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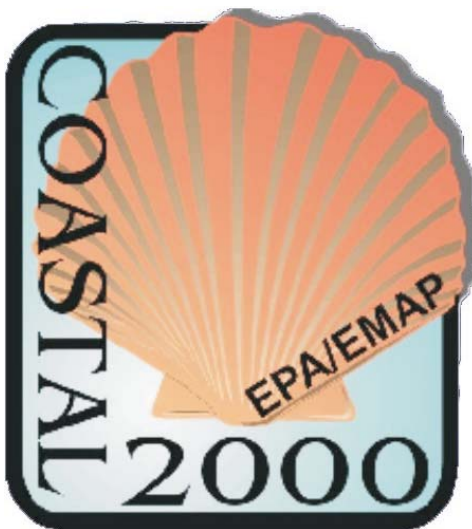
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Washington, DC 20460

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May 2001



## **National Coastal Assessment**

### **Quality Assurance Project Plan 2001 - 2004**



**Environmental Monitoring  
and Assessment Program**

# National Coastal Assessment Quality Assurance Project Plan 2001- 2004

by

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## NOTICES

The original version of this document was issued as a working draft in June 2000 for EPA/EMAP's Coastal 2000 initiative. Post Year 2000, the Coastal 2000 Program will continue as EMAP's National Coastal Assessment (NCA). The QAPP has been slightly modified to reflect certain NCA attributes (e.g., personnel); technical aspects remain basically the same. Much of this document was left as originally written for the Coastal 2000 program. As a result, the reader will encounter the terms "*Coastal 2000*, *C2000*, *CM*," etc.; in most cases those terms now imply NCA.

The information described in this document has been subjected to Agency review. Mention of trade names does not constitute endorsement or recommendation for use.

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## ABSTRACT

Attaining consistent reporting in all of the coastal ecosystems in the United States depends on our ability to focus fiscal and intellectual resources on the creation of a National Coastal Monitoring program. To be successful, such a program should be organized at the state level and carried out by a partnership between key federal agencies (EPA, NOAA, DOI, USDA) and state natural resource agencies, as well as with academia and industry. This monitoring program would provide the capability to measure, understand, analyze and forecast ecological change at national, regional and local scales. A first step in the development of this type of program was the initiation of EPA's Environmental Monitoring and Assessment Program (EMAP). This program laid the groundwork for the National Coastal Assessment program, a national estuarine monitoring program organized and executed at the state level.

This document is the Quality Assurance Project Plan (QAPP) for the National Coastal Assessment program. This QAPP was prepared and formatted in accord with the guidelines presented in EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations (EPA QA/R-5), U.S. Environmental Protection Agency Quality Management Staff (U.S. EPA, 1993). According to the type of work to be performed and the intended use of the data, four categories have been defined that vary the level of detail and rigor prescribed for a particular QAPP. This document was prepared for a Category II Project: Complementary Support to Rulemaking, Regulation, or Policy Decisions. Such projects are of sufficient scope and substance that their results could be combined with those from other projects of similar scope to provide the necessary information for decisions.

## QUALITY ASSURANCE PROJECT PLAN

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### A3 DISTRIBUTION LIST

A copy of this QAPP will be distributed to the following personnel who will participate in the collection or analysis of environmental data for the U.S. EPA-EMAP's Coastal 2000 National Survey and to those who are responsible for managerial and quality assurance aspects of the program; distribution may be either in electronic format or hardcopy. The following list does not include everyone who needs or desires a copy of this QAPP, however, the distribution appears broad enough to reach each organization or group with an active role in the program. It would be appreciated if the designated recipients assist in disseminating the document through their networks as needed. Copies also will be made available, upon request, to anyone genuinely interested in the quality program for Coastal 2000.

#### Distribution List:

##### **U.S. Environmental Protection Agency (EPA)**

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William Benson, GED  
Don Cobb, AED  
Brenda Culpepper, NHEERL  
George Craven, GED  
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Ed Decker, Region IV  
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Terrence Fleming, Region IX  
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Eric Hyatt, Region VIII  
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Catherine Libertz, Region III  
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Craig McFarlane, WED  
Stan Meiberg, Region IV  
Gene Meier, GMP  
James Moore, GED  
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Lee Doggett, ME-DEP  
Alan Everett, PA-DEP  
Christian Krahforst, MA-CZM  
Richard Langdon, UNH  
Natie Landry, NH-DES  
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Terry Romaine, LA-FW  
Jim Simons, TPWD  
Jeff Thomas, MS-DWQ

**Puerto Rico Region**

Craig Littlejohn, PR-DNR

**Alaska Region**

Susan Saupe, AK-DEC

**Hawaii Region**

Robert Brock, Univ. HW

**West Region****CA**

Brian Anderson, UC Davis  
Larry Cooper, SCCWRP  
Rusty Fairey, MLML  
Cassandra Roberts, MLML  
Bruce Thompson, SFEI  
Steve Weisberg, SCCWRP

**OR**

Greg Pettit, OR-DEQ  
Mark Bautista, OR-DEQ

**WA**

Casey Clishe, WA-Dept. Ecol.  
Maggie Dutch, WA-Dept. Ecol.  
Ken Dzinbal, WA-Dept. Ecol.

## A4 PROJECT/TASK ORGANIZATION

The U.S. Environmental Protection Agency's (EPA) Environmental Monitoring and Assessment Program (EMAP) is administered through the EPA's Office of Research and Development (ORD), National Health and Environmental Effects Research Laboratory (NHEERL). In response to the need for uniform, comparable environmental data on the Nation's coastal resources, EPA-EMAP conceptualized and developed a five-year initiative, the National Coastal Assessment (also known as Coastal 2000 or C2000) to survey the condition of estuarine and offshore waters by creating an integrated, comprehensive coastal monitoring program among the coastal states (Figure A4-1).

Planning and implementation of Coastal 2000 is under the aegis of the Coastal 2000 Steering Committee which is made up of representatives from EPA-ORD and Office of Waters, EPA-Region Offices, members from the Tribal Operations Council, and officials from state organizations.

The National Coastal 2000 Survey will be managed by the Coastal 2000 Technical Director from EPA-NHEERL's Gulf Ecology Division (GED). U.S. coastal resources will be organized into seven geographical components (not to be confused with EPA Regional Offices) each with designated federal staff to coordinate and oversee implementation by the states within their respective regions:

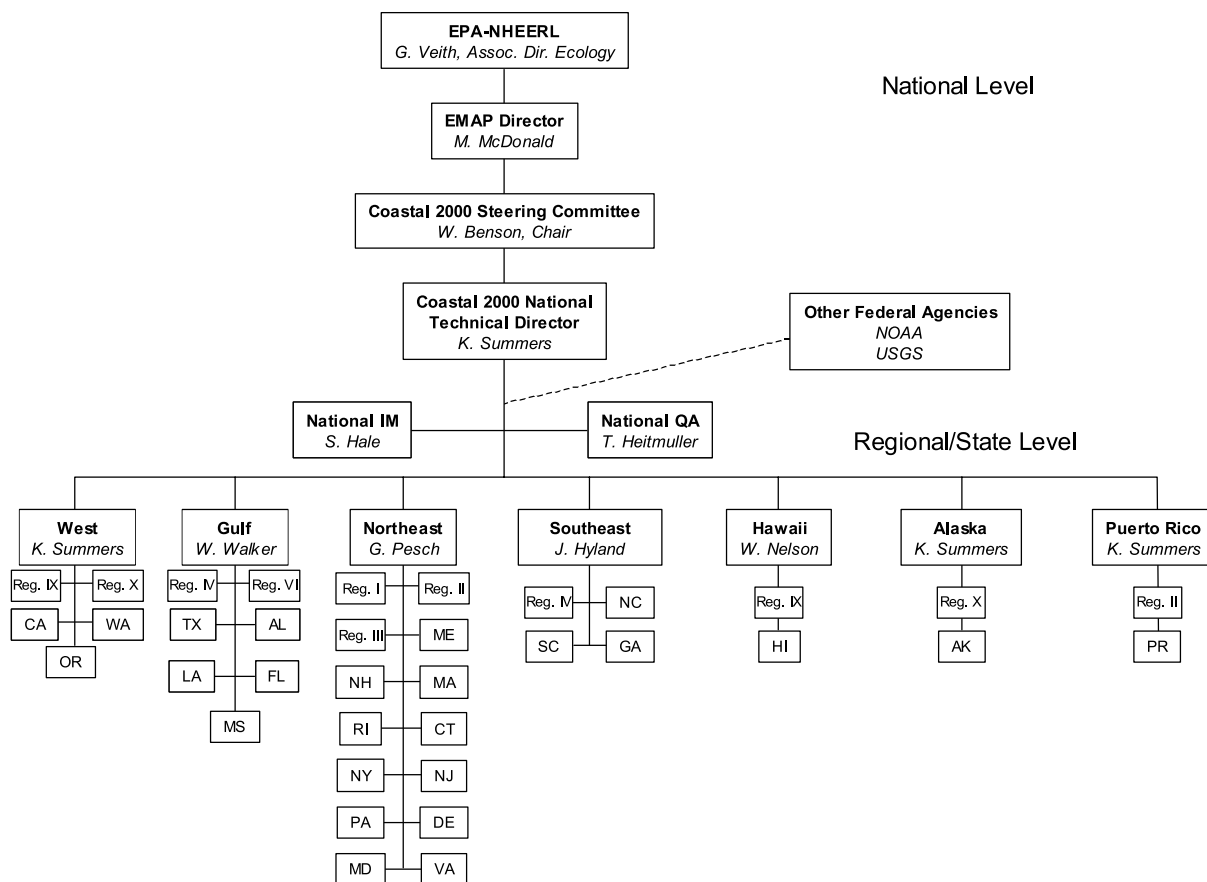
West Region	CA, OR, and WA
Northeast Region	ME, NH, MA, RI, CT, NY, PA, DE, MD, and VA
Southeast Region	NC, SC and GA
Gulf Region	FL, AL, MS, LA, and TX
Alaska Region	AK
Hawaii Region	HI
Puerto Rico Region	PR

Each regional component will have personnel responsible for information management(IM), quality assurance (QA), logistics, and administrative functions; in some instances, one individual may serve in multiple roles. The coastal states will organize in a similar manner; each state should designate a project manager, QA lead, and IM lead.

A list of key personnel and their respective roles in Coastal 2000 is presented in Table A4-1.

**Table A4-1.** List of key personnel, affiliations, and responsibilities for the Coastal 2000 National Survey.

<u>NAME</u>	<u>AFFILIATION</u>	<u>RESPONSIBILITY</u>
<b>National Program:</b>		
Gilman Veith	U.S. EPA-NHEERL	Associate Director, Ecology
M. McDonald	U.S. EPA-NHEERL	EMAP Director
W. Benson	U.S. EPA-GED	Division Director/Chairman, C2000 Steering Committee
K. Summers	U.S. EPA-GED	C2000-National Technical Director
S. Hale	U.S. EPA-AED	C2000-National Information Manager
T. Heitmuller	USGS-NWRC	C2000-National QA Gulf Breeze Office Coordinator
<b>Gulf of Mexico Region:</b>		
W. Walker	U.S. EPA-GED	C2000 Project Officer - Gulf Region
J. Moore	U.S. EPA-GED	C2000 QA Coordinator - Gulf Region
E. Decker	U.S. EPA-Region IV	C2000 Coordinator - Reg. IV
P. Crocker	U.S. EPA-Region VI	C2000 Coordinator - Reg. VI
G. Meier	Gulf of Mexico Program	C2000 Coordinator - GMP (GMP)
J. Simons	Texas Dept. of Parks and Wildlife	State Coordinator - TX
T. Romaine	Louisiana Fish and Wildlife	State Coordinator - LA
J. Thomas	Mississippi Dept. Water Quality	State Coordinator - MS
S. Brown	Alabama Dept. Envir. Mgmt.	State Coordinator - AL
G. McRae	Florida Marine Res. Inst	State Coordinator - FL
L. Harwell	U.S. EPA-GED	C2000 Gulf Region IM Coordinator
<b>Southeast Region:</b>		
J. Hyland	NOAA/NOS	NOAA Project Officer - C2000 Southeast Region



**Figure A4-1.** Management structure for U.S. EPA's Coastal 2000 National Survey.

**Table A4-1. (Continued)**

<b><u>NAME</u></b>	<b><u>AFFILIATION</u></b>	<b><u>RESPONSIBILITY</u></b>
S. Meiberg	U.S. EPA-Reg IV	Region IV C2000 Coordinator
R. Batuik	Chesapeake Bay Program (CBP)	CBP C2000 Coordinator
B. Goode	Georgia Dept. Marine Resources	State Coordinator - GA
R. Van Dolah	S. Carolina Dept. Natural	State Coordinator - SC Resources
J. Overton	North Carolina Dept. Natural	State Coordinator - NC Resources
R. Hoffman	Virginia Dept. Environ. Quality	State Coordinators - VA
D. Smith	Virginia Dept. Environ. Quality	State Coordinators - VA
M. Richards	Virginia Dept. Environ. Quality	State Coordinators - VA
R. Magnien	Maryland Dept. Natural Resources	State Coordinator - MD
<b>Northeast Region:</b>		
G. Pesch	U.S. EPA-AED	EPA Project Officers -
J. Paul	U.S. EPA-AED	C2000 Northeast Region
W. Galloway	U.S. EPA-AED	
C Strobel	U.S. EPA-AED	C2000 Northeast
D. Cobb	U.S. EPA-AED	Coordinators (field, laboratory, and logistics)
J. LiVolsi	U.S. EPA-AED	C2000 Northeast QA Coordinator
H. Buffum	OAQ, Naragansett, RI	C2000 Northeast IM Coordinator
R. Thompson	U.S. EPA-Region I	C2000 Coordinator - Reg. I
D. Adams	U.S. EPA-Region II	C2000 Coordinators - Reg. II
E. Heath	U.S. EPA-Region II	
C. Libertz	U.S. EPA-Region III	C2000 Coordinator - Reg. III
L. Doggett	Maine Dept. Environ.	State Coordinator - ME Protection
N. Landry	New Hampshire Dept. Environ. Sciences	State Coordinator - NH
R. Langdon	Univ New Hampshire	State Coordinator - NH
C. Krahforst	Massachusetts Dept. Coastal Zone Management	State Coordinator - MA
C. Deacutis	Rhode Island Dept. Environ. Management	State Coordinator - RI
C. Olson	Connecticut Dept. Environ. Protection	State Coordinator - CT
K. Chytalo	New York Dept. Environ. Conservation	State Coordinator - NY
B. Connell	New Jersey Dept. Environ. Protection	State Coordinators - NJ
M. Weinstein		
A. Everett	Pennsylvania Dept. Environ. Protection	State Coordinator - PA
B. Anderson	Delaware Dept. Natural Resources And Environ. Control	State Coordinator - DE
E. Santoro	Delaware River Basin Commission (DRBC)	C2000 Coordinator - DRBC

**Table A4-1. (Continued)**

<u>NAME</u>	<u>AFFILIATION</u>	<u>RESPONSIBILITY</u>
<b>West Region:</b>		
W. Nelson	U.S. EPA-WED	EPA Project Officer - C2000 West Region
H. Lee	U.S. EPA-WED	EPA Project Officer - C2000 West Region
C. McFarlane	U.S. EPA-WED	C2000 QA Coordinator-West Region
T. Fleming	U.S. EPA-Region IX	C2000 - Reg. IX Coordinator
L. Edmond	U.S. EPA-Region X	C2000 - Reg. X Coordinator
S. Weisberg	Southern California Coastal Waters Research Project (SCCWRP)	C2000 - SCCWRP Director

**Table A4-1. (Continued)**

<b><u>NAME</u></b>	<b><u>AFFILIATION</u></b>	<b><u>RESPONSIBILITY</u></b>
<b>West Region:</b>		
L. Cooper	SCCWRP	C2000 - West Region IM Coordinator
K. Dzinbal	Washington Dept. Ecology	State Coordinator - WA
G. Pettit	Oregon Dept. Environ. Quality	State Coordinators - OR
M. Bautista	Oregon Dept. Environ. Quality	
R. Fairey	Moss Point Marine Laboratory	State Coordinators - CA
C. Roberts	Moss Point Marine Laboratory	
B. Thompson	San Francisco Estuarine Institute (SFEI)	SFEI Director
<b>Alaska Region:</b>		
D. Landers	U.S. EPA-WED	EPA Project Officer - C2000 Alaska Region
L. Edmond	U.S. EPA-Region X	Region X C2000 Coordinator
S. Saupe	Alaska Dept. Environ. Conservation	State Coordinator - AK
<b>Hawaii Region:</b>		
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C. Lin	U.S. EPA- Region IX	Region IX C2000 Coordinator
R. Brock	University of Hawaii	State Coordinator - HI
(pending)	Hawaii Dept. of Transportation	State Coordinator - HI
<b>Puerto Rico Region:</b>		
K. Summers	U.S. EPA-GED	EPA Project Officer - C2000 Puerto Rico Region
D. Adams	U.S. EPA-Region II	Region II C2000 Coordinators
E.Heath	U.S. EPA-Region II	
G. Craven	U.S. EPA-GED	C2000 Field Coordinators
J. Macauley	U.S. EPA-Region II	
C. Littlejohn	Puerto Rico Dept. Natural Resources	Territory Coordinator - PR

## A5 PROBLEM DEFINITION/BACKGROUND

The U.S. EPA's Coastal 2000 is a five-year effort led by EPA's Office of Research and Development (ORD) to evaluate the assessment methods it has developed to advance the science of ecosystem condition monitoring. This program will survey the condition of the Nation's coastal resources (estuaries and offshore waters) by creating an integrated, comprehensive coastal monitoring program among states to assess coastal ecological condition. Coastal 2000 is being organized and managed by the U.S. EPA National Health and Environmental Effects Research Laboratory's Gulf Ecology Division in Gulf Breeze, FL.

The strategy for Coastal 2000 focuses on a strategic partnership with all 24 coastal states and Puerto Rico. Using a probabilistic design and a common set of survey indicators, each state will conduct the survey and assess the condition of their coastal resources, independently, yet, these estimates can be aggregated to assess conditions at the EPA Regional, biogeographical, and National levels.

The first year's effort (year 2000) involves monitoring estuarine systems in 20 coastal states and Puerto Rico; pilot studies may be initiated in Alaska and Hawaii. In 2001, monitoring will continue in most states and full scale monitoring projects are scheduled for Alaska and Hawaii.

## A6 PROJECT/TASK DESCRIPTION

The purpose of this project is three fold: (1) to utilize the knowledge and expertise of state agencies and local scientists in implementing C2000 to uniformly assess the coastal resources of the Nation; (2) to assist the 24 coastal states and Puerto Rico in the implementation of state-wide coastal monitoring strategies, and (3) to help the states define ambient conditions for coastal waters and support the development of biocriteria in the states.

Under the first year of this five-year program, the U.S. coastal states will work with EPA-EMAP in implementing field and laboratory efforts to meet the first objective. This involves planning of the survey, field collection, laboratory analysis, and information management. Ultimately, the States will be involved in the analysis of collected data to answer the following two questions:

- ☐ What is the condition of the ecological resources in my state?
- ☐ What stressors are associated with degradation of ecological resources in my state?

As the state data are aggregated, the same questions will be posed at regional and national levels.



## A7 DATA QUALITY OBJECTIVES FOR MEASUREMENT DATA

The primary focus of Coastal 2000 is to monitor and document a set of environmental indicators to estimate the ecological condition of the coastal resources of the U. S. or its subregions (e.g., Gulf of Mexico or state waters); secondarily, C2000 is expected to serve as a proving ground to develop research indicators; and finally, C2000 is expected to serve as a proving ground to demonstrate the utility of this approach. These aspects do not coincide all that well with the format of typical research programs designed to answer more singular, focused questions. Therefore, for C2000 project Data Quality Objectives (DQOs), alone, are not adequate to gauge the effectiveness of quality control for the component activities. As with the EMAP-E quality program, the project's emphasis is directed to measurements, therefore, a more appropriate mechanism is to establish quality goals for the individual measurements, or measurement quality objectives (MQOs). Still, there needs to be some unifying level of acceptable uncertainty for the project as a whole in order to define the individual MQOs. C2000 has established target DQOs, based on inference drawn from management's 11 years of experience with EMAP-E. These preliminary DQOs should be considered as a starting point of an iterative process and, therefore, do not necessarily constitute definite rules for accepting or rejecting results, but rather provide guidelines for continued improvement.

C2000 has established DQOs for status estimates. The target DQO for estimates of current status for indicators of condition is as follows:

**“For each indicator of condition, estimate the portion of the resource in degraded condition within  $\pm 10\%$  for the overall system and  $\pm 10\%$  for subregions (i.e., states) with 90% confidence based on a completed sampling regime.”**

Measurement quality objectives for the various measurements made in C2000 (both field and laboratory) can be expressed in terms of accuracy, precision, and completeness goals (Table A7-1). These MQOs were established by obtaining estimates of the most likely data quality that is achievable based on either the instrument manufacturer's specifications, scientific experience, or historical data.

