for the summary data, and may review key QA/QC data (e.g., laboratory duplicate data or surrogate spike recoveries). A detailed QA/QC review of the entire data package (especially original quantification reports and standard calibration data) will be conducted by QA personnel at the ERL-NARR.

In addition to assessing data comparability, results of analyses of the various QA/QC samples will be used to document the accuracy and precision of individual measurement processes. Descriptions of the use, frequency of analysis, type of information obtained, and corrective actions for each sample type are provided in the following sections.

### 5.1.2 Initial Calibration

Equipment should be calibrated at the beginning of each analytical run, before any samples are analyzed, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended control limit criteria (see Table 5-1). Summary data documenting initial calibration and any events requiring recalibration and the corresponding recalibration data should be included with the analytical results. All standards used for initial calibration will be obtained from a single source and will be provided by NOAA, These standards can be either neat compounds or concentrated standard solutions. Calibration curves should be established for each element and batch analysis from a calibration blank and three analytical standards of increasing concentration, covering the range of expected sample concentrations. Linearity of the calibration curve must be established prior to the analysis of samples.

### 5.1.3 On-going Calibration

The on-going calibration (single-point) involves analysis of a certified control solution (calibration check sample) and is used to check the assumption that the original three-point calibration curve continues to be valid. Calibration procedures should follow those

specified for a particular method. The standard solution used for the on-going calibration should be obtained from a different source than the initial calibration standards, so that it can provide an independent check on the calibration. Analysis of the calibration check sample should occur at the beginning of a sample set, once every 10 samples or every two hours during a run, and after the last analytical sample.

If the control limit for analysis of the calibration check sample is not met, the initial three-point calibration will have to be repeated. If possible, the last sample analyzed before the check sample that failed the control limit criteria should then be reanalyzed. If the relative percent difference (RPD) between the results of this reanalysis and the original analysis exceeds 20 percent, the instrument is assumed to have been out of control during the original analysis and the earlier data should be flagged or replaced. If possible, reanalysis of samples should progress in reverse order until it is determined that there is <20 RPD between initial and reanalysis results. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control check) should be flagged.



#### 5.1.4 Method Blank

Method blanks are used to assess laboratory contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one method blank should be run in every sample batch or for every 12-hour shift, whichever is more frequent. Control limits for blanks will be based on the recommended detection limits presented in Table 5-2. These limits are based on empirical results and will be refined as the method detection limits are developed.

#### 5.1.5 Surrogate Spikes

Surrogate spike compounds must be added to each sample, including QA/QC samples, prior to extraction, purging, or digestion. The recoveries of these surrogate compounds should be carefully monitored using control charts. A minimum of five surrogate compounds must be added to each sample (three neutral and two acid compounds). These surrogate compounds should cover a wide elution range and include use of the more volatile compounds (e.g., d<sub>5</sub>-phenol). Isotopically-labeled analogs of the analytes are strongly recommended as surrogate spikes.

At least one pesticide/PCB surrogate spike is required as a check on recovery. The EPA Contract Laboratory Program (CLP) uses dibutyl chlorendate. The results of surrogate spike recovery will be used to correct data as is done in the NOAA Program.



#### 5.1.6 Matrix Spikes

Matrix spike results are used to evaluate the effect of sample matrix on the recovery of the compound of interest. Matrix spike compounds should include a wide range of representative analyte types. Spikes should be added at 1 to 5 times the concentration of compounds in the sample. Recommended warning and control limits for matrix spike recoveries are presented in Table 5-1.

#### 5.1.7 Laboratory Duplicates

Laboratory duplicates provide precision information on the actual samples. Duplicate analyses are useful in assessing potential sample heterogeneity and matrix effects.

#### 5.1.8 Standard Reference Material

Standard reference material (SRM) or performance evaluation (PE) samples are used to evaluate laboratory accuracy. Since standardized methods are not specified for this project, the SRM is considered a very important aspect of the QA/QC program. Frequency and control limits for SRMs are presented in Table 5-1. NOAA will be providing the SRMs for the EMAP laboratories and will also be responsible for coordinating and evaluating the results of round-robin laboratory tests.

## 5.2 FIELD REPLICATES

Field replicates (i.e., field splits) are separate samples collected at the identical station in the field and submitted for analysis. Analytical results for these samples can be used to determine both interlaboratory and intralaboratory precision, and to provide information on field operations. Field replicate samples should be submitted double blind (unknown) to the laboratory. If it is logistically feasible, four-way field splits should be employed, with one set of two samples being sent to the primary laboratory and the second set being sent to a referee laboratory for analysis. Approximately 10 percent of the total number of samples to be collected during the Near Coastal Demonstration Project should be split in this manner. If problems arise in the field split samples, the QA Officer must initiate action to determine if the source of error is field or laboratory based and appropriate corrective action and data flagging performed.

## 5.3 METHOD DETECTION LIMITS AND FIELD CONTAMINATION

Detectability is operationally defined as the lowest concentration that can be measured above a specified value (either zero or some background value) with a specified level of confidence. There are

several different approaches presented in the literature to determine the detection limits. The use of known low-level standards has been recommended for determination of the method detection limit (MDL) by Taylor (1987) and in the federal register (Federal Register, 1984). The importance of the MDL is that it allows definition of the lowest level of analyte for which a single measurement has an associated uncertainty of less than 30 percent (Taylor, 1987). The MDL for each analyte should be established experimentally prior to the analysis of field samples using measurements of laboratory control material.

Objectives for detectability will deal with two aspects: analytical limits of detection (MDL) and the level of tolerable contamination due to collection, handling, processing, and measurement (operationally defined as "background"). Background levels in samples will be minimized by careful adherence to sampling, handling, and processing protocols, and by establishing stringent control limits for the measurement process. Analysis of blind field blank samples will allow documentation of the background levels expected in field samples. A minimum of 10% of the expected samples should have a field blank sample associated with them.

#### 5.4. TOTAL ORGANIC CARBON

Quality control for the measurement of total organic carbon in sediment samples is accomplished by strict adherence to protocol, as well as through analysis of QA/QC samples. If levels of precision or accuracy do not fall within MQO windows (see Table 4-1), the measurements should be stopped and the system corrected before continuing the analyses. Precision will be determined by duplicate analysis of a single, homogenized sample. Minimally, one set of duplicate analyses should be performed each day or for every ten samples, whichever is applicable. The relative percent difference (RPD) between the two duplicate measurements should be less than 10.