The MQOs presented in Table A7-1 are used as quality control criteria both for field and laboratory measurement processes to set the bounds of acceptable measurement error. Generally speaking, DQOs or MQOs are usually established for five aspects of data quality : representativeness, completeness, comparability, accuracy, and precision (Stanley and Vener, 1985). These terms are described in the context of their application within the C2000 to establish MQOs for each quality assurance parameter.

The relative sensitivity of an analytical method, based on the combined factors of instrument signal, sample size, and sample processing steps, must be documented in order to make a definitive statement regarding detection of an analyte at low levels - for a specific analytical method, what is the lowest concentration at which an analyte's presence can be assured above background noise? For C2000, this question will be answered by calculating Method Detection Limits (MDLs) for each type of analysis. See Section 5.3.2 of Appendix A for a full discussion on determining MDLs. Table A7-2 lists the target MDLs for most analyses to be conducted with C2000 samples. Laboratories will be expected to perform in general accord with these target MDLs.

## **Representativeness**

The concept of representativeness within the context of the C2000 refers to the ability of the project to accurately and precisely characterize the estuarine phenomena along the U.S. Coastline through the measurement of selected environmental indicators. An unbiased sampling design that includes a sufficient number of sampling sites is required to make statistically sound determinations on a system-wide basis; both spatial and temporal aspects of sampling must be considered. For C2000, a probability-based sampling approach (similar to that developed for EMAP) will be employed; the density of stations (at least 50 per state and other special study areas with 100 or more sites) is statistically robust and ensures > 90% confidence that the sampling design is representative of estuarine systems, both on regional and national scales. Temporal variation may be evaluated by repeat monitoring in 2001 for a limited number of sites, or through continued monitoring in following years by the states that elect to do so.

The data quality attribute of representativeness applies not only to the overall sampling design, but also to individual measurements and samples obtained in the course of the monitoring effort. The following examples are illustrations of sample-related factors that might affect the representativeness of the study: the integrity of the sample through periods of storage must be maintained if the sample is to be regarded as representative of the conditions at the time of sampling; the use of QA/QC samples which are similar in composition to the samples being measured to provide estimates of precision and bias that are representative of the sample measurement; and that the samples are collected in an appropriate manner by gear that is specific and standardized for the study.

## **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 Vener, 1985). C2000 has established a completeness goal of 100% for the various indicators being measured (Table A7-1). Given the probability-based design employed by EMAP projects, failure to achieve this goal will not preclude the within-year or between-year assessment of ecosystem condition. The major consequence of having less than 100% complete data from all expected stations is a relatively minor loss of statistical power in the areal estimate of condition, as depicted using Cumulative Distribution Functions (CDFs). The 100% completeness goal is established in an attempt to derive the maximum statistical power from the present sampling design. Based on past years’ experience, failure to achieve this goal usually results from the field crew’s inability to sample at some stations because of logistical barriers, such as insufficient depth, impenetrable substrate, or adverse weather conditions. In the limited number of instances where these may be encountered, extensive efforts will be made to relocate the station or re-sample the station at a later date, always in consultation with program managers. In this way, field personnel must always strive to achieve the 100% completeness goal. In addition, established protocols for tracking samples during shipment and laboratory processing must be followed to minimize data loss following successful sample collection.

## **Comparability**

Comparability is defined as “the confidence with which one data set can be compared to another” (Stanley and Vener, 1985). For C2000 to be effective, the data generated must, first, be comparable within an individual state (i.e., the results for each station sampled within a state must be of uniform quality), and, second, be comparable to that from the other state partners and regions participating in the coastal monitoring (in effect, comparable to EMAP-E data). If the C2000 is to realize its goals, the comparability of field and laboratory procedures, reporting units and calculations, detection limits, and database management processes must all be maintained on the two levels described above. To help ensure and document data comparability, C2000 will utilize various data quality indicators (e.g., performance demonstrations, reference materials, and other QC samples) in conjunction with uniform, standard methods. In addition, interlaboratory calibration exercises will be conducted for certain indicators (e.g., benthic community structure or analytical chemistry) to help evaluate the degree of variability that exist between independent processing laboratories. Details of the above applications will be discussed in following sections of this plan.

## **Accuracy and Precision**

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 (MQOs) for the various indicators are expressed separately as maximum allowable accuracy and precision goals (Table A7-1). Accuracy and precision goals may not be definable for all parameters because of the nature of the measurement type. For example, accuracy measurements are not possible for fish pathology identifications because “true” or expected values do not exist for this measurement parameter (see Table A7-1). In order to evaluate the MQOs for precision, various QA/QC sample will be collected and analyzed for most data collection activities. Table A7-3 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 (see Appendix A). The frequency of QA/QC measurements and the types of QA data resulting from these samples or processes are also presented in Table A7-3. Because several different types of QA/QC are required for the complex analyses of chemical contaminants in environmental samples, they are presented and discussed separately in Appendix A along with presentation of warning and control limits for the various chemistry QC sample types.

**TABLE A7-1.** Measurement quality objectives for EMAP-Coastal 2000 Monitoring indicators. Accuracy (bias) goals are expressed either as absolute difference ( $\pm$  value) or percent deviation from the “true” value; precision goals are expressed as relative percent difference (RPD) or relative standard deviation (RSD) between two or more replicate measurements. Completeness goal is the percentage of expected results that are obtained successfully.

<b>Indicator/Data Type</b>	<b>Maximum Allowable Accuracy (Bias) Goal</b>	<b>Maximum Allowable Precision Goal</b>	<b>Completeness Goal</b>
Sediment/tissue contaminant analyses:			
Organics	35%	30%	100%
Inorganics	20%	30%	100%
Sediment toxicity	NA	NA	100%
Benthic species composition:			
Sorting	10%	NA	100%
Counting	10%	NA	100%
Taxonomy	10%	NA	100%
Sediment characteristics:			
Particle size (% silt-clay) analysis	NA	10%	100%
Total organic carbon	10%	10%	100%
Water Column Characteristics:			
Dissolved oxygen	$\pm 0.5$ mg/L	10%	100%
Salinity	$\pm 1.0$ ppt	10%	100%
Depth	$\pm 0.5$ m	10%	100%
pH	$\pm 0.3$ units	10%	100%
Temperature	$\pm 1.0$ o C	10%	100%
Transmittance	NA	10%	100%
Secchi depth	NA	10%	100%
Water Quality Parameters:			
TSS	10%	30%	100%
Chlorophyll a	10%	30%	100%
Nutrients (nitrates, nitrites, ammonia, and phosphate)	10%	30%	100%
Fish community composition:			
Counting	10%	NA	100%
Taxonomic identification	10%	NA	100%
Gross pathology of fish	NA	10%	100%

**Table A7-2.** Target Method Detection Limits (MDLs) for laboratory analyses of Coastal 2000 samples.

**INORGANICS (NOTE: concentrations in µg/g (ppm), dry weight)**

	<u>Tissue</u>	<u>Sediments</u>
Aluminum	10.0	1500
Antimony	not measured	0.2
Arsenic	2.0	1.5
Cadmium	0.2	0.05
Chromium	0.1	5.0
Copper	5.0	5.0
Iron	50.0	500
Lead	0.1	1.0
Manganese	not measured	1.0
Mercury	0.01	0.01
Nickel	0.5	1.0
Selenium	1.0	0.1
Silver	0.05	0.05
Tin	0.05	0.1
Zinc	50.0	2.0

**ORGANICS (NOTE: concentrations in ng/g (ppb), dry weight)**

	<u>Tissue</u>	<u>Sediments</u>
PAHs	20.0	10
PCB congeners	2.0	1.0
Chlorinated pesticides	2.0	1.0
Total organic carbon (TOC)	not measured	100

**WATER SAMPLES (NOTE: concentrations in mg/L, ppm)**

	<u>Water</u>
Dissolved nutrients:	
NO <sub>2</sub> N	0.005
NO <sub>3</sub> -N	0.005
NH <sub>4</sub> -N	0.005
PO <sub>4</sub> -P	0.002
Chlorophyll a	0.0002 (based on 1.0-L filtered sample)
Total suspended solids (TSS)	2.0

**TABLE A7-3.** Quality assurance sample types, frequency of use, and types of data generated for EMAP-Coastal 2000 Monitoring (see Table 5-4 for chemical analysis QA/QC sample types).

<b>Variable</b>	<b>QA Sample Type or Measurement Procedure</b>	<b>Frequency of Use</b>	<b>Data Generated for Measurement Quality Definition</b>
<b>Sediment Toxicity Tests</b>	Reference toxicant	Each experiment	Variance of replicated over time
<b>Benthic Species Composition:</b>			
Sorting	Resort of sample	10% of each tech's work	No. animals found in re-sort
Sample counting and ID	Recount and ID of sorted animals	10% of each tech's work	No. of count and ID errors
<b>Sediment Grain Size (% silt/clay)</b>	Splits of a sample	10% of each tech's work	Duplicate results
<b>Total Organic Carbon (TOC)</b>	Duplicates and analysis of standards	Each batch	Duplicate results and standard recoveries
<b>Water Quality Parameters</b>			
Hydrolab (similar):			
Dissolved oxygen (DO)	Water-saturated air calibration	Daily	Difference between probe value and saturation level
DO	Air-saturated water measurement	Weekly	Difference between probe value and saturation level
Salinity	Seawater standard (secondary st'd)	Daily	Difference between probe measurement and standard value
Hydrolab (cont):			
pH	QC check with st'd buffers (7&10)	Daily	Difference between probe and standards
Temperature	QC check against st'd thermometer	Daily	Difference between probe and thermometer
Depth	QC check against depth markings on cable	Per use	Difference between probe measurement and standard marks



<b>Variable</b>	<b>QA Sample Type or Measurement Procedure</b>	<b>Frequency of Use</b>	<b>Data Generated for Measurement Quality Definition</b>
<b>CTDs:</b>			
DO, salinity, pH, temperature, depth, and light transmission	Performance verification at certified calibration center	Annually	Differences between instrument response and calibration standards
DO, salinity, pH, and light transmission	Calibration checks at laboratory	Monthly	Difference between instrument response and calibration standards
DO	Comparison to discrete water sample (Winklers); or side-by-side with 2 <sup>nd</sup> instrument	Daily	Difference between instrument DO and reference measurement
Salinity	Comparison to discrete water sample (refractometer)	Daily	Difference between instrument salinity and refractometer value
pH	Comparison to discrete water sample (pH meter)	Daily	Difference between instrument pH reading pH meter reading
<b>Nutrients:</b>			
N-species	Standards and duplicates	Per batch	Relative accuracy and precision
P-species	Standards and duplicates	Per batch	Relative accuracy and precision
<b>Chlorophyll a</b>	Standards and duplicates	Per batch	Relative accuracy and precision
<b>Total Suspended Solids (TSS)</b>	Duplicates	Per batch	Precision
<b>Fish Identifications</b>	Voucher collection verified by taxonomist	Per species	Number of misIDs
<b>Fish Gross Pathologies</b>	Specimens preserved for confirmation	Per occurrence	Number of confirmations

## **A8 PROJECT NARRATIVE**

Element required for QA Category IV documents only.

## **A9 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION**

All field crews that participate in Coastal 2000 Monitoring must first successfully demonstrate team proficiency in each component of field sampling and data collection before they will be authorized to collect actual field data and samples for C2000. Regional C2000 personnel will conduct structured 3 to 4-day field training sessions for those state field teams that are new to EMAP-like projects, as well as, for any state crew that requests a refresher course. These training sessions will usually be organized and presented by the Regional QA Coordinator and Regional Manager and their associates. Field training may be held either at individual state venues or at a centralized location with several states participating; logistics will determine that aspect. The field crews from a given state will be trained collectively as a state team. During the training, crews will be instructed on sampling protocols and methods developed for EMAP-E, then they will actively participate in hands-on exercises conducted in the field for 2-3 days during which all components of the field sampling will be covered. After the crew has developed proficiency in the core field activities, they will be observed and evaluated by the instructors on a pass/fail basis for each component as they conduct a full C2000 field sampling scenario. To be authorized to conduct C2000 field monitoring, the crew must pass in all areas of the certification exercise. The field reviewer will document the crew's performance on Field Crew Evaluation forms that will be turned over to the Regional QA Coordinator and become part of the permanent record. The crews will be informed verbally by the reviewer as to whether they passed or failed the certification exercise. The Regional QA Coordinator should send a written letter to the State Coordinator documenting each crew that passes the field certification exercise.



## A10 DOCUMENTATION AND RECORDS

Coastal 2000 will require that each data generating activity, both field measurements and laboratory analyses, be thoroughly documented in accord with the guidelines that are presented in this section. Field crews will record in-the-field data on hardcopy field sheets and, at a later date, all field data will be transcribed into an electronic format for transmission to a centralized Regional Data Collection Node (see Section B10). Specific formats for both written and electronically recorded data will be prescribed to document the field monitoring and pertinent steps of laboratory analyses. Ultimately, all data will be converted into an electronic format and the data sets archived in the information management system at EPA-AED.

Each state participating as a cooperative agreement partner with the USEPA on Coastal 2000 must submit hardcopies of their entire C2000 study file to the Regional QA Coordinator at the completion of the study. The study file includes: planning documents (QAPP), SOPS, field data sheets, laboratory notebooks or work sheets, study-related correspondence, records of peer reviews or QA assessments (reviews), and reports and publications. These records will be permanently archived by the USEPA.

Metadata (i.e., documentation of pertinent facts that define a process) will be required for each activity that generates C2000 data. Metadata files will be appended to each C2000 data set and include information such as who collected the data; how the data were collected (e.g., equipment/instrument and methodology); definitions of reporting units; QA/QC data; and descriptions of all aspects of data management or data analysis involved with generating the final reported value. In general, metadata should provide a future data user with a sufficient factual history of the entire process, from sample collection to final reported value, so that they can form their own assessment on the value of that data set for their particular purpose. C2000 is currently developing checklists for use in collecting the necessary information to generate metadata files for the core indicators. Data reporting and documentation requirements, presented on a per activity basis, follow.

### FIELD ACTIVITIES

Field crews will rely primarily upon hardcopy field data forms to record most field collected data; however, there may be cases where self contained dataloggers (e.g., SeaBird CTDs) are used to collect information that will be downloaded as electronic files. A generic set of standardized hardcopy forms will be developed for use in each Region (see Appendix E for examples). The individual states will be allowed to slightly modify the format to accommodate their differences in equipment and to include any additional information or parameters that a state may elect to sample; however, the core C2000 field indicators/data will be recorded in an approved, uniform manner. It is preferred that raw data be recorded by ballpoint pen on a real-time basis, but because of the complications with the use of pens in the field, due to wet or damp conditions, it will be acceptable to record field data with a soft-leaded pencil (although it goes against the tenets of QA). There should be a separate form for each measurement type; examples of field data sheet types to be used in Coastal 2000 include:

Station Information  
Hydrographic Profile

Instrument Calibration/Verification (hardcopy)  
Sediment Grab/Benthic Data  
Water Quality Parameters  
Composite Sediment Data  
Fish Trawl  
Fish Data

All field sheets must be identified with station ID code and dated; upon completion of the field entries, the person recording the data will sign each sheet. Field sheets are designed to lead the sampling team through a logical sequence of steps and checks that further ensures sampling protocols are followed. The Field Lead will verify that all field sheets are accounted for and complete prior to departing the sampling station.

All core data recorded on field data sheets will be transcribed into the field computer system within a reasonable time following collection (target period, within a week). To ensure consistency, it is preferable that one person be responsible for the data entry. Data entry will be straightforward and user friendly; the fields in the electronic format will closely resemble the hardcopy raw data forms. The hardcopy data forms filled out for a given station will be compiled into a "station data package" and xeroxed to provide in-house working copies for use by the state as well as the copies required by EPA (study files). The original field sheets should be archived by the sponsoring state agency (e.g., Oregon DEQ), as well as, backup disks for all electronic files; the state will retain these raw data on file for at least a 7-year period. The electronic field data file for a station including the CTD file (when CTDs utilized) will be transferred to the state IM Coordinator for initial validation and formatting review prior to being transmitted to the centralized Regional Information Management Node (IM Node) where additional validation screening and QC checks will be performed before the data are finally forwarded to the EMAP IM Center at EPA-AED.

A systematic approach of sample tracking must be developed to ensure accountability for the handling, storage, and transfer or shipment of the field collected samples. Chain-of-custody documentation (as per GLPs) is not required for this study; however, the system should include the following basic components:

At the collection end -

- a master inventory of all field samples that are expected to be collected (separate list(s) for each sample type and corresponding station IDs), with check off fields providing
- documentation of all samples that are collected (when, and by whom)
- sample transfer information/invoice (where, what, to whom, and when, and by whom samples are transferred or shipped)

Recipient end -

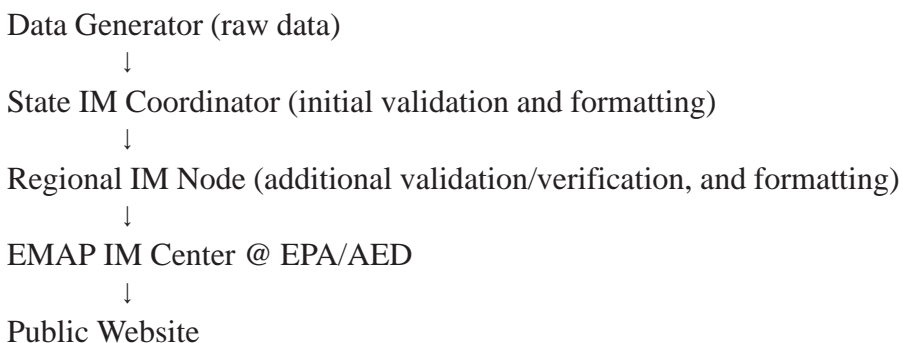
- documentation (sample log-in form) of the person receiving; when and what they receive; and general condition of shipment (e.g., breakage, thawed, etc)

- reconciliation that what was reported shipped was in fact received
- deposition/distribution of samples (e.g., where stored and holding conditions)
- sample release to analysts

As with field data sheets, each state will develop their own sample tracking system by incorporating the above in with the state's existing in-house formats. The field team will retain copies of shipping invoices and the originals will be sent with the samples as they are transferred. The field copies should be compiled into a complete set and submitted to the state field/project coordinator to be archived for at least a period of 7 years. The recipient of the samples (processing laboratory) will inventory the physical samples against the invoice and alert the state laboratory/project coordinator in the event of any missing samples. If a sample is missing, the laboratory should then go through appropriate channels to contact the field team as soon as possible so that they may attempt to locate the sample at their end or possibly re-sample.

## LABORATORY ANALYSES

As with field collected data, the overall flow of data generated from the laboratory analyses will follow the route established in the Coastal 2000 Information Management Plan (EPA, in progress)



The reporting format for electronic files is comma-delimited, ASCII. The specific reporting requirements for each of the major laboratory activities are described in the following.

Analytical and processing laboratories will retain raw data files (e.g., primary standard certification, working standard preparations, instrument calibration records, results of QC check samples/measurements, chromatograms or instrument printouts, and final data calculations) for each indicator for a period of at least 7 years. Upon issuing appropriate advance notification (i.e., minimum of 2 weeks), EPA maintains the authority to access the active files and/or request copies of specific information at any time. In addition the full set of data will be part of the study file of which EPA will receive a copy at the completion of the project; EPA will permanently archive those files.

Sediment and Tissue Contaminants: Copies of the data report for chemical contaminants in sediments and tissue should be submitted (both in hardcopy and computer-readable format) to the State IM Coordinator. The laboratory report should include the analytical results presented on a per batch (groups of 10-20 samples) basis for the major analyte categories (i.e., metals, PCBs and chlorinated pesticides, and PAHs,) including the required QA/QC information specified in Appendix

A. The preferred reporting format is ascii delimited; other formats must be approved by the information management group. Any particular difficulties or irregularities encountered during the analyses should be explained in a case narrative account included in a cover letter accompanying the final data report. The laboratory should deliver the final data report within nine months of initiating the actual analyses of the C2000 samples.

The analytical laboratory will retain all raw data (e.g., sample chromatograms, instrument calibration and QC checks, sample handling and processing logs, and quantification calculations) on file and make them readily available to the C2000 QA personnel if they request access to those data. These records should be retained for a 7-year period following submittal of the final data report. The laboratory will hold the unused portions of samples for at least 1 year following the submittal of the final data report.

Sediment Characterization Analyses: Sediment characterization will include two analyses, percent silt-clay determinations and total organic carbon (TOC) analysis. The laboratory will maintain records on sample storage conditions, analytical balance calibration checks, and instrument calibration checks and laboratory notebook documentation for the preparation of analytical standards (for TOC analysis). These are two independent parameters and will be reported separately. Both analyses are straightforward and, likewise, so are the respective data reports. A copy of each report should be submitted (both in hardcopy and in computer-readable formats) to the State IM Coordinator. The C2000 sediment characterization results should be reported as batches consisting of  $\leq 20$  sediment samples along with the QA/QC samples required on a per batch basis. The data report should be submitted within 6 months following the authorization to proceed with the analysis.

Water Quality Parameters: The suite of water quality parameters will include the analyses of water samples to determine basic nutrient loading, chlorophyll a concentration, and total suspended solids (TSS). The analysis for chlorophyll a requires accurate determination and documentation of the volume of water filtered to provide the sample; this information is recorded on the field data sheets, copies of which must accompany the samples from the field to the laboratory; or, the volume can be recorded on the outside of the petri dish used to contain the filters in the field when the sample is filtered. The data reports for water quality parameters should be submitted (both in hardcopy and computer-readable format) to the State IM Coordinator. Data reports for each of the indicators should include the analytical results along with the specified QC samples (e.g., duplicate analyses, standard reference materials, and blanks); the analyses should be conducted in batches runs consisting of  $\leq 20$  samples per batch. Data report should be submitted within 6 months of authorization to start the analyses. Each participating laboratory must also maintain records of sample storage conditions (e.g., temperature log), standard preparations, and instrument calibrations; these records will be made available upon request to C2000 management/QA personnel.

Sediment Toxicity: Toxicity tests will be conducted with amphipods exposed to test treatments of surficial sediment collected from each station. The laboratory must maintain written records on sample material, test organisms, and the actual testing; the required information for each of these areas is described in the EMAP-E Methods Manual . All data entries must be in ink and initialed. Records required on test sediments includes documentation of sample receipt and holding conditions, period of holding, and dry sieving procedures and dates. Records on test organisms will be maintained in organized laboratory notebooks or on printed data sheets including date of receipt and source of organisms, holding and acclimation regimes, observation on general health of

organisms, and results of reference toxicity tests with batches of organisms (used to construct control charts) . For the actual testing, records must be maintained to include a description of test water (source, salinity, pH, etc.); source and description of control/ reference sediment; testing conditions (e.g., lighting, temperature, aeration, etc.); daily observations during test (e.g., excessive control mortality, aeration malfunctions, general status of test); and at the termination of test, record of survival in each test container, DO and pH in representative sample of controls and treatment containers, and record of length measurement for a random sampling of test organisms. These data and records will be made available to C2000 management or QA personnel upon request. Data reports for toxicity tests will include the survival of test organisms for each test treatment and its control. Survival should be expressed as the total number of organisms alive at the termination of the exposure period for the replicated treatments (i.e., as total survival summed from the five replicate containers). The reported results should not be corrected for control mortality; that will be the responsibility of data analysts at either EPA or the states. The laboratory should provide a written narrative describing the source of organisms and the control sediment. Also, the narrative should detail any deviations from testing protocol or any other information that may be of value in the interpretation of the data.

Benthic Communities Assessments: Macroinfaunal community structure will be assessed from samples collected at each station. Macro organisms will be sieved from sediment grabs and preserved in the field for later laboratory evaluation. Laboratory data (i.e., major taxon group sorts, species identifications and counts, and QC checks) will be recorded on printed worksheets based on the example sheet suggested by the C2000. These raw data will be maintained by the laboratory and be made available upon request to C2000 management/QA personnel. The benthic laboratory will transcribe the hardcopy data into a standardized electronic format jointly developed and agreed to by the participating agencies. The data report should list by station, the taxon groups to genus species (within reason; extremely challenging IDs to be resolved upon consultation with C2000 management) and the number of individual organisms per group. The data report should be submitted (both in hardcopy and computer-readable formats) to the State IM Coordinator. The QC data should be summarized in a hardcopy table or narrative and included with the final data package. Also, a narrative report should be included in a cover letter explaining any difficulties or irregularities encountered during the assessments (e.g., taxonomic problems, sample integrity, extraneous material in the samples).



## **GROUP B MEASUREMENT/DATA ACQUISITION**

### **B1 SAMPLING PROCESS DESIGN (EXPERIMENTAL DESIGN)**

Coastal 2000 is a large-scale, comprehensive environmental monitoring strategy designed to provide regional characterization of the Nation's coastal resources (estuaries and offshore waters) by creating an integrated, comprehensive coastal monitoring program among the coastal states to assess coastal ecological condition. The strategy for C2000 focuses on a strategic partnership with all 24 coastal states. The overall design for the program is based on EPA-EMAP's sampling approach that uses Geographic Information System (GIS) technology to probabilistically generate sampling locations (Bourgeois et al, 1998). Base sites for the first year's monitoring (2000) will be distributed through 24 contiguous coastal states and Puerto Rico; each will have at least 35 randomly selected sites. In addition, specific areas have been designated for more intensive sampling, including San Francisco Bay; Puget Sound; Chesapeake Bay; and the states of Alabama, Florida, South Carolina, and Texas. The field sampling for some of these areas will continue in the summer of 2001. Monitoring activities will be initiated for Alaska and, possibly, Hawaii in 2001.

There are three basic phases to EMAP's Coastal 2000 program: field collection of environmental data and samples; laboratory analyses of samples; and data analysis and assessment.

## Field Collection of Environmental Data

Field sampling will be performed independently by each state; cooperating federal agencies may augment the states' field efforts, both in terms of equipment and personnel. Field crew members will be personnel selected primarily from the respective state environmental agencies. In most instances, 3 to 4-person field crews will conduct the sampling from smallcraft (typically, 20-25 ft) during a seasonal window spanning from July to mid-September. Sampling is planned as a one-time event per station (i.e., no scheduled repeat sampling for the base sites). However, it is likely that the states, either on their own or in conjunction with other agencies, will continue some elements of the environmental monitoring in following years. The field teams will be provided with randomly selected coordinates of latitude and longitude for each of their sampling locations. The crew will locate the sites by use of Global Positioning Satellite System (GPS), preferably, differential. Agreement between the given coordinates and the actual in-the-field siting of a sampling station should be within 0.02 nautical miles (nm), which is equivalent to a radius of approximately 120 ft. Most GPS units display the distance from an entered waypoint as 0.00 nm, therefore this is a convenient unit to use for noting distance from the given coordinates.

Field activities performed at each site should require approximately 2-3 hours per site, therefore, a team can expect to sample two stations in a normal day; of course, this is subject to such factors as weather, seas, and travel distance. At each sampling site, all C2000 crews will uniformly collect a core set of data and samples following EMAP-E methods and protocols. Core field data/samples include (these will be discussed in greater detail in following sections):

- instantaneous water column profile (DO, pH, salinity, temperature, depth, transmittance, and clarity)
- water quality parameters (nutrient load - P and N species; chlorophyll a content; total suspended solids (TSS))
- surficial sediment, top 2-3 cm, (chemical contaminants - organics and trace metals; sediment toxicity; total organic carbon, TOC; and grain size)
- benthic macroinvertebrate community structure (richness and abundance)
- fish/shellfish (community structure - richness and abundance; total lengths; pathological examination; chemical contaminants - organics and trace metals)
- habitat (general habitat-type; presence/absence: exotic species, submerged aquatic vegetation, and anthropogenic debris or perturbation).

Each state field crew has the option of gathering additional environmental information, as long as those activities are not given precedence over the core activities.

Samples collected from the field may be temporarily held at the field staging centers, under appropriate conditions for 1-5 days, to await shipment (or delivered) to centralized storage facilities or processing laboratories. Sample handling and storage guidelines are presented in Table B1-1.

## Laboratory Analyses of Samples

### National Laboratories

Because some states may not be adequately equipped and staffed to conduct certain highly specialized analyses related to several of the core C2000 indicators, and/or the cost to contact analyses for a limited number of samples may be prohibitive, the U.S. EPA will designate several “National Laboratories” to conduct these analyses for any state which so elects, at a nominal cost per sample. This approach would also ensure data uniformity between the participating states. At this time, National Laboratories are being planned for the following core activities:

- analytical chemistry (organic and metal contaminants in both matrices)
- benthic community structure
- nutrient analyses
- sediment toxicity testing

The designated National Laboratories must comply with the QA/QC requirements described in this document.



### In-State Laboratory Analyses

For any analyses other than those conducted through the above National Laboratories, each of the states participating in C2000 will be responsible for the arrangements to analyze the field samples that they collect. These agreements will be negotiated by the individual states, not through the EPA. Some analyses may be conducted in-house by state agency laboratories or universities, while others are contracted out to private laboratories or other states. However, any laboratory selected to conduct analyses with C2000 samples must demonstrate that they can meet the quality standards presented in this QAPP. Later sections will address initial demonstrations of technical capability and performance evaluations.

When possible, field samples should be promptly shipped (generally within a week) to the approved analytical or processing laboratories. These facilities are generally better geared to properly hold the samples while they await analyses. At the laboratory, samples will be processed in accord with EMAP QA/QC guidelines. The results will be submitted to the sponsoring state in a final data report.

Each laboratory is expected to review their final data for completeness, accuracy, and precision to assure that the basic quality criteria are met prior to submitting their final data report to the state. At the state-level, the data will receive further review and validation as data sets are formatted for transmission to the regional data collection node. Regional QA Coordinators will make the initial approval/disapproval of data sets and, when warranted, assign appropriate qualifier codes. After data have been qualified, data analysis and assessments then can be jointly developed through the cooperation of state and federal environmental scientists. EPA will be responsible for posting the finalized C2000 data and supporting metadata on the Internet and making them available to interested parties. Data sets that pass project QA/QC will be posted without further qualification; data that do not pass project QA/QC, but that are characterized by minor deficiencies will be flagged with appropriate qualifier codes so that individual data users can evaluate the quality of the data for their specific needs; data that consistently fail project QA/QC standards may be dropped altogether from the C2000 database. Before data are dropped, the problematic issues will be discussed between the Regional and National QA Coordinators, EPA Project Officer, and the state's Project Manager for a consensus resolution (more details on this follow in later sections).

**TABLE B1-1.** Sample handling and storage guidelines for EMAP Coastal 2000 Monitoring.

<b>SAMPLE TYPE</b>	<b>CONTAINER</b>	<b>FIELD HOLDING</b>	<b>LAB STORAGE</b>	<b>MAX HOLDING</b>
<b>Sediment:</b>				
Organic contaminants	Appropriately cleaned glass jars or I-Chem	Wet ice (4°C)	Freezer (-20°C)	1 year
Inorganic	125-cc Nalgene contaminants	Wet ice (4°C)	Freezer (-20°C)	1 year
Total organic carbon	Small glass jar	Wet ice (4°C)	Freezer (-20°C)	1 year
Silt/clay	125 cc Nalgene	Wet ice (4°C)	Refrigerator (4°C)	1 year
<b>Water Quality:</b>				
Chlorophyll	25mm GF/F in plastic petri dish (foil wrapped)	Dry ice	Ultra freezer (-50°C)	6 months
Nutrients	60 cc Nalgene bottle	Dry ice	Ultra freezer (-50°C)	6 months
Total suspended solids (TSS)	1-liter Nalgene	Wet ice (4°C)	Refrigerate (4°C)	3 months
<b>Biota:</b>				
Benthos (0.5 & 1.0 mm sieved)	500-1000 cc wide-mouth Nalgene	10% buffered formalin	Transfer to 70% ethanol	Indefinitely
Fish contaminants	Individuals foil wrapped and combined in Ziploc bag	Wet ice (4°C)	Freezer (-20°C)	1 year
Histopathology specimens	As per sample size	Dietrich's fixative	Transfer to 70% ethanol	6 months

## B2 SAMPLING METHODS REQUIREMENTS

Procedures for field collection of environmental samples and data for the Coastal 2000 Monitoring are based on methods developed by EMAP-Estuarines over its past 11 years of experience with large-scale, regional monitoring projects (e.g., EMAP-E Province Monitoring, the Mid-Atlantic Integrated Assessment, MAIA, and the Western Pilot Coastal Monitoring). EMAP sampling methods are described in several documents including EMAP-Estuarines Field Operations Manuals prepared for the Virginian, Louisianian/West Indian and Carolinian Provinces (Strobel et al, 1990; Macauley and Summers, 1991-95; Kokkinakis and Hyland, 1992- 94). Also, the Southern California Coastal Waters Research Program (SCCWRP) and California Department of Fish and Game prepared field SOPs (SCCWRP, 1995; SWRCB, 1994) specific for monitoring activities conducted in the Californian Province.

EMAP Provinces or geographic regions are differentiated by unique conditions (e.g., climate, depth, bottom type, tidal influence, biota, etc.), therefore, on occasions, it is necessary to modify “standard” EMAP field procedures to meet the needs particular to a region or subregion. Such modifications are generally approved as long as the altered procedures meet the general guidelines of established protocol and adhere to the spirit of the QA/QC established for EMAP so that the resultant data remain comparable to that collected by standard procedures.

A flexible study design is a necessity for the C2000 due to the multitude of independently equipped state field teams and because of the regional difference in estuaries the vast geographic sweep of U.S. coastal resources (e.g., the deep harbors of Puget Sound compared to the tidal flats in South Carolina). To accommodate these needs, this QAPP will set minimum performance criteria or QC requirements that field crews must meet in order to collect data that are comparable, but it will not require that the field procedures necessarily be identical. The following sections describe the general methods and procedures for each core sampling activity. Field crews should adhere to these methods as much as possible. Additional QA/QC details for the procedures will be discussed in later sections.

### Site Location

The randomly selected sampling locations for each state (or specific study area) will be provided to the field crews as coordinates of latitude/longitude in degrees-minutes, expressed to the nearest 0.01 minute (i.e., 00° 00.00'). The crews will use GPS to locate the site. **The acceptable tolerance goal for siting is that the sampling station be established within 0.02nm (±120 ft) of the given coordinates.** This reflects the accuracy expected from a properly functioning GPS unit of the caliber that will be used for the study. Note: the lat/lon coordinates of the actual anchorage, not the “intended or given” coordinates, will be recorded on the field sheet as the sampling location. The GPS’s performance should be verified on a daily basis; those details will be discussed in Section B5.

Field crews will strictly adhere to the above guidelines for siting the station, unless there are substantiated reasons that prevent sampling within that defined area. Because EMAP’s probabilistic sampling design is unbiased, potentially, some of the generated sites can fall in locations that are not amenable to sampling ( e.g., shallow conditions, inaccessible, rocky bottom, etc.). Upfront planning by the field team can help resolve these potential problems before they are encountered on the actual day of sampling. Coordinates of the random locations are made available to the teams months in

advance of the field monitoring in order that they have adequate opportunity to formulate logistical plans. The reasonable first step is to plot the given sites on NOAA nautical charts to ascertain the spatial distribution of the sites, then reconnoiter (on paper) the charted locations for obvious problem situations (e.g., water depth, hazards to navigation, etc.). If suspect sites are encountered in this exercise, it is suggested that a field reconnaissance be conducted well ahead of the scheduled sampling to determine actual conditions at the site. If an intended site location presents an obvious problem, the situation must be reported to the State Team Coordinator and/or Regional EPA Project Officer, who, in turn, will discuss the specifics with appropriate C2000 personnel for resolution options. Depending on the nature of the situation, the EPA Project Officer may elect to relocate the site within an acceptable range of the original location, or the site may be dropped from the sampling. Decisions on this level (i.e., significant changes to the sampling design) are to be made only by the EPA Project Officer, not by the field teams.

Field teams, however, will have a limited degree of onsite flexibility to relocate sampling sites when confronted with unexpected obstacles or impediments associated with locating within the  $\pm 0.02'$  guideline. The crew chief may, for good reason (e.g., danger or risk to crew, shallow conditions, excessive rocky or shelly bottom, currents, man-made obstructions), move the station to the nearest location from the intended site that is amenable to conduct the sampling; every effort must be made to relocate to an area that appears similar in character to that of the intended site. For example, if the intended site was in the channel of a stream, then the relocation should be as near to that situation as possible; it should not be relocated along side the stream bank. When it is necessary to relocate the site  $>0.02'$ , the reason for shift must be documented in the field record. Any site relocation that exceed  $0.05'$  (300 ft) will be flagged and reviewed before any data collected from the station are acceptable for inclusion to the study database. At times, crews might experience difficulty in obtaining a “good grab” when collecting sediment due to the nature of the bottom at their established site. In these situations, even after they have collected the water quality samples and data, it is permissible for them to move around within the 120-ft radius to locate more favorable sediment conditions without having to resample the water quality indicators.

### **Water Measurements**

The first activities that should be conducted upon arriving onsite are those that involve water sampling and water column measurements; these samples/data need to be collected before disturbing bottom sediments.

### Hydrographic Profile

Water column profiles will be performed at each site to measure basic water quality parameters of dissolved oxygen (DO), salinity, temperature, pH, and depth. At least one measurement of light attenuation, either transmittance or PAR, will be conducted; in addition, secchi depth also will be measured at each station.

Basic water quality parameters will be measured by using either a self-contained SeaBird CTDs to electronically log a continuous profile of the water column or by using hand-held multiparameter water quality probes (e.g., Hydrolab Surveyor or YSI Sondes) with cable connection to a deck display. Prior to conducting a CTD cast, the instrument will be allowed 2-3 minutes of warmup while being maintained at near the surface, after which, the instrument will be slowly lowered at the rate of approximately 1 meter per second while performing the down cast. In cases where hand-held probes are used to profile the water column, individual measurements at discrete intervals (with sufficient time for equilibration) will be taken as follows:

Shallow sites ( $\leq 2$  m) - every 0.5 m interval;

Typical depths ( $>2<10$  m) - 0.5 m (near-surface) and every 1-m interval to near-bottom (0.5 m off-bottom);

Deep sites ( $>10$  m) - 0.5 m (near-surface) and every 1-m interval to 10 m, then at 5-m intervals, thereafter, to near-bottom (0.5 m off-bottom).

Near-bottom conditions will be measured at 0.5 m off bottom with both instrument types by first ascertaining on-bottom (e.g., slake line/cable), then pulling up approximately 0.5 m. Allow 2-3 minutes for disturbed conditions to settle before taking the near-bottom measurements. The profile will be repeated on the ascent and recorded for validation purposes, but only data from the down trip will be the reported in the final data.

Measurements of light penetration, taken by hand-held light meters, will be recorded for conditions at discrete depth intervals in a manner similar to that for profiling water quality parameters with the hand-held probe. The underwater (UW) sensor will be hand lowered at the regime described and at each discrete interval, the deck reading and UW reading will be recorded. If the light measurements becomes negative before reaching bottom, the measurement terminates at that depth. The profile will be repeated on the ascent.

Secchi depth will be determined by using a standard 20-cm diameter black and white secchi disc. The disc will be lower to the depth at which it can no longer be discerned, then it is slowly retrieved until it just reappears; that depth is marked and recorded as secchi depth (rounded to the nearest 0.5 m).

## Water Quality Indicators

The water column will be sampled at each site for the determination of dissolved nutrients (N and P species), chlorophyll *a* concentration, and total suspended solids by using a Van Doren sampler or Niskin bottle. Depending on depth at the sampling station, water samples will be collected as follows:

Shallow sites (<2 m) - sample at 0.5 m (near-surface) and 0.5 m off-bottom;<sup>1</sup>

Standard site (>2m) - sample at 0.5 m (near-surface), mid-depth, and 0.5 m off-bottom;

<sup>1</sup> Unless the depth is so shallow that the near-surface and near-bottom overlap; then sample mid-depth, only.

An approximate 3-liter subsample will be pulled into a clean, wide-mouth Nalgene container to provide water for the remainder of the sample processing which essentially is filtration, with the filtrate becoming the dissolved nutrient sample and the filters retained for the chlorophyll *a*. Unfiltered water will be taken for TSS samples.

### Chlorophyll *a*.

A disposable, graduated 50-cc polypropylene syringe fitted with a stainless steel or polypropylene filtering assembly will be used to filter the site water through 25-mm GF/F filters; the volume of water filtered must be documented. If conditions allow (suspended solids load), up to 200 ml of site water should be filtered for each chlorophyll sample (for a 50-cc syringe, that equates to 4 refills). At each refill, carefully detach the filter assembly and fill the syringe to the mark, replace the filter and continue with the filtration until the desired volume has been processed. Use tweezers to carefully remove the filter from its holder and fold once upon the pigment side, then place it in a prelabeled, disposable 50 or 60-mm petri dish and cap. Record the volume of water filtered on both the petri dish and on the field form. Wrap the petri dish in aluminum foil and label with station ID (Sharpie ok); place the foil wrapped packet in a small instant-freeze chamber (small styrofoam ice chest with several pounds of dry ice). Repeat the filtering process for second sample and store filter in the same petri dish containing the first sample. The samples must remain frozen until time of analysis. Discard the used syringe. Rinse the filtering assembly with deionized water and store in a clean compartment between sampling stations (a small tacklebox makes a good carrying kit for supplies and equipment used in this activity).