Accuracy will be determined by analysis of a National Bureau of Standards (NBS)-traceable standard reference material; at least one standard should be analyzed every 10 samples. The RPD between the laboratory value and the standard value should be less than 10. In addition, a method blank should be analyzed with each batch of samples. If the induction furnace does not appear to be operating properly, the manufacturer's instructions for troubleshooting and repair will be followed. Total organic carbon should be reported as a percentage of

the dry weight of the unacidified sediment sample to the nearest 0.1 unit. Results should be reported for all determinations, including QA duplicates, standards, and method blanks. Any factors that may have influenced sample quality should also be reported.

#### 5.6 PHYSICAL ANALYSIS OF SEDIMENT

Quality control of sediment grain size is accomplished by strict adherence to protocol and documentation of quality control checks. Several procedures are critical to the collection of high quality particle size data. Most important to the dry sieve analysis is that the screens are clean before conducting the analysis, and that all of the sample is retrieved from them. To clean a screen, it should be inverted and tapped on a table, while making sure that the rim hits the table evenly. Further cleaning of brass screens may be performed by gentle scrubbing with a stiff bristle nylon brush. Stainless steel screens may be cleaned with a nylon or brass brush.

The most critical aspect of the pipet analysis is knowledge of the temperature of the silt-clay suspension. An increase of only 1 °C will increase the settling velocity of a particle 50 µm in diameter by 2.3 percent. It is generally recommended that the pipet analysis be

conducted at a constant temperature of 20 °C. However, Plumb (1981) provides a table to correct for settling velocities at other temperatures. Thorough mixing of the silt-clay suspension at the beginning of the analysis is also critical. A perforated, Plexiglas disc plunger is very effective for this purpose. If the mass of sediment used for pipet analysis exceeds 25 g, a subsample should be taken as described by Plumb (1981). Silt-clay samples in excess of 25 g may give erroneous results because of electrostatic interactions between the particles. Silt-clay samples less than 5 g yield a large experimental error in weighing relative to the total sample weight.

The analytical balance, drying oven, sieve shaker, and temperature bath used in the analysis should be calibrated at least monthly. Triplicate sieve and pipet analyses should be conducted on at least one sample for every 20 samples analyzed. Precision can be expressed in terms of the coefficient of variation of the weights of each size class. Acceptable precision will be 20 percent for sand, silt, and clay fractions, and 50 percent for gravel fractions. If these limits are exceeded, the data should be flagged and the laboratory protocol and/or technician's practices should be reviewed and corrected to bring the measurement error under control.



## 5.6 TOXICITY TESTING OF SEDIMENT AND WATER SAMPLES

Standard water column toxicity tests will be conducted in the demonstration project to assess their utility for regional scale assessments of environmental conditions. Three short-term methods will be used to estimate the chronic toxicity of water collected at various stations to the following species: the sea urchin Arbacia punctulata, the red macroalga Champia parvula, and the bivalve mollusc Mulinia lateralis. The toxicity of sediments collected in the field will be determined as an integral part of the benthic indicator suite, using 10-day acute bioassays with either the freshwater amphipod Hyalella azteca or the marine amphipod Ampelisca abdita. Complete descriptions of the methods employed for the water column and sediment toxicity tests are provided in the Laboratory Methods Manual (Graves et al., in preparation).

Quality assurance/quality control procedures for water column and sediment toxicity tests involve: (1) sample handling and storage; (2) the source and condition of the test organisms; (3) condition of facilities and equipment; (4) test conditions; (5) instrument calibration; (6) replication; (7) use of reference toxicants; (8) record keeping; and (9) data evaluation. These procedures are described in the following sections.

#### 5.6.1 Sample Handling and Storage

Techniques for sample collection, handling, and storage are described in the field methods manual (Strobel, et al., in preparation). Both water and sediment samples for toxicity testing should be chilled to 4°C when collected, shipped on ice, and stored in the dark in a refrigerator at 4°C until used. Water column toxicity tests should begin within 36 hours of sample collection. Sediment for toxicity testing should be stored for no longer than two weeks before the initiation of the test, and should not be frozen or allowed to dry. Sample containers should be made of inert materials to prevent contamination, which might result in artificial changes in toxicity (Strobel et al., in preparation).

To avoid contamination during collection, all sampling devices and any other instruments in contact with water or sediments should be cleaned with water and a solvent rinse between stations (see Strobel et al., in preparation). Contact of the samples with metals, including stainless steel, and plastics (including polypropylene and low density polyethylene) should be avoided as contaminant interactions may occur. Only sediments not in contact with the sides of the sampling device

should be subsampled, composited, and subsequently homogenized using instruments composed of non-reactive (i.e., inert) materials. The adequacy of the field homogenization technique for sediments will be documented in a special study prior to the start of field work.

#### 5.6.2 Quality of Test Organisms

All organisms used in the tests should be disease-free and should be positively identified to species. If organisms are collected from the field prior to testing, they should be obtained from an area known to be free of toxicants and should be held in clean, uncontaminated water and facilities. Organisms held prior to testing should be checked daily, and individuals which appear unhealthy or dead should be discarded. If greater than 5 percent of the organisms in holding containers are dead or appear unhealthy during the 48 hours preceding a test, the entire group should be discarded and not used in the test.

Whenever test organisms are obtained from an outside source (e.g., field collected or obtained from an outside culture facility), their sensitivity must be evaluated with a reference toxicant in an appropriate short-term toxicity test performed concurrently with the water column or sediment toxicity tests. For the sediment tests using

amphipods, a 96-hour toxicity test without sediment may be used to test sensitivity by generating LC-50 values. If the laboratory maintains breeding cultures of test organisms, the sensitivity of the offspring should be determined in a toxicity test performed with a reference toxicant at least once a month. If preferred, this test also may be performed concurrently with the water column or sediment toxicity tests.

Stock solutions of three reference toxicants are available from EMSL-CIN: sodium dodecyl sulfate (SDS), cadmium chloride ( $\text{CdCl}_2$ ), and copper sulfate ( $\text{CuSO}_4$ ). These reference toxicants may be obtained by contacting the Quality Assurance Branch, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268 (FTS: 684-7325; Commercial: 513-569-7325). Instructions for the use and the expected toxicity values for the reference toxicants are provided with the samples.