### Dissolved Nutrients

Approximately 40 ml of filtrate from the above chlorophyll filtration (surface water) will be collected into a prelabeled, clean 60-ml Nalgene screw-capped bottle and stored in the dry ice freezing chamber. Before placing sample in the freezer, record the approximate salinity ( $\pm 2$  ppt) on the container; this is a convenience for the analyst who will perform the nutrient analysis. Depending on the analytical instrumentation used, matrix matching of solutions (e.g., standards or wash solutions) may be required for certain of the analytes. The salinity value can be obtained from the water column data or by refractometer reading of the actual water sample taken by Van Doren/Niskin. The nutrient samples should remain frozen until time on analysis.



## Total Suspended Solids.

Approximately 1 liter of unfiltered seawater will be collected at the depths described above. The samples will be held in a 1-L polypropylene bottles on wet ice in the field and stored at 4°C to await laboratory determinations.

## Benthic Infaunal Community

Benthic infaunal samples will be collected using either a 0.04 m<sup>2</sup> or a 0.1 m<sup>2</sup> (bite size) Van Veen grab sampler. These two grab sizes represent what has historically been used during previous EMAP monitoring projects; the 0.04 m<sup>2</sup> grab for activities conducted in the Atlantic and Gulf of Mexico in the eastern regions and the 0.1 m<sup>2</sup> for West Coast activities. The collected sediment grab will be immediately processed aboard by sieving the entire contents of the grab through a series of standard sieves/screens. For the West Coast samples, a series of “stacked” 1.0 and 0.5-mm screens will be utilized; for Atlantic and Gulf samples will be processed using only the 0.5-mm seive. Organisms retained on each screen will be gently transferred to separate labeled, wide-mouth, Nalgene containers and preserved with buffered formalin (7-10% final concentration). The formalin preserved samples will be forwarded to a benthic ecology laboratory for additional processing, sorting, identification, and counting. At the laboratory, it is recommended that the formalin-fixed samples be transferred to 70% ethanol within 2 weeks of field collection to avoid undue deterioration of sample integrity that may further complicate identification (e.g., loss of heads/appendages and erosion of shells or exoskeletons).

EMAP will not require replicate grabs be taken from each site sampled with the standard 0.1 m<sup>2</sup> or 0.04 m<sup>2</sup> Van Veen. The sample design for the Coastal Monitoring (i.e., 50 random sites/ state) provides sufficient replication to characterize the benthic community assemblages at regional scale and the size of the grab further ensures a representative sample for the site.

There apparently will be some situations in which the large (0.1 m<sup>2</sup>) grab cannot be used (e.g., inaccessible shallows or small streams). Sampling options are currently being developed for these sites. One option related to benthic collection at these sites is to use a small coring cylinder (approx 6 inch diameter) to take the sample. In these cases, three replicate cores will be collected and combined for sieving; this will provide a sample on the order of that collected by the larger Van Veen grab. The use of alternative gear (e.g., coring tubes) must be documented on the field data sheet with a full description of both the gear and techniques.

## Composited Surficial Sediment

At each site, multiple sediment grabs will be taken by van Veen sampler and the surficial sediment layer (top 2-3 cm) will be collected by spatula or scoop and composited to provide sediment for the analyses of chemical contaminants, total organic carbon (TOC), toxicity testing, and grain size determinations. The number of grabs required to yield an adequate volume of composited sediment depends on the surface area described by the particular grab; however, surficial sediment from a minimum of three grabs should be composited for the final sample. Surficial sediment from the individual grabs will be combined in a clean, high-grade stainless steel or Teflon vessel. Between grabs, the container of composited sediment will be held on ice and covered with a lid to protect the sample from contamination (e.g., fuel or combustion products). Each addition of sediment to the

composite will be blended in by stirring and the final mixture will be stirred well to ensure a homogenous sample before sub-samples for the various analyses are taken as follows:

Organic chemical contaminants - approximately 300 cc of composited sediment will be placed in a clean, prelabeled, glass wide-mouth, 1-pint Mason jar or I-Chem jars; fill containers to approximately 75% of capacity to allow for expansion during freezing - **DO NOT OVERFILL; full jars tend to break when frozen !!!** (see B5 for QC requirements). The sample will be held on wet ice aboard and, upon transfer to shore storage, the sample should be frozen unless it is scheduled for extraction within 7 days; in that case, the sample may be held at 4°C to await processing.

Inorganic chemical contaminants - approximately 200 cc of composited sediment will be placed in a clean, prelabeled, wide-mouth Nalgene jar. The sample will be held on wet ice while aboard and, upon transfer to shore storage, the sample should be frozen unless it is scheduled for digestion within 7 days; in that case, the sample may be held at 4°C to await processing.

Toxicity testing - approximately 2000 - 4000 cc (depends on the number of toxicity tests to be performed) of composited sediment will be placed in a clean, prelabeled, wide-mouth Nalgene jar. The sample will be held on wet ice aboard and, upon transfer to shore storage, the sample will be held at 4°C (sample is not to be frozen) to await further processing and initiation of testing within 30 days of collection.

TOC - approximately 30 cc of composited sediment will be placed in a small, clean, prelabeled glass bottle/jar. The sample will be held on wet ice aboard and upon transfer to shore storage, the sample should be frozen to await further laboratory analysis.

Grain size determination - approximately 120 cc of composited sediment will be placed in a clean, prelabeled, wide-mouth polypropylene jar. The sample will be held on wet ice aboard and, upon transfer to the shore storage, the sample will be held at 4°C (sample is not to be frozen) to await further laboratory processing.

Additional quality control measures pertaining to the composited sediment samples are described in Section B5.

### **Habitat**

Several observations will be made in the field to document certain attributes or conditions of the site that will help to characterize the overall ecological health. Observations will be made and noted for the occurrence of submerged aquatic vegetation (SAV), the presence of marine debris, and on West Coast, the occurrence of macroalgae beds/mats. Also, if there is obvious evidence of disruptive anthropogenic activities (e.g., dredging or landfill activity), these observations should be noted with a brief description on the appropriate field form.



## Exotic Species

The introduction of non-indigenous organisms and plants has the potential to upset the balance within an ecological system through opportunistic marauding. Coastal 2000 is interested in documenting the occurrences of this condition and will designate several species of both flora and fauna as exotics to be monitored for as laboratory evaluations are conducted; field crews are not expected to make onsite evaluations for exotics.

## Fish and Epibenthic Invertebrate Collection

Fish trawls will be conducted at each site, where possible, to collect fish/shellfish for community structure and abundance estimates, target species for contaminant analyses, and specimens for histopathological examination. Historically, standard EMAP trawls have been conducted by using a 16-ft otter trawl to conduct least one trawl for a  $10 \pm 2$  minutes duration to yield valid community structure data (i.e., the fish data on richness and abundance and individual lengths). Additional trawls of unspecified durations may be conducted to supplement the sample for contaminant analyses. Although not required, it is strongly suggested that the vessel used for trawling be equipped with a boom or A-frame assembly and a powered winch. In situations where the use of nominal craft is prohibited (e.g., narrow stream or shallow conditions), it is possible to manually deploy and retrieve a small trawl, but it is not advised as routine procedure. Trawling should be the last field activity that the crew performs while onsite because of their disturbance to conditions at the site.

In open water, the trawl should be conducted in a straight line with the site location near center. Additional trawls can be taken along the same general line by going in the opposite direction; however, tides and seas conditions may dictate the direction of the trawl. Timing of the trawl begins after the length of towline has been payed out and the net begins its plow. The speed over bottom should be approximately 3-4 knots. When possible, conduct the trawl for the entire 10-minute period, after which the boat will be placed in neutral and the trawl net retrieved and brought aboard. Contents of the bag will be emptied into an appropriately sized trough or livebox to await sorting, identifying, measuring, and sub-sampling. Every effort will be made to return any rare or endangered species back to the water before they suffer undue stress.

## Community Structure

Fish from a successful trawl (fulltime on bottom with no hangs or other interruptions) will first be sorted by species and identified to genus species. Up to thirty individual per species will be measured by using a fish measuring board to the nearest centimeter (fork length when tail forked, otherwise overall length - snout to tip of caudal). The lengths will be recorded on a field form and a total count made for each species. All fish not retained for histopathology or chemistry will be returned to the estuary. Invertebrates will sampled as directed later (still under review, will differ from region to region).

Coastal 2000 recommends that states without established fish inventorying programs adhere to the above guidelines in order to collect comparable fish community data. However, some states already have regimes in place for continuing, comprehensive fish studies that do not comply with EMAP standards. C2000 will review these states' programs on a case-by-case basis and may allow a

state to substitute their procedures for the EMAP standard.

### Contaminant Analyses

Several species of demersal fishes will be designated as target samples for analyses of chemical contaminants in whole-body tissue. Specific target lists will be generated for each region that generally include flatfishes and other commonly occurring demersal species from higher trophic levels. At sites where target species are captured in sufficient numbers, five to ten individuals of a species will be combined into a composited sample. The fish will first be measured and recorded on the sampling form as chemistry fish. The fish will then be rinsed with site water, individually wrapped with heavy duty aluminum foil (the length of each individual fish should be imprinted on the foil wrap to facilitate the possible later selection of specific individual at the laboratory), and placed together in a plastic, Ziploc bag labeled with the Station ID Code and a Species ID Code (e.g., the first four letters of both the genus and species). The fish chemistry samples will be held on wet ice in the field until they are transferred to shore where they will be frozen to await laboratory analysis.

### **Gross Pathology**

All fish will be screened in the field for external gross pathologies as they are measured and counted for the community structure evaluation. Each fish will be briefly examined for any obvious external conditions such as lesions, lumps, tumors, and fin erosion; also, the gills will be examined for discoloration or erosion. Any fish that exhibits a pathological condition will be saved for further laboratory histopathological evaluation. A generic description of the observed condition will be recorded by field personnel on the Fish Data form; then, the specimen will be tagged and immediately preserved in Dietrich's solution to await shipment to the laboratory. Each fish to be preserved will have its body cavity opened to expose internal tissues to the fixative. Stainless steel surgical scissors will be used to open the body starting at the anal pore and cutting anteriorly through the body wall, taking care not to cause undue damage to the internal organs; the cut should continue through the thoracic region and over to the gill slits. The body cavity should then be spread apart (popped open) by hand to further ensure the fixative floods the internal organs. The tagged fish is then added to an appropriate container (e.g., a 1-2 gallon plastic bucket with enough Dietrich's to completely cover the specimen). As long as fish are well tagged, multiple samples can be held in a common container.

### B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

A comprehensive project such as EMAP-Coastal 2000 requires a structured system to ensure that all pertinent data are documented and that samples are appropriately labeled, handled, stored, and transferred through all phases from field collection to final analysis. The following section will outline data/sample accountability guidelines for the project. Although standard formats for data/sample collection and reporting will be established for field and laboratory activities, not all aspects of sample handling will be addressed by the forms alone. Therefore, additional written documentation is required to augment cradle to grave history for sample possession within C2000.

#### Field Data

##### Field Data Forms

C2000 field crews will record most of their raw field data on hardcopy data sheets (see Appendix E for examples). Some crews may also use instrumentation with self-contained datalogging capabilities (e.g., SeaBird CTD units) that store values in electronic format which can be downloaded later as electronic files. To maintain uniformity across the various states within a region, the template for field data sheets will be designed by regional QA and IM personnel to systematically query the crew for all pertinent information required to document the conditions and activities performed for a sample collection. All pertinent field data will eventually be transcribed into an electronic format standardized for each region so that the data can be transmitted to the regional data collection node; therefore the field sheets and electronic tables should closely resemble one another.

##### Site/Sample Identity Codes

Regional IM Coordinators will provide each state team with a list of unique site and sample identity (ID) codes. The site codes will be configured in a series specific to each state to include a state's two character abbreviation, year designator, and a sequential numerical series; for example, Florida's sites for year 2000 will be coded **FL00-0001** through **FL00-1000** (or however many sites are designated). Sample ID codes will simply be an abbreviated code to describing the sample type; for example, the sample for sediment organics would be **SO**. Together, the two IDs, **FL00-0001-SO**, constitute a code totally unique to that sample (Florida, year 2000, site 1, sediment chemistry sample). The combined version facilitates the option of barcoded labels, as some regions have so indicated an interest; all the necessary information is on one label.

For those regions and states that do not intend to use barcodes, the two types of codes can be printed on separate labels. Therefore, for a specific site, the crew should be provided with an abundance of preprinted site ID labels (e.g., one-hundred FL00-0001 labels) that they can use to label field sheets, sample containers, or any thing related to that site; sample ID codes would be generic and usable at all sites.

Regardless of which labeling approach is utilized, it is suggested that sampling packets for each site be made up ahead of time by placing a complete set of field data forms and preprinted labels into a large envelope; mark or label the outside of the envelope with the site ID code, estuary or area, and sampling location in coordinates of latitude, longitude. These packets can then be filed numerically in a box file or cooler for transport to the field. A day or two prior to a scheduled

sampling, the crew can pull the specific site packet and label a complete set of sample containers (if the labels are not waterproof, they should be covered with clear cellophane tape), then consolidate the prelabeled sample containers, data sheets, and extra labels in an appropriate size plastic bag for easy storage and transport aboard the boat, come dat sampling day. Such measures save time in the field and help to ensure that sampling proceeds in an orderly manner.

#### Data Transfer

Field information recorded on hardcopy must be transferred to an electronic format for transmission to the IM Node. The hardcopy field data should be transcribed periodically to the electronic format, before the trail goes cold and, also, to avoid heaping an unwelcomed burden upon a hapless individual at the termination of the field phase. The task may be assumed by a land support team or it may be the added responsibility of a member of the boat crew. The electronic format will be a template similar to the hardcopy form; the same data will be entered to the electronic file that was recorded in the field.

Certain field data may be collected electronically (e.g., CTD casts). If possible, these files should be downloaded and reviewed while still on site to ascertain validity (screened for incomplete files or obvious outliers). If there are any apparent problems, attempts should be made to rectify the situation and retake the profile. Certain ancillary information related to electronically logged data still must be recorded on hardcopy forms to document data quality associated with the activity (e.g., calibration information, QC checks, etc.). These data must be indexed to the event by location, date, and time (e.g., information to document that discrete Winkler samples were collected for Site XX at YY meters).

All electronic files created during field activities must be periodically backed up on disks.

#### Sample Transfer

Each state will be responsible for establishing their own sample tracking scheme to document the transfer of samples from field collection to final analysis. Most agencies already have very structured systems related to this function and should have no problems in meeting the needs of C2000. While the C2000 will not require the stringency of Good Laboratory Practices (GLPs) - Chain-of-Custody protocols, the following level of accountability is expected.

When the field crew returns to the dock or staging area, they will turn both the field samples and respective data forms over to their land-based support team (or designated recipient) who will again verify that all samples are accounted by comparing actual sample containers against the field data forms. Upon inventorying, samples will then be temporarily stored under designated conditions to await shipment or delivery to the processing laboratories. In the event that a sample is missing, the person checking in samples will record the sample as missing on the inventory sheet. The boat crew responsible for the collection of that sample will be informed so that they may check the sample storage areas on the vessel. It may be that conditions in the field prevented the collect of a particular sample; in that situation, the reasons should have been recorded as a comment on the field data form. If the sample is not recovered, the crew chief will make the decision for corrective action, whether simply to re-sample while still in the area or to schedule a make-up sampling on a later date.

Samples will be held under temporary field storage for only a few days, at the most, before they are shipped or delivered to the appropriate processing laboratory or long-term storage facility. A complete invoice, listing each sample ID codes, date packed, and name of person who packed the samples will accompany every batch of field samples sent from the field to a receiving facility; the field unit will retain a copy of the invoice. On the receiving end, as each sample is unpacked it will be checked-off of the invoice as received and immediately stored under prescribed holding conditions. The person receiving samples will sign, date and file the invoice. The receiving facility should immediately report any missing samples to their respective State Coordinator or EPA Regional Coordinator, who will initiate appropriate corrective action.

Once a complete set of field collected samples are received by a processing laboratory, a master list will be compiled of all sets of samples and where they reside (e.g., freezer A, refrigerator B, or storage shed Z). The master list should be filed in the general area where the samples are held. When samples are released to (or checked out by) an analyst, the transfer will be documented on the master list by initial and date; the quantity of sample released should be recorded. If the sample or portions of it are returned to the central storage area, this should also be logged on the master list. When the laboratory uses an internal tracking codes, they must be indexed to the original C200 sample ID code (both site and sample identifiers) and all analytical results will be reported using the C2000 ID code.



## B4 ANALYTICAL METHODS REQUIREMENTS

Analytical procedures for Coastal 2000 range from straightforward determinations such as percent silt/clay to comprehensive analyses of chemical contaminants in complex environmental matrices. Most procedures for the various analyses are based on those developed for EMAP-E and specific details for the analytical processes are documented in existing documents. Where appropriate, this QAPP will reference those documents or include them as Appendices.

### **Analyses of Chemical Contaminants**

The analyses of chemical contaminants (organic and inorganic) in sediments and tissue represent the more difficult analytical challenges. The scope of analytes is broad and the concentrations occurring in environmental samples can be very low. No specific, U.S. EPA Methods are required for these analyses; it is left to each laboratory to develop its own best analytical procedures based on their available resources, personnel and bench state of the art. However, the QA/QC for these analyses is performance-based, and the laboratory must meet the minimum quality criteria set forth. EMAP-E's performance-based approach to QA/QC for analytical chemistry is involved and is described in Appendix A of this document. Although each laboratory develops their own method, for most of the analyses to be performed, certain approaches are generally accepted. For example, the analysis of organochloride pesticides and PCBs will probably be conducted by using gas chromatography with electron capture detection (GC-ECD), regardless of the laboratory, but the extraction or cleanup procedure may vary from lab to lab. Other general methods suggested for CM chemical analyses include PAH analysis by GC-MS; trace metals analysis by either atomic absorption spectrophotometry or ICP. **See Appendix A for a full discussion of the quality criteria that govern these analytical chemistry procedures.**

### **Water Quality Indicators**

Conditions of water quality will be evaluated for each C2000 station through the analyses of indicators of anthropogenic enrichment, including nutrient levels and chlorophyll a content. Samples for these indicators will be obtained by filtering site water (collected at the depth regimes described in Section B3) and retaining the material filtered out for the analyses of chlorophyll a; the filtrate will be used for the analyses of soluble nutrients. The basic laboratory methods for these analyses will be:

- chlorophyll a analysis - acetone extraction, spectrophotometric analysis
- soluble nutrients - spectrophotometry (autoanalyzer)

Each of the analyses will be conducted in accord with generally accepted laboratory procedures such as those described in Standard Methods for the Examination of Water and Wastewater or U.S. EPA Methods. Appropriate QC samples (e.g., standards, reagent blanks, duplicates, and standard reference materials) will be run with each batch of samples. If the prescribed quality criteria are not consistently met, the analyst will confer with the laboratory supervisor for corrective measures before proceeding with additional samples.

Total suspended solids (TSS) determinations will be conducted with samples of unfiltered water collected at the same time that water for the anthropogenic enrichment samples is collected

(see above). A sample of approximately 1 liter (or volume adjusted to yield <200 mg/l of TSS) will be filtered, then the filter dried at 103 - 105°C and weighed to determine the amount of TSS in the sample. Laboratories performing these determinations will generally follow the procedures described in Standard Methods for the Examination of Water and Wastewater (APHA, 1989).

### **Sediment Silt-Clay Content Determination**

Silt-clay will be determined for sediment collected from each station by the differentiation of whole sediment into two fractions: that which passes through a 63-um sieve (silt-clay), and that which is retained on the screen (sands/gravel). The results will be expressed as percent silt-clay. The procedures to be used should be based on those developed for EMAP-E and described in “EMAP-Estuarines Laboratory Methods Manual Volume 1- Biological and Physical Analyses” (U.S. EPA, 1995).

### **Total Organic Carbon (TOC)**

Analysis of sediment TOC will be conducted with sediment sampled from each C2000 station. The sediment will be dried and acidified to remove sources of inorganic carbon (e.g. carbonates); the analysis will be conducted using a TOC analyzer to combust the sample to form CO<sub>2</sub> which is measured by infrared detection (U.S. EPA, 1995).

### **Macrobenthic Community Assessments**

Macrobenthic organisms collected and preserved at each C 2000 station will be analyzed at the laboratory for species composition and abundance. The laboratory evaluations will be based on methods described in “Section 3-Benthic Macroinvertebrate Methods Macrobenthic Assessment” of EMAP Laboratory Methods Manual -Estuaries, Volume 1: Biological and Physical Analyses (U.S. EPA, 1995). The sample will first be sorted into major taxon groups which then will be further identified to species and counted. A senior taxonomist will oversee and periodically review the work performed by technicians.

### **Sediment Toxicity Testing**

At each C2000 station, surficial sediment will be collected for use in acute toxicity tests in which marine amphipods will be exposed to test treatments of sediment for up to 10 days under static conditions; the tests will be aerated. The toxicity tests will be conducted in accord to the standard method described in “Section 2: Sediment Toxicity Test Method” of the EMAP Laboratory Methods Manual Volume 1 (U.S. EPA, 1995) ; these protocols are based on American Society for Testing and Materials (ASTM) Standard Method E-1367-90 (ASTM, 1991). After 10 days exposure, the surviving amphipods will be counted and results expressed as test treatment survival compared to control survival. EMAP has historically used the marine amphipod , *Ampelisca abdita*, as the standard test species for this bioassay. *A. abdita* will continue as the “standard” test organisms for C2000 sediment toxicity testing, however, other species may also be tested to further investigate the efficacy of alternative species, especially for those regions where *A. abdita* is not an indigenous species. C2000 is receptive to broadening its list of approved test species



for these tests and will maintain a flexible policy regarding what species to permit as test organisms. Several other toxicity tests have previously been conducted in association with EMAP- sponsored projects including, MicroToxR solid- phase with sediments; sediment porewater - sea urchin fertilization/embryological development test (Carr et al. 1998); and alternative species of amphipods (as described above). Although marine amphipods will be the standard test organisms for the core toxicity tests, Regions may, at their discretion, specify one or more of these alternative approaches for sediment toxicity testing within their jurisdiction, or individual states may elect to pursue additional testing on their own.

Basic test protocols for MicroTox exposures were developed by the Microbics Corporation and were issued with their system at purchase (Microbics Corporation, 1992) (since that time, MicroTox rights, have been purchased by Azur Environmental, Carlsbad, CA). Many researchers, however, modify the basic methods to adapt the system to their particular situation. Since C2000 considers MicroTox testing as a research indicator under development, most modifications will be acceptable provided that the laboratory adequately documents departure from established methods.

## B5 QUALITY CONTROL REQUIREMENTS

Each analysis or measurement conducted for Coastal 2000 Monitoring will have prescribed quality control (QC) checks with quality criteria or acceptable tolerances established, where applicable. In general, the QC guidelines for C2000 have been adopted from those developed for the EMAP-E quality program. For that reason, this document will summarize the key QC elements for C2000 field and laboratory measurements. Table A7.1 and A7.2, in this document, present summaries of the measurement quality objectives and of the QA sample types for core C2000 indicators. Because the involved nature of the QA/QC program developed for analytical chemistry, an entire section has been dedicated to address those issues (see Appendix A). General discussion of the QC for individual field and laboratory activities follows.

### FIELD ACTIVITIES

QC elements associated with field monitoring activities relate to locating the sampling site, the collection and handling of environmental samples, and direct measurements taken onsite are presented in the following.

#### Locating station

Field crews will use differential Global Position Satellite (GPS) navigation systems to locate the C2000 sampling stations. Coordinates of latitude and longitude for the previously selected random sampling stations will be issued to the field crews along with their sampling packages; the coordinates will be expressed in units to the nearest 0.01 minute. The vessel operator should review navigation plans for a site at least a day prior to the scheduled sampling. Before leaving the dock, the station position will be entered into the GPS system and the operator will safely navigate to the area. As the vessel closes in on the general location, the operator will decrease speed and allow the GPS to guide the vessel onto the location and then weigh anchor. After anchoring, the sampling vessel should come to rest within 0.02 nautical miles (nm) of the “intended” location; the actual coordinates of the anchorage will be recorded on the Station Information Data Sheet.

While 0.02 nm is the target criteria for accuracy in siting the station, the crew will be granted a buffer zone of up to 0.05 nm (~300 ft) from the intended position in the event that there are mitigating circumstances to justify exercising that allowance (e.g., currents, obstacles, boat traffic, etc). This buffer zone will be used only for those situations when locating within the 0.02-nm goal is not feasible.

In cases where the vessel cannot navigate to within 0.05 nm of the intended site (e.g., the site is actually landlocked or the depth too shallow), the crew will record the station as “intended-unsamplable” and thoroughly document the reason(s) on the Station Information Data Sheet. The crew will then relocate to the nearest position that permits sampling and conduct the monitoring. It is not anticipated that situations like that will occur very often and less likely if suspect areas were reconnoitered prior to the monitoring window. This degree of latitude is to be used only when truly warranted, not as a matter of mere convenience or preference.

## Water column measurements

Because of the multiple field crews to be involved in C2000, an array of water quality instrumentation will be employed for water column profiling. Basically, two general type of units will be used for this activity: self-contained CTD units that log continuous profiles that are electronically captured as the unit is lowered and retrieved through the water column; and, multiparameter water quality monitoring probes (e.g., Hydrolab or Yellow Springs Instruments, YSI, sondes) which are connected by hardline to a deck display unit and measurements are manually recorded as the probe is lowered or retrieved through the water column at discrete intervals of depth.

Proper maintenance and routine calibration checks are the key elements related to quality control for these instruments. Calibration of the CTD units is an involved procedure that is usually performed only periodically (e.g., biannually) and at a center that is equipped for that function; however, the instruments have an established track record and tend to be reliable for the intervals between calibrations. In-field calibration checks will be conducted on a daily basis when the CTD unit is in use to document the instruments performance. The probe/deck display units, on-the-other-hand, are easy to calibrate; these units will undergo QC checks on a daily basis and be calibrated if out of tolerance. Calibration requirements and QC checks for the various instruments are described in the following sections. Because of EMAP's familiarity with Hydrolab units, procedures that have proven successful in its performance will be presented as a template for similar instruments.

### Hydrolab H20 Multiprobe

The Hydrolab H20 multiprobe water quality profiling instrument has proven to be a dependable instrument that, if properly maintained and correctly calibrated, can be relied on to perform with in the range of accuracy that C2000 requires for basic water quality parameters of temperature, salinity, pH, dissolved oxygen (DO), and depth. The H20 will be calibrated daily, preferably at dockside on the morning of its intended use; the calibration will be documented on the Hydrographic Profile Data Sheet. Calibration of the dissolved oxygen polarographic sensor is based on using a water-saturated air environment as the standard; for pH, a two point calibration curve is established with standard buffer solution of pH 7 and 10; the salinity/conductivity probe is calibrated using a secondary seawater standard that has been standardized against IAPSO Standard Seawater using a WESCOR vapor pressure osmometer; the depth sensor, a pressure activated transducer, is set to a zero pressure while out of the water. Temperature is a fixed function set by the manufacturer and cannot be adjusted in the field (to date, no problems have ben encountered with the temperature sensor); the instrument reading is verified against a hand- held laboratory thermometer.

For each of the water quality parameters, EMAP has established a maximum range of allowable difference that the instrument may deviate from calibration standard (Table B5.1). It should be noted that while these limits are acceptable for the purpose of qualifying field measurements taken with the unit, when performing the daily QC check, crews should set the instrument to as near the standard as possible. The daily QC checks should not require more than slight adjustments to bring the instrument into agreement. If an instrument's performance becomes erratic or requires significant adjustments to calibrate, the unit should be thoroughly trouble-shot; problems generally can be determined as being probe-specific or related to power source (e.g., low

battery voltage or faulty connections). Routine maintenance and cleaning should be performed as per the manufacturer's recommendation.

Table B5.1 Maximum acceptable differences for instrument field calibration and QC checks.

Instrument	Frequency of Check	Parameter	Checked Against	Maximum Acceptable Difference
Hydrolab	Daily	Temperature	Thermometer	$\pm 1^{\circ}\text{C}$
		Salinity	Standard seawater	$\pm 0.2$ ppt
		pH	pH buffer solution	$\pm 0.1$ pH units
		DO	100% saturation	$\pm 3.0\%$
		Depth	Sea level	$\pm 0.2$ m

Failed QC or calibration checks should initiate a thorough inspection of the unit for obvious sign of malfunction (e.g., loose connections, damaged probes, power source, fouling on DO membrane, etc.). After any maintenance required to correct problems, the unit will be re-calibrated with documentation on the appropriate field data form. In most cases, unless a probe is actually broken or damaged, the Hydrolab H20 can be corrected in the field. If the unit will calibrate within the guidelines, continue with the water column measurements. If one or more parameters remain suspect, fully document the nature of the problem on the field form and report the situation to the Regional QA Coordinator for resolution. Depending on the importance of the suspect parameter, the site may require a revisit to log an acceptable water column profile. Of course, it is always advisable to have a backup instrument available.

#### CTD Water Column Datalogger

SeaBird CTDs are generally accepted by oceanographers and marine scientists as the workhorse instrument for logging physical water quality parameter, especially for deepwater situations. If properly maintained and operated by investigators who routinely utilize CTDs, these instruments produce very reliable data. The following schedule of servicing is recommended.

On an annual basis, CTDs should be cycled through a comprehensive maintenance check and calibration verification performed by the manufacturer or a certified servicing center. Before a unit is scheduled for field deployment, it must first undergo a thorough calibration check conducted a laboratory or facility that is set up to conduct the procedures (e.g., water tanks large enough to accommodate submerging the unit, capability to alter conditions of dissolved oxygen to provide environments of high, mid, and low levels of DO, and laboratory capabilities to conduct Winkler titrations, pH determination, and salinity/conductivity determinations. Once the CTD unit passes these checks it can be expected to perform within the QC guidelines required by C2000, for nominal periods of 1-2 months. At a minimum, CTD units used in C2000 will be required to undergo laboratory conducted QC checks just prior to their field deployment for the summer sampling and immediately upon the conclusion of the sampling in September (approximately 6 weeks); more

frequent lab verifications are preferred (i.e., monthly).

In addition to the calibration schedule described above, the field crews who use CTDs will conduct daily QC checks to validate that the unit is functioning properly and in compliance with the CM data quality criteria. These daily checks can be conducted as a side-by-side comparison against a calibrated (documented) water quality probe (e.g., YSI or Hydrolab), or by collecting water samples simultaneously Van Doren or Niskin bottles from discrete depths as the CTD logs a cast. Water quality parameters for these samples will be measured independently to provide comparisons to the CTD values. Of those parameters, salinity, temperature, and depth can readily be validated onsite. The approximate salinity ( $\pm 1$  ppt) of the water sample can be measured by using a refractometer to immediately check the salinity and the temperature can likewise be checked with a conventional hand-held thermometer; depth at bottom can be checked against the vessel's depth recorder. The above QC checks will be performed as realtime validation checks, not calibration verifications. The CTD values will not be questioned unless the comparisons between the two measurements are in obvious disagreement; in which case, the crew will conduct follow up checks to ascertain which measures are valid. For DO validation, water samples will be preserved for Winkler titration to be conducted later, back onshore; pH can be checked from an unadulterated water sample held for pH determination back onshore.

The measurement quality objectives (MQOs) for accuracy of the CTD units, based on comparison of the unit's performance against reference standards or instruments, are:

Dissolved oxygen	$\pm 0.5$ mg/L
Salinity	$\pm 1.0$ ppt
pH	$\pm 0.3$ units
Temperature	$\pm 1.0^{\circ}$ C
Depth	$\pm 0.5$ m ( $\sim 2$ ft)

The measurements for transmissivity will be periodically verified at the time of scheduled major calibration and maintenance.

A failed QC check for the CTD should initiate an immediate check of the instrument for obvious signs of malfunction (e.g., loose connections or plugged lines). If the instrument cannot be brought into acceptable tolerances, the data files must be flagged as being out of compliance and a description of the problem will be noted on the field data form. The situation will be reported to the Regional QA Coordinator, who will make the decision on repeating the water column profile with a properly functioning instrument.

### LICOR L1100 light meter and Secchi disk

No daily field calibration procedures are required for the LICOR light meter; however, the manufacturer recommends that the instrument be returned to the factory for annual calibration check and resetting of the calibration coefficient. Calibration kits are available from LICOR and this procedure can be performed at the laboratory (see LICOR operation manual). There are several field QC measures to help ensure taking accurate measurements of light penetration. The “deck” sensor must be situated in full sunlight (i.e., out of any shadows), likewise, the submerged sensor must be deployed from the sunny side of the vessel and care should be taken to avoid positioning the sensor in the shadow of the vessel. For the comparative light readings of deck and submerged sensors, (ratio of ambient vs. submerged), the time interval between readings should be held to a minimal (approximately 1 sec).

No field calibration procedures are required for the Secchi disk. QC procedures, when using the Secchi disk to make water clarity measurements, include designating a specific crew member as the Secchi depth taker; take all measurements from the shady side of the boat (unlike LICOR measurements which are taken from the sunny side); and do not wear sunglasses when taking Secchi readings.



## Prelabeled Sample Containers

The following sections describe QC/QA procedures related to the collection of field samples. Proper labeling of samples is a very important QA aspect and cannot be overstressed. All sample containers for a site should be prelabeled prior to arriving on station. Prelabeling clean, dry containers helps to ensure that labels adhere properly to the containers. A little bit of sea spray or condensation wrecks havoc on labeling. Therefore, affix all labels to sample containers in the clean comfort of the lab or motel; not at the dock, not onsite. It is best to have a “sampling packet” for each station consisting of data sheets, lat/lon coordinates of station, prelabeled containers, and extra labels - all contained in a single plastic bag. The crew can then grab the packets for that day’s stations, along with extra unlabeled set, as they head out for the day.

## Water Quality Samples

Field procedures for the collection of water quality samples basically involve the collection and filtration of water samples. The field crews will assemble a water sampling kit consisting of a 4-l, plexiglass Van Dorn water sampler or Niskin bottle; a Nalgene reservoir container; glass fiber filters, GF/F- 25 mm, for use with stainless steel filter holding units that fit standard luerlock syringes, or 47-mm GF/F for use with hand vacuum pump systems; and several sets of stainless steel tweezers. These implements will be maintained in a clean environment (e.g., in a clean tackle box) and will be reused to process samples at each station. In addition, a separate sampling packet will be issued for each station that contains a new, sterile disposable 60 cc plastic syringe; a clean 60 cc Nalgene bottle; plastic petri dishes; and several squares of aluminum foil. All water quality related samples will be immediately frozen on dry ice upon collection; unless in cases where states have requested and been approved to use alternative methods for sample preservation. Additional QC guidelines for the collection of water samples include the following.

Site water collected will be taken from a depth regimes as described in Section B2 using a Van Dorn sampler or Niskin bottle. The sampler should be lowered to depth and maneuvered horizontally for several seconds before triggering to ensure that the water captured is from the designated depth. A small amount (~500 ml) of the collected water should be used to rinse the reservoir before adding the remainder of the water for sample processing. The filter holders must be rinsed well with deionized water prior to loading with their appropriate filter; care must be taken in general to set up in a relative clean work space for the filtering process.

Chlorophyll samples will be collected by filtering 100-200 cc of site water (or sufficient volume to produce a visible green residue on the filter) through the 25 mm GFF; duplicate samples are advised; **the volume of sample water filtered must be recorded on the field data form** (also, recording the volume on the petri dish containing the filters is encouraged because it provides the laboratory analyst with the information without having to look up from field data forms). Chlorophyll is light sensitive, therefore, the filters containing the phytoplankton samples will be shielded from light by placing them in a labeled, plastic petri dish and wrapping it in aluminum foil before storing on dry ice.

The dissolved nutrient sample will be collected by injecting approximately 40 ml of the filtrate into a clean 60 cc Nalgene bottle. The sample will be capped and placed on dry ice.

After completing the water sample processing for a station, the filter holders should be



thoroughly rinsed with deionized water prior to loading for the next station. All samples will be held on dry ice during transport back to the shore holding facility, where they will be temporarily held on dry ice or in a conventional freezer to await shipment to the processing laboratory. Temporary storage in a conventional freezer should be held to a minimum, no longer than 2-3 days; even then, bacterial action may slightly compromise the sample, especially for ammonia. It is recommended that all enrichment samples be held at the laboratory in an ultrafreezer at  $< -50^{\circ}\text{C}$ .

## Sediment Collection

Surficial sediment will be collected from each station for the analyses of chemical contaminants, toxicity testing, TOC, and percent silt-clay. The QC requirements for the collection of the samples relate to obtaining a successful grab and to avoiding outside contamination to the sediment while processing. For a grab sample to be successful, the jaws of the grab must be totally closed upon retrieval (i.e., no obstruction - oyster shells, sticks, etc); the grab should be  $>75\%$  filled; and sample should appear intact with little disruption to the surficial portion. If these conditions are not met, the grab should be discarded and another sample collected. Overlying water will be carefully siphoned off or the grab jaws may be slightly opened to allow the water to drain very slowly without any channeling effects.

When a good grab is obtained, only the top 2-3 cm of sediment will be taken. All implements used for the collection and processing of the sediment (e.g., stainless steel spoon and mixing pan) must be clean and rinsed thoroughly with site water prior to using. Surficial sediment will be collected from three successful grabs and composited in the stainless steel mixing bowl; the bowl will remain covered between grabs to protect from possible atmospheric contamination. The composited sample will be mixed well using a stainless steel spoon to ensure homogeneity. Care must be exercised not to introduce human- or vessel-sourced contaminants such as blood, sweat, and tears, sunscreen, or fuel. After mixing, appropriate volumes of the homogenated sediment will be distributed into sample containers, filling each to approximately 75% (this allows for expansion during later freezing). In the field, all sediment samples will be held on wet ice. At the lab, all, but the silt-clay and toxicity samples, will be frozen in conventional freezers ( $-20^{\circ}\text{C}$ ) to await analyses; the silt-clay and toxicity samples will be refrigerated ( $4^{\circ}\text{C}$ ).

## Fish Collection

At all C2000 stations, attempts will be made to collect fish by trawl to provide data on diversity and abundance and to provide samples for histopathological examination and for the analyses of chemical contaminants. The QC guidelines for fish collections relate to the conduct of the trawl, the correct identification of the catch, and to the processing and preservation of the various sample types. A successful trawl requires that the net deploys with the doors upright and spread and that the net fishes on bottom for a  $10 \pm 2$  min duration without interruption. The trawl data will be recorded on the trawl Information Data Sheet. All fish/shellfish will be identified to species and a total count recorded; if extremely large numbers are caught, the count may be estimated. List of the target species for analyses of chemical contaminants (“chemistry fish”) will be generated on a regional-specific basis, but will generally include demersal species (e.g., most flatfishes). All fish identifications, counts, and final disposition will be recorded on Fish Data Sheets.

### Chemistry Fish

Fish sampled for chemistry will be individually wrapped in heavy aluminum foil and collectively placed in a clean plastic Ziploc bag with a station and sample label and held on wet ice during transport back to the lab. Care must be taken while processing the fish to avoid contamination from outside sources such as fuel. A sample size of five individuals per species is desirable and they should be composited into one sample bag. If the fish are small and the catch is abundant, 10 or more individual fish should be sampled to ensure adequate tissue ( $\geq 200$  g) for the analyses. If the catch is large, multiple ziplock bags may be used to hold a composited sample; be sure to label all bags with the site ID and sample ID codes and note the number of bags on the field data form.

### Histopathology Fish

As the crew processes the fish catch they will briefly examine each fish for gross external pathologies. Any fish observed with a gross external pathological condition (e.g., tumors or lesions), will be processed in the field by using a clean scalpel or scissors open up the body cavity from the anus to the thorax region, followed by manually popping open the incision, and then immediately submersing in a container of Dietrich’s fixative. The specimen will be properly labeled either on the container or by tagging the fish with an impervious, solvent-proof label. The labeling must at least index the specimen to the site; additional information will be included on the Fish Data Sheet (e.g., species, length, etc).

West Coast field crews may use an alternative field method to process fish with observed gross external pathologies. A small portion the affected tissue will be excised from the specimen using clean scissors or a scalpel, being sure to also include a small section of the adjacent healthy appearing base tissue. The excised tissue will then be placed in a histological cassette and labeled with a “patho number” (both furnished by the NOAA/NMFS Seattle laboratory). The cassette can then be immersed in a container of Dietrich’s fixative. The QC requirements are that the fish be expediently processed and to ensure sample integrity. If multiple cassettes are held in a single container of Dietrich’s, they must not be crowded; a container should not be more than 50% filled with samples (i.e., the volume of Dietrich’s should be twice the volume of sample). Also, it is mandatory to cross index the samples in a manner that each fish is identifiable to species and

station; the field data sheets will have a field to account for this. Once fixed in Dietrich's, the samples are stable indefinitely, however, all samples should be submitted to the histopathological laboratory within 2-3 weeks of collection.

### **Macrobenthic organisms**

Macrobenthic organisms will be collected at each C2000 station for later laboratory assessments of benthic community structure. At these stations, one sediment grab will be taken by a Van Veen grab sampler and sieved through a stacked (nested) set of sieves; a 1.0- mm sieve prior to a 0.5- mm sieve. All materials retained on each of the sieve will be placed in separate plastic containers and fixed with buffered formalin (final concentration of 10% formalin in the jar). Experience has proven that it very important to use quality containers that seal tightly. Before investing in a large supply of "untested" containers, it is advisable obtain a sample product and put it through your own trial by ordeal - invert a filled container and allow it to sit overnight, drop one on the floor, etc.