### 5.6.3 Facilities and Equipment

Laboratory and bioassay temperature control equipment must be adequate to maintain recommended test temperatures. Recommended materials must be used in the fabrication of the test equipment which comes in contact with the water or sediment being tested (Graves et al.,

in preparation). The acceptability of new holding or testing facilities should be demonstrated by conducting "non-toxicant" tests in which test chambers contain control sediment and clean seawater or dilution water, as appropriate for a given method. Such tests may be performed concurrent with, and serve as controls for, the reference toxicant tests used to assess single laboratory precision. These tests will demonstrate whether facilities, water, control sediment, and handling techniques are adequate to result in acceptable control level survival.

#### 5.6.4 Test Conditions

Parameters such as water temperature, salinity (conductivity), dissolved oxygen, alkalinity, water hardness, and pH should be checked as required for each test and maintained within the specified limits. Instruments used for routine measurements must be calibrated and standardized according to instrument manufacturer's procedures (see EPA methods 150.1, 360.1, 170.1, and 120.1, U.S. EPA, 1979a). All routine chemical and physical analyses must include established quality assurance practices as outlined in Agency methods manuals (U.S. EPA, 1979a,b). The wet chemical method used to measure alkalinity must be standardized according to the procedure in the specific EPA method (see EPA Method 130.2, U.S. EPA 1979a).

Overlying water or dilution water for the tests described here must meet the requirements for uniform quality specified for each method (Graves et al., in preparation). The minimum requirement for acceptable dilution or overlying water is that it allows acceptable control survival without signs of organism disease or apparent stress (i.e., unusual behavior or changes in appearance). The dilution water used in the water column toxicity tests and the overlying water used in the sediment toxicity tests with Ampelisca may be natural seawater, hypersaline brine (100 o/oo) prepared from natural seawater, or artificial seawater prepared from sea salts if recommended in the method. If natural seawater is used, it should be obtained from an uncontaminated area known to support a healthy, reproducing population of the test organism or a comparably sensitive species. Hypersaline brine prepared from uncontaminated, natural seawater also may be used to raise the salinity of fresh or intermediate salinity water samples to the appropriate levels for water column toxicity testing. Distilled or deionized water from a properly operated unit may be used to lower test water salinity. Whatever dilution water ultimately is used should be appropriate to the objectives of the study and the logistical constraints.

Fresh overlying water used in the sediment tests with Hyaella may be reconstituted water prepared by adding specified amounts of reagent grade chemicals to high quality distilled or deionized water, or natural water obtained from an uncontaminated well, spring, or surface source. Sea salt or hypersaline brine prepared from uncontaminated, natural seawater may be used to raise the salinity of this water, as appropriate to the study design.

#### 5.6.4.1 Test Acceptability

Survival of organisms in control treatments should be assessed during each test as an indication of both the validity of the test and the overall health of the test organism population. The results of the sea urchin test using Arbacia punctulata are acceptable if control egg fertilization equals or exceeds 70 percent. However, greater than 90 percent fertilization may result in masking of toxic responses. The macroalga test using Champia parvula is acceptable if survival is 100 percent, and the mean number of cystocarps per plant in the controls equals or exceeds 10. The bivalve larvae test using Mulinia lateralis is acceptable if greater than 60 percent of the embryos in the control treatments result in live larvae with completely developed shells at the

end of the test. The amphipod tests with Ampelisca abdita or Hyalella azteca are acceptable if mean control survival is greater than or equal to 90 percent, and if survival in individual control test chambers exceeds 80 percent.

Additional guidelines for acceptability of the individual water and sediment toxicity tests are presented in the Laboratory Methods Manual (Graves et al., in preparation). An individual test may be conditionally acceptable if temperature, dissolved oxygen (DO), and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests. Any deviations from test specifications must be noted and reported to the QA Officer when reporting the data so that a determination can be made of test acceptability.

#### 5.6.5 Precision

The ability of the laboratory personnel to obtain consistent, precise results must be demonstrated with reference toxicants before attempts are made to measure the toxicity of actual samples. The single laboratory precision of each type of test used in the laboratory should be determined by performing at least five or more preliminary tests with



a reference toxicant. For the amphipod tests, short-term (i.e., 96-hour) reference toxicant tests without sediments may be used for this purpose.

The trimmed Spearman-Kärber method of regression analysis (Hamilton et al., 1977) or the monotonic regression analysis developed by DeGraeve et al. (1988) can be used to determine an LC-50 or IC-50 value for each reference toxicant test. Precision then can be described by the LC-50 or IC-50 mean, standard deviation, and percent relative standard deviation (coefficient of variation, or CV) of the five (or more) replicate reference toxicant tests. Based on data reported by Morrison et al. (1989), a CV of 40 percent or less for the Champia test and a CV of 50 percent or less for the Arbacia test will be considered acceptable for demonstrating single laboratory precision prior to testing of actual samples. If the laboratory fails to achieve these precision levels in the five preliminary reference toxicant tests, the test procedure should be examined for defects and the appropriate corrective actions should be taken. The tests will then be repeated until acceptable precision is demonstrated. Throughout the testing period, precision will be assessed continually through the use of control charts.

Single laboratory precision for the Mulinia lateralis larvae test and the amphipod tests using Ampelisca and Hyaella has not been previously determined, but will be assessed prior to and during the conduct of the Near Coastal Demonstration Project to establish acceptable precision levels in the future.

#### 5.6.6 Replication and Test Sensitivity

The sensitivity of the tests will depend in part on the number of replicates, the probability level selected, and the type of statistical analysis used. The minimum recommended number of replicates varies with the test and the statistical method(s) used to address the study objectives. Test sensitivity generally increases as the number of replicates is increased, but the point of diminishing returns in test sensitivity may be reached rather quickly. The number of replicates chosen for a test should be adequate for testing hypotheses and detecting departures from the assumptions of the particular statistical analyses employed.

#### 5.6.7 Control Charts

A control chart should be prepared for each reference toxicant-organism combination, and successive toxicity values should be

plotted and examined to determine if the results are within prescribed limits (see example in Figure 9-1). In this technique, a running plot is maintained for the toxicity values ( $X_i$ ) from successive tests with a given reference toxicant. The types of control charts illustrated (U.S. EPA, 1979b) are used to evaluate the cumulative trend of results from a series of samples. For regression analysis results (such as LC-50s or IC-50s), the mean ( $\bar{X}$ ) and upper and lower control limits ( $\pm 2S$ ) are recalculated with each successive point until the statistics stabilize. Outliers, which are values which fall outside the upper and lower control limits, and trends of increasing or decreasing sensitivity, are readily identified. At the  $P=0.05$  probability level, one in twenty tests would be expected to fall outside of the control limits by chance alone.