The QC for this activity includes guidelines for the sieving process and for the preservation of the samples. Passive sieving (i.e., sieving without the use of directed water - no jets) is encouraged as much as possible to avoid damage to the soft bodied organisms. However, some difficult samples may require limited use of a gentle water flow to carry out the sieving. During the sieving operation, no overflow or spills will be permitted; if either of these occurs, the sample will be aborted and a new grab taken for processing. The sieving action should be continued until the processing water remains clear, indicating that the muddy fraction of the sample has been purged. The materials remaining on the sieve are to be gently rinsed into a sample jar. The container should not be filled more than 50% with sample, use additional jars to contain the total sample, labeling them in sequential series. Each jar should be filled to 80% then topped off with 50% formalin to yield a final concentration of 10% formalin. Aboard the boat, the fixed samples should be stored out of direct sunlight; at the laboratory, the samples should be maintained in a dry, cool environment to await further processing.

## LABORATORY ANALYSES

The laboratory analyses of C2000 samples include analyses of sediment, fish, and water samples, sediment toxicity tests, evaluations of macrobenthic community structure, and the histopathological examination of fish. These laboratory activities are based upon procedures or analytical methods established for EMAP-Estuaries and the QC associated with each is well documented in existing methods manuals and QAPPs ( U.S. EPA, 1995 and Heitmuller and Peacher, 1995). This QAPP will summarize the QC requirements for the various analytical operations, but for detailed discuss of the QC procedures for a specific activity, the user is referred to the above documents.

### **Analyses of Chemical Contaminants in Environmental Samples**

The analyses of chemical contaminants represent the more challenging and involved analytical efforts within the scope of Coastal 2000 and include the analyses of both organic and inorganic analytes for two matrices, sediment and tissue; see Table B5-2 for the list of analytes to be measured. To be relevant for C2000 assessments, the levels of detection required for many of the analytes are very low and may prove taxing to some analytical laboratories. Appendix A of this document is a copy of the analytical chemistry section used in preexistent EMAP-Estuaries QAPPs and it presents the established QA/QC requirements for these analyses in great detail. Three primary areas are addressed: initial demonstration of the laboratory's technical capability; the actual analysis and its associated performance-based QA with quality criteria described for accuracy and precision; and data documentation and reporting.

**TABLE B5-2.** Chemicals to be measured in sediments and tissue by EMAP-Coastal 2000 Monitoring.**Polynuclear Aromatic Hydrocarbons (PAHs)    21 PCB Congeners**

	<u>PCB No.</u>	<u>Compound Name</u>
Acenaphthene		
Anthracene	8	2,4'-dichlorobiphenyl
Benz(a)anthracene	18	2,2',5-trichlorobiphenyl
Benzo(a)pyrene	28	2,4,4'-trichlorobiphenyl
Biphenyl	44	2,2',3,5'-tetrachlorobiphenyl
Chrysene	52	2,2',5,5'-tetrachlorobiphenyl
Dibenz(a,h)anthracene	66	2,3',4,4'-tetrachlorobiphenyl
Dibenzothiophene	101	2,2',4,5,5'-pentachlorobiphenyl
2,6-dimethylnaphthalene	105	2,3,3',4,4'-pentachlorobiphenyl
Fluoranthene	110/77	2,3,3',4',6-pentachlorobiphenyl
Fluorene		3,3',4,4'-tetrachlorobiphenyl
2-methylnaphthalene	118	2,3',4,4',5-pentachlorobiphenyl
1-methylnaphthalene	126	3,3',4,4',5-pentachlorobiphenyl
1-methylphenanthrene	128	2,2',3,3',4,4'-hexachlorobiphenyl
2,6-dimethylnaphthalene	138	2,2',3,4,4',5'-hexachlorobiphenyl
Naphthalene	153	2,2',4,4',5,5'-hexachlorobiphenyl
Pyrene	170	2,2',3,3',4,4',5-heptachlorobiphenyl
Benzo(b)fluoranthene	180	2,2',3,4,4',5,5'-heptachlorobiphenyl
Acenaphthylene	187	2,2',3,4',5,5',6-heptachlorobiphenyl
Benzo(k)fluoranthene	195	2,2',3,3',4,4',5,6-octachlorobiphenyl
Benzo(g,h,i)perylene	206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl
Indeno(1,2,3-c,d)pyrene	209	2,2'3,3',4,4',5,5',6,6 '-decachlorobiphenyl
2,3,5-trimethylnaphthalene		

**DDT and its metabolites****Chlorinated pesticides other than DDT****Trace Elements**

2,4'-DDD	Aldrin	Aluminum
4,4'-DDD	Alpha-Chlordane	Antimony (sediment, only)
2,4'-DDE	Dieldrin	Arsenic
4,4'-DDE	Endosulfan I	Cadmium
2,4'-DDT	Endosulfan II	Chromium
4,4'-DDT	Endosulfan sulfate	Copper
	Endrin	Iron
	Heptachlor	Lead
	Heptachlor epoxide	Manganese (sediment, only)
	Hexachlorobenzene	Mercury
	Lindane (gamma-BHC)	Nickel
	Mirex	Selenium
	Toxaphene	Silver
	Trans-Nonachlor	Tin
		Zinc
		<b>Other Measurements</b>
		Total organic carbon (sediments)

Before a laboratory is authorized to analyze actual field collected samples, the lab must provide documentation to demonstrate its technical capability to perform at the level required by EMAP. The required documentation varies according to an individual laboratory's history and established track record. Laboratories that have successfully participated in the NIST/NRCC/NOAA/EPA Intercomparison Exercises may submit their recent results to C2000 Regional QA Coordinators for evaluation, while a laboratory new to the EMAP program may be required to complete the more structured, step-by-step demonstration of technical capability prescribed in the following.

The first step of this process is for the laboratory to calculate and submit method detection limits (MDLs) for each analyte of interest for the each matrix which they plan to analyze. Each laboratory is required to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate MDLs for each analytical method employed. To indicate the level of detection required, target MDLs have been established by EMAP (Table 5-5, Appendix A) and the MDLs reported by candidate laboratories should be equal to or less than the target values. It is important that a laboratory establishes, upfront, its capability to generally meet the MDL requirements; this is a key factor that must be established before proceeding further with the performance evaluation (PE).

Once the MDL requirements are met for an analyte class and matrix type, the laboratory will be issued a PE sample to analyze. The PE sample will be provided by the C2000 QA Coordinator and it will be representative of a naturally occurring environmental sample, matching, as closely as possible, the matrix and analyte concentration levels that the lab plans to analyze for C2000. When available, standard reference materials (SRMs) or Certified Reference Material (CRMs) should be used in these exercises. The basic quality criteria for these PE exercise are that the laboratory results generally meet accuracy goals set by CM. For the organic analysis, the general goal for accuracy is laboratory agreement within  $\pm 35\%$  of the certified or "true value" for the analytes of interest; for inorganic analysis, laboratory agreement within  $\pm 20\%$  of the accepted true value. These requirements apply only to those analytes with certified values  $\geq 10$  times the laboratory's calculated MDL (see Appendix A for further discussion). The participating laboratory will submit the results of their completed PE exercises to the C2000 QA Coordinator to be evaluated.

Only after a laboratory that successfully completes the PE exercises, will it be authorized to commence with the analyses of actual C2000 samples. In the performance-based QA approach for analytical chemistry, no set method is required of the laboratory as long as the laboratory continues to meet the quality standards of the program. Samples should be processed and analyzed as designated batches consisting of 20 or less samples and each batch will include prescribed QC samples (e.g., reagent blanks, matrix spikes and matrix spike duplicates, and SRMs). These QC samples represent the basic elements that provide estimates of accuracy and precision for the analyses of chemical contaminants. The overall analytical process involves several additional QC-related components or checks (e.g., calibration curves, use of internal standards, and control charts). When these QC checks are embedded in each batch, the analyst should be able to quickly assess the overall data quality on a per batch basis and take corrective measures if there are deficiencies. If data for a class of compounds consistently fails any of the C2000 quality standards, the laboratory management must notify the State QA Coordinator of the problem and seek recommended corrective actions prior to submitting the final data report. Table 5-4 in Appendix A presents a comprehensive listing of the key quality control elements for chemical analyses.



The QA/QC requirements described in this section are those developed for EMAP- Estuaries and are presented here to provide general guidance for the analytical chemistry conducted within C2000. It is possible that these requirements may prove too stringent for the candidate laboratories' current state of the art. In that event, on a case by case basis, these situations will be carefully reviewed by C2000 management and if a laboratory's best available technical capabilities are felt to be legitimately exceeded (e.g., due to limited instrumentation), the requirements may be amended to better reflect the laboratory's true potential. This should not be misinterpreted as a loophole; it will only be enacted for substantiated claims.

## Water Quality Analyses

Both filtered site water and particulate materials retained on the filters will provide samples to evaluate conditions of water quality at each station; these analyses will include soluble nutrients, chlorophyll content, and total suspended solids. Nutrient and chlorophyll samples will be immediately frozen on dry ice in the field; while awaiting analyses at the laboratory, the samples may be stored in a conventional freezer (-20°C), however, an ultrafreezer (-50°C) is recommended. In addition, a raw (unfiltered) sample of seawater will be collected for the analysis of total suspended solids. The following sections describe the methods and the QC samples to be incorporated with each analysis.

### Nutrient Analyses

Dissolved nutrients (i.e., nitrates, nitrites, phosphates, and ammonia) will be measured by using an Autoanalyzer; the methodology is based on spectrophotometric determinations described in A Practical Handbook for Seawater Analysis (Strickland and Parsons, 1969) and A Manual of Chemical and Biological Methods for Seawater Analysis (Parsons et al, 1984). Coastal 2000 has established a target Method Detection Limits (MDLs) for dissolved nutrients at 0.005 mg/L (5 ppb) for nitrites, nitrites+nitrates, and ammonia, and 0.002 mg/L (2 ppb) for ortho-phosphates (Table A7-2). Analytical sets or batches should be held to 20 or less samples and must include appropriate QC samples uniquely indexed to the sample batch. The minimum QC samples required for nutrient analysis on a per batch basis include a four point standard curve for each nutrient of interest; reagent blanks at the start and completion of a run; one duplicated sample; and one reference treatment for each nutrient. The performance criteria for an acceptable batch are: accuracy - the reported measurements for the reference samples be within 90-110% of the true value for each component nutrient and, precision - a relative percent difference between duplicate analyses of  $\leq 30\%$  for each component nutrient. Any batch not meeting the QA/QC requirements will be re-analyzed.

If certified reference solutions are not readily available, the laboratory may prepare its own laboratory control treatments (LCT) by spiking filtered seawater with the nutrients of interest. The concentration of the each component should be sufficient enough to result in a good instrument response while at the same time, remain environmentally realistic. For the LCT to be acceptable, the laboratory must demonstrate nominal recovery efficiencies of  $\geq 95\%$  for each component.



### Chlorophyll *a* Analysis

Chlorophyll *a* content of phytoplankton filtered from a known volume of site-collected water will be analyzed fluorometrically in the laboratory. The recommended method (described by Turner Designs) is a **non-acidification** variation of EPA Method 445.0: "In Vitro Determination of Chlorophyll *a* and Pheophytin *a* in Marine and Freshwater Phytoplankton by Fluorescence" (Arar and Collins, 1992). See Appendix D for details and appropriate references.

Basically, the filtered samples will be extracted with 90% acetone or methanol and the resultant extracted pigment will then be measured on a fluorometer configured with the lamps and filters specified by Turner Designs to optically exclude pheophytins. The target MDL for chlorophyll *a* is 0.2 ug/L, based on a filtered 1-L water sample; note that samples filtered in the field may be limited to volumes of 100- 200 ml, thus increasing the MDL. Based on difficulties experienced previously with obtaining a cleared sample after centrifuging, it is not required that the GF/F filter containing the sample be ground up as part of the extraction procedure; the filter may be extracted whole using a sonication bath to enhance the process.

The QA/QC requirements for chlorophyll analysis require that the laboratory first successfully complete an initial demonstration of capability prior to conducting analyses of the C2000 field samples. This exercise includes the determination of a linear dynamic range (LDR) using a series of chlorophyll stock standard solutions prepared from commercially available standards as described in Standard Method 445.0. Also, the laboratory should determine and report both instrument detection limits (IDLs) and method detection limits (MDLs). Upon the establishment of a LDR, the performance of the instrument should be verified by the analysis of a standard reference material (SRM) (e.g., Sigma - *Anacystis*).

During the routine analyses of C2000 chlorophyll samples, the following QC samples should be included on a per batch basis: a reagent blank and standard reference samples (analyzed in duplicate); a batch should consist of  $\leq 20$  field samples. The performance criteria for an acceptable batch are: accuracy - measured concentration for the reference sample be within 90-110% of the true value; and for precision - the relative percent difference (RPD) between duplicate analyses be  $\leq 30\%$ .

Although not required by the program, it is a wise practice to collect duplicate filtered chlorophyll samples while in the field. The second filter provides insurance in the event that the first is lost or that an extracted sample is mishandled or spilled.

### Total Suspended Solids (TSS)

The determination of TSS in unfiltered water samples is a straightforward process as described in the EMAP - Estuaries Laboratory Methods Manual Volume 1 - Biological and Physical Analyses, Section 6 - Residue, Non-Filterable (Suspended Solids) (US EPA, 1995): a known volume of water is filtered through a tared filter; the filter retained and dried; then weighed to determine the mass of TSS (APHA, 1984). An approximate 500-ml sample of water will be filtered through a tared 47-mm glass fiber filter; the practical range of determination is 4 to 20,000 mg/L. A sample size that results in no more than 200 mg is desired to avoid possible interference from clogging the filter; the exact volume will be recorded. The filters will dried in a clean aluminum weighing boat for at least 1 hr at 103-105°C, then cooled in a desiccator to balance temperature before weighing.

Initially, a laboratory should conduct a series of drying and weighing, followed by and re-drying and re-weighing to determine the degree of drying required to obtain a stable weight; the variance should not exceed 4% or 0.5 mg between weighings. Duplicate samples should be analyzed for each batch of  $\leq 20$  samples; the relative percent difference should be  $<30\%$  for samples with TSS results greater than the 8.0 mg/L (2 x minimum range of determination).

## **Sediment Characterization**

The physical properties of sediment including silt-clay and total organic carbon (TOC) content will be determined for sediment samples collected from each C2000 station. Laboratory procedures for both analyses are based on those described in the EMAP-Estuaries Laboratory Methods Manual Volume 1 - Biological and Physical Analyses (U.S. EPA, 1995). Percent silt-clay will be determined by using a 63  $\mu\text{m}$  sieve for the separation of whole sediment into a large particle fraction (sands/gravel) and fine particle fraction (silt-clays). TOC will be determined by combusting pre-acidified sediment samples in a TOC analyzer and measuring the volume of  $\text{CO}_2$  gas produced. Methods for these analyses are relatively straight forward, however, both include tedious procedures (e.g., precise sample weighing and pipetting) which require strict attention to laboratory technique. The following sections present the QC guidelines specific for each analysis.

### Silt-Clay

Sediment samples for percent silt-clay determinations will be held at the laboratory under refrigeration at approximately  $4^\circ\text{C}$ ; they should not be frozen. Sieves used for the silt-clay will have stainless steel screens and they should be used exclusively for the silt-clay analysis; the sieves should be cleaned with copious amounts of water and brushes should not be used because they may distort the openings. An analytical balance accurate to 0.1 mg will be used for all weighings. Prior to each period of use, the balance will be zeroed and calibrated. Its calibration will be verified using a standard weight; written documentation will be maintained. The two sediment fractions are oven dried for 24 hrs, then weighed. To ensure that the drying process had gone to completion, the weighed samples are returned to the drying oven for an additional 24 hrs and randomly selected subsample is re-weighed as a check for stability of the dry weights. All sample weighings will be recorded on preprinted data sheets.

The primary QC checks associated with the determination of percent silt-clay are related to the degree of reproducibility between duplicate samples (re-analysis). Silt-clay determinations should be conducted in batches consisting of 10-20 samples. Within a given batch, the samples should be of similar textural composition (i.e., either silty or sandy). Approximately 10% (but at least 2 samples) of each batch completed by the same technician will be randomly selected for re-analysis by the technician. If the absolute difference between the original silt-clay percentage and the second value is  $>10\%$ , then a third analysis will be completed and the value closest to the third value will be recorded in the data set. If more than 10% of the data from a batch are in error, the entire batch will be re-analyzed. A third check of 10 % of the re-analyzed samples should be conducted by a different technician to assure that the re-analyzed values are correct. The re-analysis and QC checks should be conducted within 30 days of the original analysis.

## Sediment TOC

Sediment samples for TOC analysis will be held at the laboratory in a freezer at approximately -20°C to await analysis. This is a modification to the procedural guidelines listed in the EMAP-Estuaries Laboratory Methods Manual which recommends holding samples under refrigeration at 4-5°C. TOC samples will be processed and analyzed as batches consisting of 20- 25 samples. QC samples to be included with each batch run are: method blank, at least one duplicated sample, and a certified reference material (CRM). Any one of several marine sediments CRMs distributed by the National Research Council of Canada's Marine Analytical Chemistry Standards Program (e.g., the CRMs: BCSS-1, MESS-2, and PACS-1) have certified concentrations of total carbon and are recommended for this use. The following quality criteria must be met for each batch of TOC samples. The method blank results should contain less than 10 ppm of carbon; the percent recovery for the CRM should be 95-105% of the certified value; and the RPD between duplicate samples should be <10%. If a batch fails to meet these QC requirements, the entire batch will be re-analyzed along with all required QC samples.

## **Macrobenthic Community Assessments**

Sediment grabs will be taken from each C2000 station and sieved on site through nested 1.0 and 0.5 mm screens to collect macrobenthic infaunal organisms for community structure assessments. The samples from each sieve will be preserved separately in 10% formalin with Rose Bengal vital stain (optional) to await later laboratory sorting, identifications, and counts.

Laboratory procedures and prescribed QA/QC requirements for benthic sample processing will be based on those described in Section 3 - "Benthic Macroinvertebrate Methods Macrobenthic Community Assessment" of the EMAP-Estuaries Laboratory Methods Manual - Volume 1: Biological and Physical Analyses (U.S. EPA, 1995). The samples should be stored in a dry, cool area and away from direct sunlight. The field preserved samples should be transferred to 70% ethanol within 2 weeks of collection.

A fairly regimented process of QC checks has been developed and widely adopted by most benthic ecology laboratories. Through a series random checks of sorted samples (major taxon groups separated from debris), at least 10% of each technician's work is verified by a senior taxonomist. The re-sorts will be conducted on a regular basis on batches of 10 samples. The quality criteria for the PBS benthic sorting are that the QCed sorts from a technician's work be evaluated at  $\geq 90\%$  efficiency; that is the minimum level of acceptability, in most instances without undue complicators (e.g., excessive detritus), the sorting efficiency should run  $\leq 95\%$ . Sorting efficiency (%) will be calculated using the following formula:

$$\frac{\text{\# organisms originally sorted}}{\text{\# organisms originally sorted} + \text{additional \# found in re-sort}} \times 100$$

If the QCed work is substandard, all that technician's samples subsequent to the last passed check must be re-sorted and the technician will be offered further instruction to correct the deficiency. Only after the technician demonstrates to a senior taxonomist that the problem has been rectified, will he/she be allowed to process additional samples. Experience has shown that in most situations of this nature, appropriate corrective measures are readily implemented and that the work continues with little delay. Standard data forms will be used to record the results for the original sorts and the

QCed re-sorts.

Species identification and enumerations will be performed by or under the close supervision of a senior taxonomist and only taxonomic technicians with demonstrated ability will be allowed to assist in these tasks. As with the sorting process, at least 10% of each taxonomic technician's work will be checked by a senior taxonomist or a designated competent taxonomic technician to verify accuracy of species identification and enumerations. The QC check will consist of confirming identifications and recounting individuals of each taxon group composing the sample. The total number of errors (either mis-IDs or miscounts) will be recorded and the overall percent accuracy will be computed using the following formula:

$$\frac{\text{Total \# organisms in QC recount} - \text{total \# or errors}}{\text{Total \# of organisms in QC recount}} \times 100$$

The minimum acceptable taxonomic efficiency will be 90%. If the efficiency is greater than 95%, no corrective action is required. However, if taxonomic efficiency is 90 - 95 %, the taxonomist will be consulted and problem areas will be identified. Taxonomic efficiencies below 90% will require re-identifying and enumerating all samples that comprised that batch. The taxonomist must demonstrate an understanding of the problematic areas before continuing with additional samples, and then, his/her performance will be closely monitored for sustained improvement.

In addition to the QC checks of taxonomist work, the QA program for benthic taxonomy requires that the laboratory maintains a voucher collection representative specimens of all species identified in the WPCM benthic samples. If possible, the collection should have the identifications verified by an outside source. The verified specimens should then become a part of the laboratory's permanent reference collection which can be used in training new taxonomists.