If the toxicity value from a given test with the reference toxicant does not fall in the expected range for the test organisms, the sensitivity of the organisms and the overall credibility of the test are suspect. In this case, the test procedure should be examined for defects and, if possible, the test should be repeated with a different batch of test organisms.

#### 5.6.8 Record Keeping and Reporting

Proper record keeping is mandatory. Bound notebooks should be used to maintain detailed records of the test organisms such as species, source, age, date of receipt, and other pertinent information relating to their history and health, and information on the calibration of equipment and instruments, test conditions employed, and test results. Annotations should be made on a real time basis to prevent loss of information. Data for all QA/QC variables, such as reference toxicant test results and copies of control charts, should be submitted by the laboratory as part of the data package.

#### 5.7 BENTHIC COMMUNITY ANALYSIS

Analysis of species composition, abundance, and biomass will help to determine the ecological condition of the benthic community. Since benthic communities are relatively immobile, and therefore cannot easily escape unhealthy ecological conditions, this indicator represents an integrative component of the near coastal ecological system.

Sediment samples for benthic community analysis will be collected at each station using a Young-modified Van Veen grab sampler. These samples will be sieved in the field through a 0.5 mm screen and

the material collected on the screen preserved and returned to the laboratory for processing. Details of field and laboratory processing procedures can be found in Strobel et al. (in preparation) and Graves et al. (in preparation), respectively.

#### 5.7.1 Species Composition and Abundance

Quality control for processing grab samples involves both sorting and counting check systems for quality control. A check on the efficiency of the sorting process is required to document the accuracy of the organism extraction process. In addition to sorting QC, it is necessary to perform checks on the accuracy of sample counting. This can be done in conjunction with taxonomic identification and uses the same criteria presented below for taxonomic identification quality control.

Sorting QC can be separated into two levels of intensity. Inexperienced sorters require an intensive QC check system, while experienced personnel require a less frequent QC schedule. It is recommended that experienced sorters or taxonomists check each sample for missed organisms until proficiency in organism extraction is demonstrated by inexperienced personnel.

Two types of QC sorting criteria are recommended to maintain control and comparability of the sorting process. One criterion for completion of sorting that has been used successfully in fresh water systems is to sort a sample until the sorter feels that the sample is finished, then continue to sort until no organisms or fragments can be found in a one-minute continuous examination (Pollard and Melancon, 1984; Peck et al., 1988). The time criterion for completion of a sort will depend on the composition of the sample and will need to be established for marine benthic samples, but must be initially based on the sorter's judgement that the sample sort is complete. The criterion that is used for initial sorting of a sample should also be used for the quality control sort. The second criterion for sorting acceptability is the extraction efficiency of a given sorter. Acceptable quality for sorting extraction should be that no more than 10 percent of the original organism count is removed upon a QC check sort. A minimum of 10 percent of samples processed by a given sorter should be subjected to a QC sort at regular intervals during sample processing. If a sorter fails QC sorts, then all samples processed from the last successful QC check are resorted and any additional animals found are added to each sample. If QC sorting passes, but some animals are found, these animals are not added to the original sample sort.

As organisms are identified and corrected, a voucher specimen collection will be compiled. This specimen collection can be used for training new taxonomists and as a quality crosscheck by sending specimens to a separate laboratory for identification. All specimens should be taxonomically confirmed by an outside source and any discrepancies resolved. Identification and enumeration accuracy should be checked internally by a second taxonomist for at least 10 percent of the samples processed by a given technician. There should be no more than 10 percent error in identification or enumeration in any sample. The same procedures for sample reprocessing that are used for sorting apply to identification and counting.

#### 5.7.2 Biomass

Biomass determination procedures involve ashing the sample, and, as a consequence, cannot be controlled and corrected in a similar manner to the sorting, identification, and enumeration processes. Duplicate weight measurements by a separate technician will be taken before and after ashing of the samples to control and document the precision of this measurement process. If the two technician's results differ by more than 10 percent, the laboratory manager will then be notified and the reasons for this discrepancy identified and resolved.

## 5.8 LARGE BIVALVE SAMPLING

Large bivalves collected with a rocking chair dredge will be identified to species and measured in the field. Samples will be placed in bags and iced prior to transport and storage (see Strobel et al., in preparation, for details of field procedures). Quality of identification and measurement will be documented during training and during the final field audit. The acceptance criteria for abundance and composition is to be accurate within 10 percent of the original determination.

## 5.9 FISH SAMPLING

### 5.9.1 Species Composition and Abundance

Fish species composition and abundance will be determined in the field following protocols presented in the field methods manual (Strobel et al., in preparation). Documentation of the quality of these data will be accomplished by performing field crew training and QA audits using personnel qualified to verify the identification and enumeration of the field crew. Acceptance criteria for abundance and composition is to be accurate within 10 percent of the original determination. In



addition, fish sent to the laboratory will be checked for taxonomic determination accuracy. The acceptable error rate for this procedure has not been established, but will be recorded as quality assurance documentation.

#### 5.9.2 Fish Length Measurements

A random subset of the fish measured in the field will be set aside for duplicate measurements by a second technician. The acceptable error in this procedure is  $\pm 1$  cm. If this procedure cannot be followed due to logistical constraints, then quality assurance documentation of fish length will be accomplished during field auditing.

#### 5.9.3 Fish Gross Pathology

The field procedures to be used for determination of fish pathology are detailed in Strobel, et al., in preparation. The quality of gross scanning for fish pathology will be documented during field training and QA audits. In addition, the quality of fixation techniques and laboratory techniques will be documented during the QA audits.

#### 5.10. SEDIMENT-PROFILE PHOTOGRAPHY

The field procedures for sediment-profile photography are described in the field methods manual (Strobel, et al., in preparation). The techniques for measuring various physical and biological parameters (e.g., sediment grain size, camera penetration depth, redox potential discontinuity (RPD) depth, infaunal successional stage) in the sediment-profile photographs are described in the laboratory methods manual (Graves, et al., in preparation). The main features of the quality assurance/quality control protocol for sediment-profile photography are described in the following sections.

At the beginning of each field operation, the time on the data logger mounted on the sediment-profile camera should be synchronized with the clock on the navigation system computer. Each photograph can then be identified by the time recorded on the film, and matched with the time recorded on the computer along with vessel position. Redundant sample logs should be kept by the field crew and by computer printout. Test photographs should be taken on deck at the beginning and end of each roll of film to verify that all internal electronic systems are working to design specifications. Spare cameras and charged batteries should be carried in the field at all times to insure uninterrupted sample acquisition.

After deployment of the camera at each sampling site, the frame counter (digital display) should be checked to make sure that the requisite number of replicate photographs has been taken. In addition, the prism penetration depth indicator on the camera frame should be checked to see that the optical prism has actually penetrated the bottom to a sufficient depth to acquire a profile image. If photographs have been missed (frame counter indicator) or the penetration depth is insufficient (penetration indicator), additional replicates should be taken. All film should be developed at the end of every survey day to verify successful data acquisition; strict controls should be maintained for development temperatures, times, and chemicals to insure consistent density on the film emulsion to minimize interpretive error by the computer image analysis system. After it is developed, the film should be visually inspected under magnification. Any images that are of insufficient quality for computer image analysis should be noted, and, if possible, the appropriate sampling site should be revisited at a future date.

During computer analysis of the sediment-profile photographs, all measurements from each photograph are stored on disk and a summary display is made on the computer screen so the operator can visually verify if the values stored in memory for each variable are within the expected range. If anomalous values are detected, software options

allow remeasurement and recalculation before storage on disk. All computer data disks are backed-up by redundant copies at the end of each analytical day. All data stored on disks also are printed out on data sheets to provide a hard copy backup; a separate data sheet is generated for each sediment-profile photograph which has been analyzed. As a final quality control check, all data sheets are edited and verified by a senior-level scientist before being approved for final data synthesis, statistical analyses, and interpretation.

#### 5.11 DISSOLVED OXYGEN MEASUREMENTS

Dissolved oxygen will be measured using both a recording DataSonde III Hydrolab unit and a Seabird CTD instrument, both of which are rated by the manufacturer as being accurate to 0.2 ppm (Strobel et al., in preparation). The CTD will be used for daily measurements and the Hydrolab for long-term measurements (i.e., 10-day continuous deployments). The oxygen meters will be calibrated in saturated seawater following manufacturer's specifications, and the calibration values recorded prior to probe use. Calibration will be checked each time either probe is deployed, and when the Hydrolab is retrieved, by taking a simultaneous water sample and measuring dissolved oxygen concentration by Winkler titration. If the Winkler results and those obtained from either probe differ by greater than 0.5 ppm at the time

the probe is deployed, the probe will have to be checked for malfunctions, recalibrated, then rechecked for calibration before it can be redeployed. If the Winkler results and those obtained from the Hydrolab probe differ by greater than 0.5 ppm when this probe is retrieved after the long-term deployment, the data will be flagged as being outside the quality control criteria and will be reviewed for validity prior to data release.

## 5.12 Ancillary Measurements

### 5.12.1 Salinity

Salinity will be measured using the Seabird CTD profiling recording probe which is rated by the manufacturer as being accurate to 1 percent (Strobel, et al., in preparation). Salinity meters are calibrated by the manufacturer; this calibration will be checked each time the probe is deployed using a simple refractometer. Drift in these recorded calibration values will be monitored and used as a criteria for data flagging. If the quality control check results differ from the probe values by greater than 1 part per thousand, the instrument will be checked thoroughly and a determination made of the need for recalibration.

### 5.12.2 Temperature

Temperature will be measured using the Seabird CTD profiling recording probe which is rated by the manufacturer as being accurate to 0.2 °C (Strobel et al., in preparation). The temperature sensor on the probe will be calibrated by the manufacturer using a National Bureau of Standards [NBS] certified thermometer, and the calibration value recorded prior to probe use. Probes will be tested for calibration stability upon deployment and retrieval, and that value recorded. Drift from the original calibration will be used as a criteria for data quality acceptance and as a data flagging criteria. If calibration results differ from the original calibration by greater than 0.5 °C, the data will be flagged as being outside the quality control criteria and will then be reviewed for validity prior to data release.

### 5.12.3 pH Measurements

pH measurements will be taken with the Seabird CTD. The instrument will be calibrated to pH 7 and pH 10 as described in Strobel, et al., in preparation, Appendix H. Following calibration, a QC check will be performed using an intermediate range buffer solution (pH 8 is suggested). The QC check should be within 0.2 pH units of the true value for the buffer solution. If the QC check is outside control

limits, the instrument calibration should be checked. Quality control checks should be performed and recorded prior to and following deployment of the CTD.

#### 5.12.4 Fluorometry

Chlorophyll<sub>a</sub> readings will be taken using the Seabird CTD. The instrument will be calibrated as specified in Strobel, et al. (in preparation). In addition, a surface grab sample will be collected, filtered, and frozen for later chlorophyll<sub>a</sub> analysis. This sample will be used for QA documentation.

#### 5.12.5 Transmissometry

No QA/QC procedures are specified for this parameter other than calibration procedures outlined in Strobel, et al., in preparation.

#### 5.12.6 Photosynthetically Active Radiation

No QA/QC procedures are specified for this parameter other than those outlined in Strobel, et al., in preparation.

#### 5.12.7 Sediment Mixing Depth

The depth of the black layer in sediments will be determined to  $\pm$  5mm in the field. The accuracy of this measurement will be assured by initial training efforts and documented during field QA audits.

#### 5.12.8 Acid Volatile Sulfides

Acid volatile sulfides within sediments will be measured in the laboratory following the procedures outlined in Graves, et al., in preparation. Precision of this measurement will be monitored by taking laboratory duplicates and maintaining a 10% relative percent difference between duplicates. Accuracy of the method will be measured by analyzing a sodium sulfide crystal of known weight and comparing the results of this analysis and the expected value the results should agree within  $\pm$  10%.



## SECTION 6

### FIELD OPERATIONS AND PREVENTIVE MAINTENANCE

#### 6.1 TRAINING AND SAFETY

A critical aspect of quality control is to ensure that the individuals involved in each activity are properly trained to conduct the activity. Field sampling personnel are being asked to conduct a wide variety of activities using comparable protocols. Each field team will consist of a Team Leader and two 4-member crews. Each crew will have a Crew Chief (one of which is the Team Leader), who will be the captain of the boat and will be the ultimate on-site decision maker regarding safety, technical direction, and communication with the Operations Center.

Qualifications for the Team Leaders and Crew Chiefs an M.S. degree in Biological/Ecological Sciences and three years of experience with field data collection activities, or a B.S. degree and five years experience. The remaining three crew members generally will be required to have a B.S. degree and, preferably, at least one year's experience. All field team members will be required to take part in an intensive one month training period.