NOTE:

Interlaboratory Calibration Exercise. Benthic community structure is a very critical element to the overall assessment of the ecological condition of an estuarine system. The procedures to sort and correctly identify benthos are extremely tedious and require a high degree of expertise. Because of benthos' importance to the study and the level of difficulty involved in processing, to evaluate comparability among the three states, CM will conduct interlaboratory calibration exercises in which replicate (or similar) benthic samples will analyzed by the multiple laboratories involved. The specifics of the exercise are currently being formulated among the state agencies and the EPA Regional Coordinators and, upon, finalization, the procedures will appended to the QAPP.

### **Sediment Toxicity**

Sediment toxicity tests (sedtox) with marine amphipods will be conducted in accord to the guidelines in "Section 2- Sediment Toxicity", EPA EMAP-Estuaries Laboratory Methods Manual Volume 1 - Biological and Physical Analyses (EPA, 1995); this method describes test requirements and conditions in detail. The QC procedures pertain to two phases: pretest phase - initial demonstration of technical ability; and, testing phase - daily monitoring of test conditions.



### Initial Demonstration of Capability

Before being authorized to conduct sedtox tests with C2000 sediments, a laboratory must provide documentation of their technical capabilities by demonstrating that they have both the facilities and personnel to meet the challenges to successfully conduct static toxicity tests for the durations specified (i.e., 10-day exposures for amphipods).

If a laboratory has an established history of toxicity testing, then a review of their records may be all that is required to ascertain their technical competence; examples of such records would include current control charts for exposure of routine test species to reference toxicants, survival rate for control organisms during recent test runs, and test organisms culturing/holding logbooks.

On the other hand, if the laboratory is relatively unknown or newly organized, then it is highly suggested that they first conduct a series of performance evaluation (PE) exercises prior to being authorized to conduct toxicity test with C2000 sediments; also, a site visit to the testing facility is recommended to verify the laboratory's physical conditions. PE exercises should include having the laboratory capture/culture or commercially obtain batches of approved test species and hold them under the conditions described by test methods, without exposure to toxic agents, to ensure that the laboratory technicians have the expertise required and that the laboratory's systems are adequate to support the organisms in an apparent healthy state for the designated period of testing (e.g., 10 days for marine amphipods). The laboratory should also conduct a series of replicated exposures to reference toxicants to determine if the organisms respond to the range of concentrations where effects are expected and to evaluate the laboratory's degree of precision or reproducibility. Acceptability criteria for these PEs are for the laboratory to demonstrate that they can successfully hold test organisms for up to 10 days with survival rates of  $\geq 90\%$ . For reference toxicant tests, the laboratory should produce calculated LC50s (concentration estimated to be lethal to 50 percent of the organisms exposed to a test treatment) within the range routinely reported by other testing laboratories with established programs, and, the degree of precision between 4 or more replicated tests should be within a range of 2 standard deviations (2 sigma).

Evaluation of a laboratory's initial capability should be made by the Regional QA Coordinator. A laboratory should not start testing with CM sediments until notified in writing from the QA Coordinator that they are qualified to initiate testing.

### QC Checks During Test Phase

Tests will be conducted in accord to the procedures described in EPA, 1995. QC requirements during the test period include: daily checks of testing conditions (e.g., dissolved oxygen concentration, temperature, and lighting) and observations on condition of test organisms. These data will be checked on a daily basis and recorded on standard data sheets as prescribed by the test method. Testing temperature should remain within  $20 \pm 2^\circ \text{C}$ ; it can be measured from a beaker of water held in proximity to the test chambers (e.g., in the water bath or temperature-controlled chamber) a recording temperature gauge should be utilized. DO concentration should remain  $\geq 60\%$  saturation; if the aeration system malfunctions, the DO must be measured in all containers in which there was no aeration (no visible bubbles from tube). Lighting will be constant (no night/day regime) for the duration of the exposure period. For the test to be valid, survival in the control treatments must remain  $\geq 90\%$  on average for the replicated control chambers, and no less than 85% in any one container.

## **Data Reporting Units**

Both field measurements and results of laboratory analyses should be reported to the Intermediate Node in standardized formats. Table B5-3 list the preferred data reporting formats for the core indicators. It is anticipated that measurements recorded by the various dataloggers will not all be displayed to the same number of places and that there will be differences due to the use of significant figures, however, effort should be made to maintain uniformity.

**TABLE B5-3.** Data reporting format for EMAP-Coastal 2000 Monitoring.

MEASUREMENT	UNITS	EXPRESSED TO NEAREST
<b>Field Measurements</b>		
DO	mg/l; ppm	0.1
Salinity	ppt	0.1
pH	units	0.1
Temperature	°C	0.1
PAR	mE/m <sup>2</sup> /s	integer
Light Penetration	%	integer
Depth	meters	0.5
Secchi Depth	meters	0.5
Fish Lengths (fork or total)	cm	integer
<b>Laboratory Analyses</b>		
Sediment Chem:		
Pesticides and PCBs	ng/g; ppb (dry wt)	0.01
PAHs	ug/g; ppm (dry wt)	0.01
Metals	ug/g; ppm (dry wt)	0.01
Hg	ug/g; ppm(dry wt)	0.001
Tissue Chem:		
Pesticides and PCBs	ng/g; ppb (wet wt)	0.01
PAHs	ug/g; ppm (wet wt)	0.01
Tissue Chem:		
Metals ug/g; ppm (wet wt)	0.01	
Hg	ug/g; ppm (wet wt)	0.001
Water Quality Parameters:		
NO <sub>2</sub> /NO <sub>3</sub> - N	ug/l; ppb	0.01
NO <sub>2</sub> - N	ug/l; ppb	0.01
NO <sub>3</sub> -N	ug/l; ppb	0.01
Ammonia -N	ug/l; ppb	0.01
PO <sub>4</sub> - P	ug/l; ppb	0.01
Chlorophyll a	ug/l; ppb	0.01
Total Suspended Solids	mg/l	0.01
Composited Sediment:		
TOC	%C	0.01
% Silt/Clay	%	0.01
SedTox	%	survival integer



## Other Laboratory Evaluations

Some of the cooperative partners on the C2000 have elected to collect and analyze additional/ supplemental indicators, among them, phytoplankton samples, specific tissues or organs for histopathological evaluations for fish, and variations of size grabs for benthic community evaluations. These types of analyses or investigations, not required program-wide, will be conducted under the purview of the individual state's existing QA/QC program.

## B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Several pieces of equipment that may be utilized to collect or analyze environmental data for Coastal 2000 should have periodic maintenance and calibration verification performed by manufacturer's representatives or service consultants. These procedures should be documented by date and the signature of person performing the inspection.

CTDs - annual maintenance and calibration check by manufacturer or certified service center;  
Light Meters - biannual verification of calibration coefficient by manufacturer;  
Analytical Balances - annual verification by service representative;  
Analytical Instrumentation (ICPs, GCs, AAs, TOC Analyzer, AutoAnalyzer, etc.) - as per need based on general performance; service contracts recommended.

All other sampling gear and laboratory instrumentation will be maintained in good repair as per manufacturer's recommendations or common sense to ensure proper function.

## **B7 INSTRUMENT CALIBRATION AND FREQUENCY**

Both field and laboratory equipment and instruments require routine calibration checks to verify that their performance is within acceptable quality standards. The following sections will discuss the procedures and frequency for the various instrument calibrations that are key in the collection of accurate environmental data for the Coastal Monitoring.

### **FIELD CALIBRATIONS**

To ensure that field measurements meet the accuracy goals established for C2000, quality controls checks are performed on a regular basis for most of the field equipment/instruments used to generate monitoring data. When QC checks indicate instrument performance outside of C2000 acceptance criteria, the instrument will be calibrated (for those instruments that allow adjustments) against an appropriate standard to re-establish acceptable level of performance; the procedure will be documented on field data forms.

Some instruments have fixed functions that cannot be adjusted under field condition. In cases where these types of measurements fail the field-QC checks, the degree of variance will be documented in field records; if possible, the situation will be rectified by changing out the faulty equipment with a backup unit until the failed unit can be repaired. If no backup is available, depending on the relative importance of that particular measurement to overall success of the monitoring operation, the crew chief must decide whether to continue operations with slightly compromised or deficient data or to suspend sampling until the situation is corrected. For example, if the GPS system was found to be totally unreliable, sampling activities should be suspended until a reliable unit was in place; to continue field operations without GPS to locate sampling sites would have dire consequences to the study design. On the other hand, if a pH probe were to break or become faulty, sampling could continue without seriously compromising the overall characterization of the environmental condition for a site. It becomes a judgement call, and if the crew has difficulty in making a decision, they should call their State QA Coordinator for guidance.

### **Differential GPS**

A functional differential GPS system provides very accurate positioning data and, when in use on a regular basis, can be relied upon to operate properly from day to day. The units have a signal strength display that indicates the degree of accuracy at which the unit is currently performing. If signal strength is nominal the unit should be accurate within 20 feet; a weak signal may reduce accuracy to a level of 100 feet. Even though the GPS may appear to be problem-free, it should still be periodically verified by checking against a known location, such as the coordinates of latitude/longitude for home dock or a fixed navigational marker. These verifications should be done daily in an informal mode (quick check as vessel is being readied for day) and at least once per week with documentation in the vessel logbook. If the QC check indicates the GPS to be off by more than 200 feet of the known position, wait for a stronger signal or for possible interference to clear then re-check. If the unit consistently fails, a replacement should be put online.

## SeaBird CTD Units

SeaBird CTDs are routinely used in deep water or oceanographic surveys to measure and electronically log various water column parameters. When properly maintained and serviced, they have an established history of dependable utilization. The units can be configured with different arrays of probes; for the purposes of the C2000, the units will be equipped to measure DO, temperature, salinity/conductivity, pH, and depth. Some units may also be outfitted with light sensors to measure transmittance and/or fluorometers to measure chlorophyll concentration.

The CTDs will be subjected to a series of rigorous laboratory performance evaluations and thorough maintenance checks prior to being sent to the field. CTDs will be serviced annually at a certified facility (e.g., the Northwest Regional Calibration Center) for the DO, temperature, and conductivity functions and biennially for pressure sensor (depth) (WA Dept. of Ecology, 1999). In-house calibrations will be conducted monthly on DO and pH sensors and on the light transmissiometer. The calibration procedures will follow those prescribed by Sea-Bird Electronics and should be performed at a facility set up for that purpose.

Because in-the-field calibrations of CTDs are not feasible, QC checks on the core parameters will be conducted daily either by taking water samples from known depths and analyzing them later for DO (field fixed for Winkler titration), pH, and salinity and comparing those results with the logged water column data at the depth, or by conducting a side-by-side, realtime comparison against another water quality monitoring probe (e.g., Hydrolab H20). Depth measurement on bottom can be confirmed onsite by comparing the CTD reading to that on the vessel's depth finder display (not meant to imply that the vessel's depth finder is more accurate, just a quick confirmation that the two instruments are in the same ballpark). The QC check information will be recorded on standardized data forms. The CTD's serial number or property ID will be used to identify the unit; the person performing the QC checks will initial and date the data form. These data will be included (or referenced) in the data package for each station sampled that day using the designated CTD unit. The QC information recorded on the data forms will be transcribed into an electronic file. See Section B5 for the acceptability criteria for the various parameters.

### Hydrolab Water Quality Probes (or similar)

Because Hydrolab Corporation's H20 multiprobe water quality instruments have been extensively utilized in previous EMAP-E monitoring programs, this section will present calibration details specific for that instrument. The actual instruments used for C2000 field monitoring may be models or brands different from the H20, but the procedures discussed here should be generic enough to address the QC issues for most other instruments of a similar design.

Hydrolab Corporation's H20 requires calibration checks on a daily basis during periods of use. The H20 is used to make instantaneous (real time) measurements that are read from a deckside display unit while the probe is lowered and raised at discrete depth intervals (e.g., at 1- m increments) through the water column. Calibration procedures are described in detail in the Hydrolab Scout 2 (display unit) and H20 (probe) Operating Manuals (and Performance Manual) (Hydrolab Corporation, 1991). The Hydrolab units will be used in applications to measure dissolved oxygen (DO), salinity, pH, temperature, and depth. Discussion of the calibration procedures and standards specific to the individual parameters follows.

DO will be calibrated by allowing the probe to equilibrate in an air-saturated-with-water environment, which represents 100% DO saturation at conditions of standard atmospheric pressure (760 mm Hg). This environment is established by positioning the polarographic DO sensor in a calibration cup that is filled with freshwater to a level just below the surface of the sensor's membrane and then placing a lid or cover over the cup to create a saturated humidity. When equilibrium is attained, the operator will activate the Hydrolab instrument to accept the condition as the calibration input for 100% DO saturation. Once calibrated, a properly functioning instrument should hold its DO calibration from day to day with only a slight drift of 2-3% from the 100% saturation standard; drift exceeding that level is indicative of the need to change the membrane and electrolyte solution.

The pH probe requires the establishment of a two point calibration curve using two standard buffer solutions to bracket the nominal range of pH expected to be measured. For C2000, standard buffers of pH 7.0 and 10.0 will be used to calibrate the Hydrolab equipment. The buffer solutions must be commercially supplied with accuracy of  $\pm 0.02$  pH units (or better), referenced to NIST SRMs; calibration solutions should be replaced with fresh buffer every 3-4 days.

The conductivity /salinity cell will be calibrated using a secondary, seawater standard that has had its salinity referenced against a certified standard. These procedures and results data for the preparation of the secondary standard will be logged into a QA notebook that will be maintained by the State Field Coordinators or in-house QA personnel. Salinity of the seawater standard should be generally representative of the conditions expected in the field (e.g., for C2000, a mid-range salinity, 20-30 ppt). A bulk supply (5 gal) of the secondary standard can be maintained in a central location and field crews should replace their calibration allotments (300- 500 ml portions) with fresh standard every 3-4 days, or at any time that it becomes suspect.

The depth sensor (a pressure transducer) is calibrated to 0.0 m of depth while the instrument is non-immersed (absence of water pressure); this in effect becomes the standard for depth calibration.

The temperature function of the Hydrolab instruments is set by the manufacturer and can not be adjusted or calibrated in the field; historically, during 5 years of EMAP activities, there have been no malfunctions with Hydrolab's temperature sensor. However, as part of the daily calibration checks, the instrument's temperature reading will be compared to that of a hand-held laboratory thermometer (accuracy,  $\pm 1^\circ\text{C}$ ) as a pass/fail screen.

## LABORATORY CALIBRATIONS

Analytical Instrumentations: An array of laboratory-based stoichiometric determinations will be conducted with a variety of environmental samples collected for C2000. These analyses require extensive utilization of certified standards for instrument calibration, plus, many incorporate the use of SRMs as a routine QC samples. The analytical standards and SRMs for all analyses will be provided by established, reputable suppliers and when available, only certified materials will be used; in cases where certified standards are not available, the analysts will obtain high purity (e.g., analytical or reagent grade) compounds to prepare in-house standards. Although the following is not a complete list, it will serve to indicate the degree of quality expected for analytical standards used to calibrate and verify analytical instrumentation:

**Analyses of chemical contaminants (e.g., PCBs, chlorinated pesticides, PAHs, and trace metals) in sediments and tissue:**

Organics - NIST calibration solutions and matrix-specific SRMs

Inorganics - NIST or Baker calibration solutions; NRCC reference materials

**Analysis of total organic carbon (TOC) in sediment:**

NIST acetanilide standard

Certified reference materials such as BCSS-1(NRCC)

**Analyses of eutrophication indicators in water:**

Chlorophyll - Chl a extract from *Anacystis* (Sigma Chemicals)

Nutrients - in-house stocks prepared from reagent compounds

In general, instrument calibration for the above analyses should be verified at least twice during a batch run (i.e., continuing calibration check); when appropriate, somewhere near the middle of the run and at the end. If the analyses are run on a continual basis, the end of one run is essentially the beginning of another; if the analysis is down for a period or discontinuous, then an initial calibration check must be conducted with the first batch of the renewed series.

**General Laboratory Equipment:** This category includes the routine tools common to most laboratories (e.g., analytical balances, drying ovens, freezers, etc.); if not actual calibration, all of these require some documentation of performance. Each piece of equipment should have an assigned logbook in which the calibration or performance records are maintained.

Of particular interest are records for the analytical balances used for weighing out standards or analytical samples. These balances must be maintained under the manufacturer's recommended calibration schedule and the performance of the balances should be verified before each series of weighings by using a set of NIST (or previous NBS)-approved standard weights. If the performance of a particular balance is historically stable, then the verifications may only be required on an appropriate periodic basis (e.g., weekly). As much as possible, the verifications should be conducted using standard weights that reflect the magnitude of the actual weighing. The results of the verifications should be recorded in the logbook for the balance.

Certain of the C2000 samples (e.g., dissolved nutrient and chlorophyll) require storage under extremely cold conditions ( $< -50^{\circ}\text{C}$ ). These samples should be held at  $-70^{\circ}\text{C}$  in an ultrafreezer that will activate an alarm if the temperature exceeds  $-65^{\circ}\text{C}$ . Other equipment such as sample drying ovens should be monitored on a routine basis during periods of use ensure their performance.

**B8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES**

Element required for QA Category I documents only.



## **B9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)**

Coastal 2000 will utilize Geographical Information System (GIS) applicaitons to plot data collection stations on maps that can be used for logistical planning as well as to generate gradient presentations based on the results of the monitoring (e.g., demarcation of low DO conditions). The estuaries of the U.S. Pacific will be extracted from a U.S. Geological Survey digital line graph (1:100,000 scale) hydrographic layer to create an estuary basemap for each WP subregion (state). The uncertainty associazted with this approach for ground siting and graphic presentaiton is intrinsically linked to the resolution attainable at the scale of 1: 100,000.

## **B10 DATA MANAGEMENT**

### **Information Management System**

Because of the multiple organizations participating in Coastal 2000 and the sheer volume of data they will generate, a tiered, National Information Management System has been developed to systematically collect, aggregate, and transmit data (Hale et al., 1999). Individual states will submit appropriately formatted data to respective regional data nodes. There, the data will be verified, reviewed for QA, and further formatted as specified in Appendix B: Coastal 2000 - Information Management (USEPA, 2000) for transmission to the national collection node and incorporation into the EMAP National Coastal Database. Long-term archival will be in STORET (STORET 2000).

Each regional data collection node will have latitude in designing their own data management system as long as they comply with the requirements set by the National Information Management System for the submission of the finalized data sets to the national database.

During the 1999 EMAP-Western Pilot Coastal Monitoring (WPCM), the Southern California Coastal Water Research Program (SCCWRP) developed a regional data management system for the WPCM that has been further revised for use in C2000- West Region. A copy of "West EMAP Revised Information Management Plan For 2000" (SCCWRP, 2000) is appended to this document (Appendix C) as an example of a proven IM system for use on the regional level. Basically, raw data (either hardcopy or electronic) are transferred from their source (field or laboratory) to respective State IM Coordinators for initial review and grooming (conversion to a standardized electronic format developed by SCCWRP); the groomed electronic data sets are then transmitted on to the Western EMAP IM Coordinator at SCCWRP where additional formatting and data verification is performed before the data are approved and entered into an in-house, regional database. At that point, the data are readily available to all participating WP partners for use in preparing state or regional assessments, reports, and publications. Finalized WP data sets will be submitted to the EMAP Information Management Coordinator at the EMAP Information Center, EPA's Atlantic Ecology Division in Narragansett, RI for archiving and posting on a public website.

## C. ASSESSMENT/OVERSIGHT

### C1 ASSESSMENT AND RESPONSIVE ACTIONS

Coastal 2000 represents a matrix of diverse environmental monitoring measurements and data acquisition activities. Data quality criteria have been established for most of these measurements and the QA program will monitor the success rate of C2000 in meeting the quality goals. While all of the data acquisition activities are of value to the project, certain of them have a higher degree of import than others and will, therefore, receive priority regarding review and assessment of the data quality, especially in the more structured format of audits. Nonetheless, for those activities that are not audited, there are sufficient QA/QC elements associated with each data generating activity to enable the responsible analyst to make a determination on the acceptability of the data. In most cases if the process fails QC checks, the QA policy requires that the samples be re-analyzed until acceptable data are attained. The following sections outline the structured data reviews and assessments of data quality planned for C2000. Note, if situations warrant, any QA Coordinator delegated C2000 responsibilities will have authority to initiate an audit or review of any C2000 environmental data collection activity that fall under their purview. The States may also elect to initiate audits of their respective in-house activities, at anytime.