Classroom training will be conducted by the University of Rhode Island's Marine Advisory Service and Fisheries Department. The instructors and staff of this department have wide-ranging experience in training scientific personnel in routine sampling operations (e.g., collection techniques, small boat handling). Their expertise will be supplemented by recognized experts in such specialized areas as fish pathology (Dr. Linda Despres-Patanjo NMFS, Woods Hole, Massachusetts and Mr. John Ziskowski, NMFS, Milford, Connecticut); fish identification (Dr. Don Flescher, NMFS, Woods Hole); benthic sampling (Ms. Anna Shaughnessy, Versar, Inc., Columbia, Maryland); first aid, including cardio pulmonary resuscitation (CPR) (American Red Cross); and field computer/navigation system use (Mr. Jeffrey Parker, Science Applications International Corporation, Newport, Rhode Island).

All EMAP equipment (e.g., boats, sampling gear, computers) will be used during the training sessions, and by the end of the course, all crews members must demonstrate proficiency in:

- o Towing and launching the boat.
- o Making predeployment checks on all sampling equipment.

- o Locating stations using the appropriate navigation system (LORAN and/or GPS).
- o Entering and retrieving data from the onboard lap-top computers.
- o Using all the sampling gear.
- o Administering first aid, including CPR.
- o General safety practices.

In addition, all field crew members must be able to swim and will be required to demonstrate that ability.

Some sampling activities (e.g., fish taxonomy, gross pathology, net repair, etc.) require specialized knowledge. While all crew members will be exposed to these topics during the training sessions, it is beyond the scope of the training program to develop proficiency for all crew members in these areas. For each of the specialized activities, selected crew members, generally those with prior experience in a particular area, will be provided intensive training. At the conclusion of the training program, at least one member of each crew will have

demonstrated proficiency in fish taxonomy, mollusk taxonomy, gross pathology, net repair, gear deployment, and navigation.

All phases of field operations are detailed in the field methods manual (Strobel, et al., in preparation) that will be distributed to all trainees prior to the training period. The manual will include a checklist of all equipment, instructions on the use of all equipment, and sample collection procedures that the field crews will be required to conduct. In addition, the manual will include flow charts and a schedule of activities to be conducted at each sampling location. It will also contain a list of potential hazards associated with each sampling site.

## 6.2 FIELD QUALITY CONTROL

Quality control of field measurements will be accomplished by use of a variety of QC sample types. Specific field QC protocols can be found in Strobel et al. (in preparation). A description of the general protocols, control limits, and sample types used for this purpose can be found in sections 4 and 5 of this document.

### 6.3 FIELD AUDITS

Initial review of the field team observations will be performed by training personnel during the training program. Following training, an initial site assistance audit should be performed by a combination of QA and training personnel. This audit should be considered a "shake down" assistance procedure to help field teams provide a consistent approach to collection of samples and generation of data. At least once during the program, a formal site audit will be performed by the QAO and the Demonstration Project manager to determine compliance with the QA plan and field operations document. Checklists and audit procedures will be developed for this audit that are similar to those presented in U.S. EPA (1985). Corrective action and/or retraining of crew personnel will be initiated if discrepancies are noted.

### 6.4 PREVENTIVE MAINTENANCE

The importance of proper maintenance of all gear cannot be understated. Failure of any piece of major equipment, especially when back-up equipment will be used by a fourth team, could result in a

significant loss of data. Maintenance of equipment should be performed as described in Strobel et al (in preparation). It will be the responsibility of the Team Leader to maintain a record of equipment usage, and assure that proper maintenance is performed at the prescribed time intervals.

The following equipment will be regularly checked and/or serviced as specified in Strobel, et al. (in preparation): Boat trailers, boats, outboard engines, electronics, hydraulics, rigging, vehicles, grid computers, Seabird CTD's and DataSonde III Hydrolabs.

## SECTION 7

### LABORATORY OPERATIONS

#### 7.1 LABORATORY PERSONNEL, TRAINING, AND SAFETY

Laboratory operations and preventive maintenance necessary for proper operation of laboratory equipment are discussed in detail in Graves et al. (in preparation). This section addresses only general laboratory operation considerations, while the laboratory QA/QC considerations are presented in sections 4 and 5.

The toxicity or carcinogenicity of individual compounds or reagents used in this project has not been precisely determined. Therefore, each chemical should be treated as a potential health hazard and good laboratory practices should be implemented accordingly. Laboratory personnel should be well versed in standard laboratory safety practices and standard operating procedures (SOPs) strictly followed as presented in Graves, et al. (in preparation). It is the responsibility of the laboratory manager and 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) regulations regarding the safe handling of the chemicals specified for this

project and individual chemical safety data sheets. These procedures and documents should be made available to and followed by all personnel involved in this project.

## 7.2 QUALITY CONTROL DOCUMENTATION

The following documents and information must be current, and must be available to all laboratory personnel and to the principal investigators:

- o Laboratory methods manual - A document containing detailed instructions about laboratory and instrument operations (Graves et al., in preparation).
- o Quality assurance plan - Clearly defined laboratory protocols, including personnel responsibilities and the use of QA/QC protocols (this document).
- o Instrument performance study information - Information on baseline noise, calibration standard response, precision as a function of concentration, and detection limits.



- o Training and field operations and manual (Strobel et al., in preparation) including quality control performance criteria (e.g., calibration routines and acceptance criteria).

### 7.3 SAMPLE PROCESSING AND PRESERVATION

Sample processing and preservation protocols are presented in Strobel et al. (in preparation) for field collected data, and in Graves et al. (in preparation) for laboratory processed data. Strict adherence to the protocols provided in these documents is critical to maintain data integrity.

### 7.4 SAMPLE STORAGE AND HOLDING TIMES

Water samples for toxicity testing should be shipped on ice, but not frozen. Transit and subsequent holding time should not exceed 48 hours. Sieved biota from sediments must be preserved on the boat according to procedures presented in Strobel et al. (in preparation). For the analyses of organic contaminants in sediments, it is recommended that the sediment samples be extracted within 10 days and extracts analyzed within 40 days following extraction (Contract Laboratory Program [CLP], Statement of Work [SOW] 288). For inorganic sediment

contaminants (except mercury), it is recommended that samples be digested within 180 days and the extracts analyzed within 1 day (for Sb, Pb, Hg, Se, and Ag), within 2 days (for As and Cd), and within 1 week (for Cr, Cn, Ni, and Zn). For mercury, the holding time is 26 days (CLP SOW 288). For the analyses of contaminants in fish muscle tissue, the whole fish will be shipped frozen on dry ice and should be held frozen until the time of analysis.

#### 7.5 LABORATORY PERFORMANCE AUDITS

Initially, a QA assistance and performance audit will be performed by QA personnel to determine if each laboratory effort is in compliance with the procedures outlined in the QA plan and to assist the laboratory where needed. Additionally, once during the study, a formal laboratory audit following protocols similar to those presented in U.S. EPA (1985) checklists that are appropriate for each laboratory operation will be developed and approved by the QA Officer prior to the audits.

## SECTION 8

### QUALITY ASSURANCE AND QUALITY CONTROL FOR MANAGEMENT OF DATA AND INFORMATION

#### 8.1 SYSTEM DESCRIPTION

The prototype of the Near Coastal Information Management System (NCIMS) will be developed at the Environmental Research Laboratory in Narragansett (ERL-N). The design for this system will be reviewed by the EMAP Information Management committee and by the technical director of the Near Coastal Demonstration Project. Ultimately, the NCIMS will:

- o document sampling activities and standard methods,
- o support program logistics, sample tracking and shipments,
- o process and organize both the data collected in the field and the results generated at analytical laboratories,
- o perform quality control checks,
- o facilitate the dissemination of information, and
- o provide interaction with the EMAP Central Information System.

### 8.1.1 Field Navigation and Data Logging System

Portable microcomputers modified to withstand the rigors of use on small boats represent an important component of the data management system for the Near Coastal project. The software on these machines will provide navigation and real time positioning of the boat, and control all sampling activities, sample logging, and data storage through an interactive menu. The software to be used is a modification of the Integrated Navigation and Survey System (INSS) developed by Science Applications International Corporation.

The INSS is a simple, automated, menu-driven software package with complete logging facility; it has been used successfully on numerous environmental field programs during the past decade.

### 8.2 QUALITY ASSURANCE/QUALITY CONTROL

Two general types of problems which should be resolved in developing 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 following features of the NCIMS will provide a foundation for the management and quality assurance of all data collected and reported during the life of the project.

#### 8.2.1 Standardization

A systematic numbering system will be developed for unique identification of individual samples, sampling events, stations, shipments, equipment, and diskettes. The sample numbering system will contain codes which will allow the computer system to distinguish among several different sample types (e.g., actual samples, quality control samples, sample replicates, etc.). This system will be flexible enough to allow changes during the demonstration project, while maintaining a structure which allows easy comprehension of the sample type.

Clearly stated standard operating procedures will be given to the field crews with respect to the use of the field computer systems and the entry of data in the field. Contingency plans will also be stated explicitly in the event that the field systems fail.

### 8.2.2 Prelabeling of Equipment and Sample Containers

Whenever possible, sample containers, equipment, and diskettes will be pre-labeled to eliminate confusion in the field. The pre-labeling will reduce the number of incorrect or poorly-affixed labels. Containers with all the required pre-labeled sample containers, sample sheets, and data diskettes will be prepared for the field crews prior to each sampling event (an event is defined as a single visit by a crew to a sampling site). These containers will be called "event boxes". Each event box will have the event number affixed to it using both handwritten and bar code labels.

### 8.2.3 Data Entry, Transcription, and Transfer

To minimize the errors associated with entry and transcription of data from one medium to another, data will be captured electronically. When manual entry is required, the data should be entered twice by different data entry operators and then checked for non-matches to identify and correct errors. In many instances, the use of bar code labels should eliminate the need for manual entry of routine information.

Each group transmitting data to the information center will be given a separate account on the Near Coastal VAX 3300. Standard formats for data transfer will be established by the Information Management Team. A specific format will be developed for each file type within each discipline. If data are sent to the Near Coastal Information Center in formats other than those specified, the files will be deleted and the sending laboratory or agency will be asked to resubmit the data in the established format.

The communications protocols used to transfer data electronically will have mechanisms by which the completeness and accuracy of the transfer can be checked. In addition, the group sending the information should specify the number of bytes and file names of the transferred files. These data characteristics should be verified upon receipt of the data. If the file cannot be verified, a new file transfer should be requested. Whenever feasible, a hard copy of all data should be provided with transfer files.

The data files transmitted from the field will be fixed format text files. These files will be "parsed" by the system. The parsing process involves transferring records of similar type into files containing only those types of records. For example, observation on fish species and size will be copied from the original log file transmitted from the

field to a "fish" data file. After the records have been parsed from the field log files, the individual data files will be checked automatically for erroneous values, as described in the following section. Records in the field log file which are not entered into the data base (e.g., comments in text form) will be archived for documentation or future extraction.

#### 8.2.4 Automated Data Verification

Erroneous numeric data will be identified using automatic range checks and filtering algorithms. When data fall outside of an acceptable range, they will be flagged in a report for the quality assurance officer (QAO), or his designee. This type of report will be generated daily and should detail the files processed and the status of the QA checks. The report will be generated both on disk and in hardcopy for permanent filing. The QAO will review the report and release data which 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 QAO finds that the data check ranges are not reasonable, the values can be changed by written request. The written request should include a justification for changing the established ranges. If the QAO finds the need for additional codes,



they can be entered by the senior data librarian. After such changes are made, the files may be passed through the QA procedure again. In the event that the QA check identifies incorrect data, the QAO will archive the erroneous file and request that the originator corrects the error and retransmits the data.

Data base entries which are 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 data base users. When a code cannot be verified in the appropriate look up table, the observation should be flagged in the QAO report for appropriate corrective action (e.g., update of the look up table or removal of the erroneous code).

#### 8.2.5 Sample Tracking

Samples collected in the field will be shipped to analytical laboratories. All shipping information required to adequately track the samples (sample numbers, number of containers, shipment numbers, dates, etc.) will be transmitted by phone to the information center at the end of each sample day, using modems built into the portable field computers. Once the field crew have transmitted the data, it will be

the responsibility of the data management team to confirm that the samples arrive at their destination. To facilitate this, the receiving laboratories will be required, upon receipt of the samples, to record and similarly transmit all tracking information (e.g., sample identification numbers, shipment numbers and the status of the samples) to the information center, using either microcomputers or the VAX. The information management team will generate special programs to create fixed format records containing this information.

#### 8.2.6 Reporting

Following analysis of the samples, the summary data packages transmitted from the laboratories will include sample tracking information, results, quality assurance and quality control 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. The analytical laboratories will be responsible for permanent archiving of all raw data used in generating the results.

#### 8.2.7 Redundancy (Backups)

All files in the NCIMS 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, information stored on the hard drive will be sent to the on-board printer to provide a real time hardcopy backup. The information on the hard drive also will be copied to diskettes at the end of each day of sampling. At the Near Coastal Information Center in Narragansett, incremental backups to removable disk will be performed on all files which have changed on a daily basis. In addition, backups of all EMAP directories and intermediate files will be performed on a weekly basis to provide a backup in the event of a complete loss of the Near Coastal Information Center facility.

All original data files will be saved on-line for at least two years, after which the files will be permanently archived on floppy diskette. All original files, especially those containing the raw field data, will be protected so that they can only be read (i.e., write and delete privileges will be removed from these files).

#### 8.2.8 Human Review

All discrepancies which are identified by the computer will be documented in hard copy. These discrepancy logs will be saved as part of the EMAP archive. All identified discrepancies should be brought to the attention of the QAO or his designee, who will determine the appropriate corrective action to be taken. Data will not be transferred to the data base until all discrepancies have been resolved by the QAO. Once data have been entered into the data base, changes will not be made without the written consent of the QAO, who will be responsible for justifying and documenting the change. A record of all additions will be entered into a data set index and kept in hard copy.

#### 8.3 DOCUMENTATION AND RELEASE OF DATA

Comprehensive documentation of information relevant to users of the NCIMS 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 Near Coastal data base. All changes will be carefully documented and controlled by the senior data librarian. 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 DEC VAX security procedures. Information on access rights to all EMAP-NC directories, files, and data bases will be provided to all potential users.

The release of data from the NCIMS will occur on a graduated schedule. Different classes of users will be given access to the data only after it reaches a specified level of quality assurance. Each group will use the data on a restricted basis, under explicit agreements with the Near Coastal Task Group.

The following four groups are defined for access to data:

- I. The Near Coastal central group, including the information management team, the field coordinator, the logistics coordinator, the demonstration project coordinator, the QA officer and the field crew chiefs.

- II. Near Coastal primary users - ERLN, VERSAR, SAIC, Gulf Breeze personnel, NOAA Near Coastal EMAP personnel, and EMAP quality assurance personnel.
  
- III. EMAP data users - All other task groups within EPA, NOAA, and other federal agencies.
  
- IV. General Public - university personnel, other EPA offices (includes regional offices), and other federal, state, and local governments.

The following table summarizes the policy of the Near Coastal Task Group with respect to the distribution of data. The Roman numerals in the table refer to the groups listed above.

Requests for premature release of data will be submitted to the Information Management Team. The senior data analyst and the QAO will determine if the data can be released. The final authority on the release of all data, however, is the decision of the technical director of EMAP Near Coastal.



## SECTION 9

### QUALITY ASSURANCE REPORTS TO MANAGEMENT

The first annual report for the Near Coastal project is scheduled in June of 1991 after completion of the Near Coastal Demonstration Project in the Virginian Province. This report will, in part, provide an assessment of QA activities and an evaluation of the design and research indicators initially used for the project. After full implementation of the Near Coastal component of EMAP, progress will be reported on an annual basis.

Control charts will be used extensively to document measurement process control. An example of a control chart is shown in Figure 9-1. Control charts must be used with QC check standards for controlling instrument drift, matrix spike, or surrogate recoveries to measure extraction efficiency or matrix interference, certified performance evaluation samples and blank samples to control overall laboratory performance, and with reference toxicant data to assess laboratory precision and variability in bioassay test species sensitivity. These control charts should be maintained at the laboratory and included as part of the data packages.



A quality assurance report (or section of the project report) will be prepared following the project's completion, which will summarize the measurement error estimates for the various data types using the QA/QC sample data (see Section 4 and 5). Precision, accuracy, comparability, completeness, and representativeness of the data will be addressed in this document and method detection limits reported.

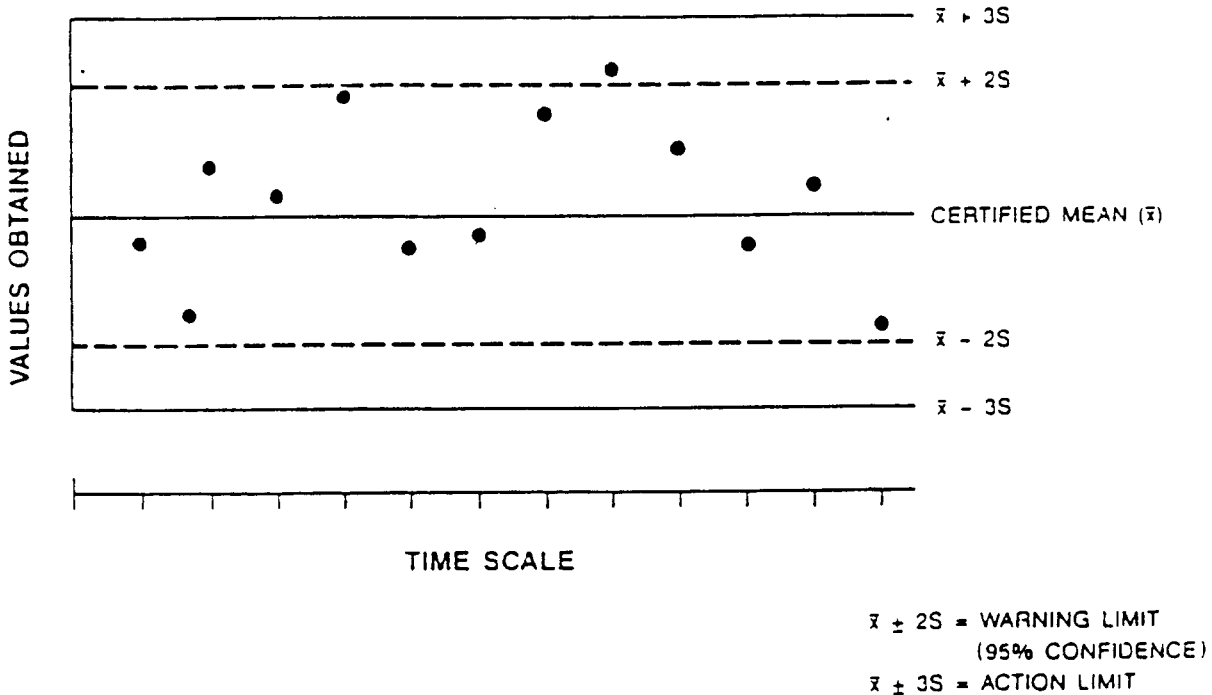


Figure 9-1. Example of a control chart.

SECTION 10

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