#### FIELD MONITORING

##### **Field Crew Certification**

Prior to the start of the 2000 field monitoring, each field crew will be required to complete a 3-4-day field training to be authorized to collect actual C2000 field data and samples. Training will consist primarily of hands-on sessions during which field crew members will be instructed by the Regional QA and Logistics Coordinators (and associates) on the sampling methods and protocols developed for C2000. If the schedule permits, training for each crew should culminate with a certification exercise in which crew members are observed and evaluated as they perform the full suite of core field activities (i.e., complete sampling for a C2000 site). Although that is the preferred approach, because of time and logistical constraints, it may be necessary to certify the crews as they master each major component (e.g., sediment grabs for surficial sediment), then move on to the next, without observing in the context of a real world situation. Crews that successfully demonstrate technical competence and a thorough appreciation of field QA/QC requirements will have a letter of certification issued from the Regional QA Coordinator addressed to their State Project Manager; the crew will then be authorized to initiate C2000 field activities. If a crew fails to qualify on some aspect, the members will receive further instruction in the area of their deficiencies until they perform at an acceptable level.

##### **Field Reviews**

State field teams will be responsible for the collection of environmental data and samples from the majority of C200 sampling sites. An important element of the C2000 strategy is to build upon the existing state programs, as much as possible. However, it is necessary to maintain an acceptable degree of uniformity between the multiple groups conducting these tasks. C2000 develops standard protocols and guidelines to help ensure that the data collected are of known quality. These guidelines allow for the use of different equipment (e.g., various hydrographic meters,

work vessels, etc.) as long as the data generated meet C2000 acceptability criteria. Such performance-based QA/QC is a key factor to C2000's success in deriving comparable data from diverse participants. Prior to the actual collection of C2000 field data, the field crews are instructed in the approved field methods and protocols during their required initial training.

To further ensure that the actual field collections are conducted in accordance with C2000 standards, the performance of field crews will be periodically evaluated. The format for the evaluations will be more of a field "surveillance review" than "audit." The surveillance reviews will be conducted by appropriate C2000 Regional personnel. The goal is to conduct at least one review per crew per year. The evaluator will meet the crew in the field and accompany them as they conduct full-scale monitoring activities at one or more sampling sites. The evaluator will use an approved checklist to systematically document acceptable/unacceptable performance on all pertinent aspects of the sampling. The checklists should be generated and approved at the regional level to reflect geographical or resource differences (see the attached example of the checklist used for the Gulf/Southeast Region). Because the activities are generally carried out in concert by the crew, the evaluation will be based on the performance of the crew as a team. The field evaluation checklist will be retained at the Regional Centers as part of the permanent record.

Any minor deficiencies observed during a field surveillance (e.g., slight deviation from approved procedures labeling irregularities, data reporting, etc.) should be immediately pointed out to the crew and corrective actions imposed on-the-spot. The evaluator will document with a brief note on the checklist and no further writeups are required. If significant deficiencies (i.e., data quality is seriously compromised) are observed, the evaluator will make the appropriate on-the-spot correction, and, if the case warrants, call a halt to the field activities until the problems are resolved to the satisfaction of the Regional QA Coordinator. All cases of this nature will be documented through a written report submitted to the Regional QA Coordinator.

An example of the C200 Field Crew Evaluation Checklist used in Gulf/Southeast Region during the 2000 sampling is attached in Appendix F. Also, a blank set of the standard Field Data Forms is attached as Appendix E. A completed checklist along with a copy of the completed field data forms from the station, provide the basic documentation for an evaluation of the crew's overall performance at that site.

## LABORATORY ACTIVITIES

### **Analytical Chemistry**

The analyses of chemical contaminants (organics and inorganics) in environmental samples are the more difficult analytical activities within the project. C2000 has a vigorous performance based QA/QC program to help ensure that data are of known and acceptable quality (see Appendix A of this document for detailed description). Because these analyses are technically challenging and relatively expensive to conduct, C2000 will require each analytical laboratory to successfully complete an initial demonstration of technical capability, prior to being authorized to conduct analyses with actual C2000 samples.

First the laboratory must demonstrate that it is capable of meeting the target method detection limits (MDLs) for each analyte of interest in the matrices to be analyzed. Each laboratory must

calculate and report MDLs following the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984). The matrix and the amount of sample used to determine MDLs should match as closely as possible the matrix and amount of sample that will be used in the analyses of the field samples.

After generating acceptable MDLs, the next step of the qualifying process will be for the laboratory to analyze a “blind” (unknown) representative sample issued by the Regional QA Coordinator. Typically this performance evaluation (PE) sample will be an SRM or other reference sample with documented analytical results and the laboratory’s results will be evaluated against the known values. The requirements for acceptable performance are: organics,  $\pm 35\%$  general agreement between laboratory’s results and accepted values; inorganics,  $\pm 20\%$ . These criteria only apply to those analytes with accepted values at levels that are  $\geq 10\times$  the laboratory’s declared MDLs for the analyte of interest. Only after the C2000 is satisfied with a laboratory’s demonstrated technical competence, will the lab be authorized to begin analyses with the C2000 field samples. Authorization, based on consensus agreement between the Regional Project Manager and QA Coordinator, will be issued to the laboratory in written format.

Routine analyses of C2000 samples will be conducted in batch runs consisting of 25 or less field samples along with a full complement of QC samples, typically including: continuing calibration curves, reagent blanks, matrix spikes (MS) and MS duplicates, and a reference material (either a SRM or a laboratory control material). These QC samples should be sufficient to allow the analyst, on a real time basis, to evaluate the overall data quality of the sample batch; please refer to Appendix A for a comprehensive discussion of the performance-based QC philosophy and components. If the quality criteria are not met, the analyst should take corrective actions and rerun the batch. When laboratories adhere to this level of in-house data review, only batches that pass the general QC checks should be submitted as final data to the C2000.

Data reports submitted for to C2000 from analytical chemistry laboratories should include the results of all required QC samples. These data will be thoroughly reviewed by C2000 personnel to verify that the quality goals were satisfied. Analytical results that do not meet the general QC requirements will be identified in the C2000 data set with an appropriate QC code; the Regional QA Coordinator will assign/approve the qualifier codes.

Laboratories conducting C2000 analyses are subject to audits at all phases of their association with the project. The audits can be relatively informal site visits or technical systems audits (TSA) conducted prior to, or early in, the project, primarily to confirm that the laboratory has appropriate facilities, personnel, and resources required to conduct the analyses. A more formalized “audit of data quality” may be scheduled after the analyses are well underway or completed, but not beyond a 2-year period of their completion. Audits of data quality are formatted to determine if the QA/QC requirements outlined in the QAPP were in fact followed and documented. If at all possible, C2000 will conduct both TSAs and audits of data quality for each analytical laboratory participating in the project. These audits will be announced well in advance (no surprise audits). However, C2000 retains the right to request periodic briefing on the status of QA/QC or specific QC data at any time and if there is reason to suspect that the quality standards are not being met, the C2000 management (i.e, Project Manager or QA Coordinator) can suspend the analysis until the laboratory demonstrates the analytical process is back in control.



## Water Quality Analyses

This suite of analyses consists of separate laboratory determinations for several indices of eutrophication conditions in water (e.g., soluble nutrient levels and chlorophyll content). Although different methods and instrumentation are utilized for the specific measurements, all are conducted using analytical systems that incorporate similar QC requirements (e.g., standard curves, blanks, replicates, and spikes or reference samples) on a batch basis. The QC elements provide the analyst with an immediate indicator of the data quality for a given batch of field samples. If a batch run is substandard, the analyst should halt the analytical process until the problem has been resolved. If the problem is straightforward, the analyst should make the appropriate corrective actions, document the event, then continue or repeated the analysis. If the problem appears complex, for example - such that the entire data set is jeopardized, then the analyst (or laboratory) must inform the State or Regional QA Coordinator of the situation and await further guidance before resuming with the analysis.

The performance level for these analyses will be assessed during several stages of their conduct. First, in keeping with the general QA policy for C2000, an initial demonstration of capability will be required for each analysis before the C2000 field samples are analyzed. The performance evaluations may include the analysis of a blind sample, but since certified SRMs are not available for most of the determinations, technical competence may be confirmed by reviewing the basic QC checks for a particular determination during preliminary analyses. The C2000 or Regional QA Coordinator must first approve the overall performance for the analytical process before the laboratory (or analyst) is authorized to proceed with the analysis of C2000 field samples. C2000 management personnel will attempt to visit each group, firsthand, and observe the analyses while in progress. If at any time, C2000 management is not satisfied that the quality standards are being met, the analysis may be suspended until corrective measures are taken and the analysis is shown to be under control. The data report submitted by each group should include all QA/QC results. An audit of data quality may be conducted for any of the analytical activities within 2 years following their completion.

## Sediment Characterizations

Percent Silt-Clay - Sediment grain size will be characterized as percent silt-clay. The procedures, while tedious, are basically a gravimetric determination. The primary QA governing this analysis is strict adherence to the methods described in EMAP-Estuaries Laboratory Methods Manual - Vol. 1 (US EPA, 1995). The QC checks for this activity involve replicate samples (10% of all samples) as a check on precision; there are no accuracy-based checks. If the QC replicate fails the quality criteria, the technician will re-analyze all samples from the failed batch.

Before silt-clay determinations are conducted with actual C2000 samples, the laboratories slated to perform the assays may be provided with a series of performance evaluation samples representing the range of silt-clay expected in the CM sediments. The results for the PE samples, as well as the degree of overall technical competence exhibited, will be reviewed by C2000 management. The laboratory must demonstrate consistently valid results before receiving authorization from the QA Coordinator to begin the silt-clay determinations with actual CM samples. An audit of data quality may be conducted for this activity at anytime during a 2-year period following its completion.



Total Organic Carbon (TOC) - Sediment samples from each C2000 sampling station will be analyzed for TOC. These analyses will be conducted by using a TOC analyzer; QC samples including carbon standards, blanks, duplicate samples, and a SRM will be utilized on a per batch basis. Once the TOC analyzer is calibrated, the analysis is relatively straightforward. Prior to the startup of actual C2000 sample analysis, the analyst must demonstrate that the instrument is in calibration and producing precise, accurate results for a certified reference material. The C2000 field samples should be analyzed in batches of 25 or less samples; the analyst will review the results of the QC samples upon the completion of the analytical run. If the quality criteria are not met, the batch will be re-analyzed. Sediment TOC data is subject to an audit of data quality during the 2-year period following the completion of the analysis.

### **Benthic Community Assessment**

Sediment grabs will be collected from each C2000 station for evaluations of macrobenthic infaunal community structure. These types of benthic evaluations should only be undertaken by experienced personnel with demonstrated competence in the field of benthic ecology. An established regime of in-house QC checks will be adhered to in which a portion of each technician's work is reviewed by a senior taxonomist; a failed check requires that all of that technician's samples, since the last passed check, be re-sorted or re-identified (depending on the assigned task). The same type of QC checks apply throughout the process of identifying and quantifying the benthos; technicians and taxonomists have their work verified by a peer or more senior taxonomist. The QC checks must be well documented in a laboratory notebook that will be available to C2000 QA personnel upon request. The benthic data will be subject to an audit of data quality during the 2-year period following the completion of the benthic community assessments.

### **Histopathological Examination of Fish**

Fish trawls will be conducted at each of the C2000 sampling stations where possible. The catch will be inventoried by species and total length measured for a representative subsample of each species. As the fish are sorted and processed, each individual will be briefly examined for evidence of external gross pathology (e.g., tumors, lesions, fin erosion). If a pathological condition is encountered, the fish will be immediately preserved in Dietrich's fixative and held for later submission to a designated laboratory where trained pathologists will conduct in-depth histopathological evaluations. Because these activities will be limited to events only when affected fish are caught and because of the highly specialized and research aspects of study, C2000 will not set QA standards for histopathological evaluations. However, standard procedures for the routine laboratory examination of finfish for pathological abnormalities are described in Section 4 of the EMAP-Estuaries Laboratory Methods Manual Vol.1 (US EPA, 1995). The QA/QC recommendations for these studies are that the samples be properly preserved (field activity) and that qualified pathologists conduct the laboratory examinations.

## C2 REPORTS TO MANAGEMENT

During the implementation and execution of C2000, several reports are required to appropriately document QA/QC activities and to ensure that management is aware of pertinent items related to the general status of the project. The following reports will be expected on a routine basis, but other reports may be warranted as situations dictate.

### **Status Reports**

Periodic status reports should be generated from both the participating investigators and from within the C2000 management team. Each core activity should submit a general summary report stating their progress on the tasks with emphasis directed to any QA/QC issues. The schedule for these reports will differ depending on the complexity and duration of the activity.

### Field Teams

The field supervisors should update their State or Regional Coordinators on the general status of the field team's activities on a regular basis (e.g., weekly, and any time that significant problems arise). These updates will be informal and can be communicated by telephone or e-mail. Although not required, a carbon copy or similar briefing to the Regional QA Coordinator provides a realtime overview on the progress or problems related to ongoing field collections.

### Regional Coordinators

Regional Coordinators should update the EPA Project Officer with monthly status reports on the operations under their supervision (e.g., field monitoring, laboratory analyses, and data management). These reports should briefly address accomplishments, problems, and anticipated needs. Direct communication through conference calls between the C2000 management team members may preclude the need for written status reports on a monthly basis, however, the EPA Project Officer has the authority to call for written status reports at anytime.

### **Performance Evaluations and System Audits**

The results of initial laboratory performance evaluations (PEs) will be submitted to Regional QA Coordinators for review. If the laboratory's results clearly meet C2000 quality criteria, the Regional QA Coordinator will issue a letter of approval to the laboratory authorizing them to commence analyses or processing with C2000 samples. If the laboratory's initial PE results appears deficient, the Regional QA Coordinator will report of his assessment and recommended actions to the C2000 EPA Project Officer and QA Coordinator for concurrence or alternative corrective action. Based on that outcome, the Regional QA Coordinator will then issue a letter to the laboratory detailing the recommended actions.

The results of all system audits (e.g., facility visits or field reviews) will be reported by the reviewer to the QA Coordinator (if other than he conducted the review). The QA Coordinator will evaluate the review and formulate corrective actions where needed. As with PE evaluations (discussed above), if there are no significant deficiencies, the Regional QA Coordinator will issue a

final report of the audit results and the corrective actions, where needed, to the EPA Project Officer and C2000 QA Coordinator, with copies sent to all key personnel involved with the project audited. If the audit results indicate serious problems or deficiencies, the Regional QA Coordinator should immediately notify the EPA Project Manager and C2000 QAC. Based upon a consensus agreement, the Regional QA Coordinator will issue a letter, to the laboratory/activity under review, detailing the plan of action.

### **Periodic Data Assessment and Quality Assurance Issues**

The Regional QA Coordinators will remain in contact with their respective Regional Project Manager through personal communications during the extent of the project. As specific phases of the project are completed (e.g., organic analyses with sediments), the Regional QA Coordinator will provide the Regional Project Manager, C2000 QA Coordinator, and EPA Project Officer with a summary report detailing the overall data quality for that activity. These reports will be issued on a case-by-case basis. The State Project Managers will also receive copies of these reports related to their respective state's activities.

When audits of data quality are conducted onsite, the lead auditor should issue a short verbal briefing to the key personnel at the facility being audited as part of an exit interview. The briefing should address any significant observations, both positive and negative, and provide the staff with a general sense of the audit's results. If possible, a short written interim report should be prepared by the audit team and left with the appropriate staff members. A formal written report of the audit results will be issued within a month by the audit team addressed to the C2000 EPA Project Officer and distributed to the C2000 QA Coordinator, appropriate Regional Project Manager, appropriate State Project Manager, and the appropriate senior staff at the facility audited.

Anytime, when a significantly negative QA issue is encountered, it must be immediately reported to the Regional QA Coordinator or Project Manager, who will assess the matter and, if necessary, consult with appropriate advisors to formulate corrective actions. Finding of this nature must be detailed in a report submitted to the C2000 EPA Project Officer.

After the completion (all analytical results reported) of the Coastal 2000 Monitoring, the C2000 management team will issue a QA Summary Report for the entire study. This report will be submitted to the C2000 EPA Project Officer and will also be made available to all C2000 participants that express interest.

## **D. DATA VALIDATION AND USABILITY**

### **D1 DATA REVIEWS, VALIDATION, AND VERIFICATION REQUIREMENTS**

The data generated by Coastal 2000 will be evaluated at several junctures of the along their pathway from source to final incorporation into the C2000 database.

The first and, therefore, a very critical level of data review, validation, and verification of C2000 data will be conducted at the state-level when the raw data from the field or laboratory are reviewed while being formatted for transmission to the Regional Data Node. Participating investigators should submit final data package(s) to C2000 State Managers that consist of: a cover letter signed by the Principal Investigator; hard copies of all results (including QA/QC results); and accompanying computer diskettes (even, as in some cases, the data are directly transmitted to the Regional Data Node). If the laboratory has adhered to C2000's performance-based QA/QC requirements prescribed for their activity during the analytical phase, the submitted data should be in a reasonably sound condition. Data packages received by a state will first be reviewed by the state's designated QA Lead for basic completeness and content (i.e., are these the data requested and are they expressed in appropriate units and format?). The overall data quality of each data set will then be evaluated in terms of accuracy and precision (when applicable) using the quality criteria described in this QAPP (see Section B5). These data reviews may be conducted by either the state's QA Coordinator or other qualified state personnel (e.g., Project Manager, Information Manager, and persons with specific expertise). The Regional QA Coordinators may assist with the state-level data reviews (e.g., offer advice and guidance), but should not be expected to perform these first-cut reviews; they would simply be overwhelmed by the load.

After data are received at the Regional Data Node, the IM will further groom the data sets and ready them for review by the Regional QA Coordinator. Data sets that meet the prescribed quality criteria will be accepted without further qualification for use in making environmental assessments of the estuarine systems of the U.S. Coastal regions. Data that do not meet all of the C2000 acceptability goals because of minor deficiencies will be assigned data qualifier codes to "flag" the values in question and they may still be included in the data set as estimates. This will enable individual data users to decide for themselves whether the data are acceptable for their specific purposes. Because of the multiple indicators and the diverse nature of possible data deficits, at this point, a list of data qualifiers will not be issued, but the list is currently being developed by C2000 QA and IM staff. As the data are reviewed, the appropriate qualifier codes with their definitions will be appended to each data file. Flagged data will be reviewed by C2000 management on a case-by-case basis to determine if the data are acceptable for making environmental assessments of the estuarine resource on regional or national levels. Data that consistently fail one or more quality criteria by a significant margin will be rejected and not used for C2000 assessments.

## D2 VALIDATION AND VERIFICATION METHODS

Data generated for the Coastal 2000 will be systematically reviewed with varying levels of scrutiny at several junctures along the path from time of collection to final reporting; from quick, on-the-spot screening to in-depth evaluation against established criteria or standards. For much of the field collected data, the first level of validation, a cursory screening, will occur as data are recorded; persons conducting and documenting realtime observations should be aware of the range that constitutes realistic values for a specific measure. Certainly a water temperature of 40° C in the Pacific NW should jump out as an obvious outlier and trigger an immediate response to find the source of the error. With other types of data, the initial validation may not occur in such an immediate time frame; for example, in the case of nutrient analysis, the analyst may first need to run several calculations to arrive at a meaningful result. Nonetheless, most data are amenable to some form of quick screening soon after being generated and the responsibility for this is first- cut validation falls on the personnel performing the measurement. In addition, most laboratory analyses of C2000 samples will be monitored by a series of in-stream QC checks that indicate the general level of data quality for a given batch of samples. If routine screens and QC checks are adhered to and proper corrective measures enacted, there is little reason for seriously flawed data to make it any further down the data stream. However, that assumption cannot be totally relied upon, so additional, documented verifications are required to determine if data quality remains at a level acceptable for the program. The following sections outline the format and procedures to be used for evaluating and documenting data quality for C2000 and discuss how issues will be resolved when they occur.

Using the West Region for an example case, the following discussions on data validation and verification, will reference the regional data collection system developed by SCCWRP for West EMAP activities; actual procedures and details will vary slightly from region to region.

### FIELD COLLECTED DATA

C2000 field crews have the option to record field data on hardcopy data sheets or use the field computer system to directly enter the information, or a combination of both. The field computer system has a separate page for each of the primary activities conducted during the field sampling (e.g., Station Data, Water Quality Data, Sediment Data, and Fish Data). The pages from the computer system generically resembles hardcopy data sheets used for previous EMAP studies. The system queries the crew for specific information relevant to a sampling activity in a manner that systematically leads them through the preferred sequence of steps for collecting the field information. Regardless of the mode used to initially record data, all field data will be entered into the field computer system soon after collection (within the week is recommended). Upon completion of the sampling cycle (mid-September to early-October), each State IM Coordinator will submit their state's entire set of electronic field data to the Regional Information Node.

### Validation of Field Data

In the context of this document, the definition of "data validation" can be expressed as a series of questions: are the data received actually the data expected? are the data expressed in correct units? are the data realistic? and are the data complete? In other words, "I was expecting one dozen oranges, did I get one dozen oranges?"



As mentioned, first-cut validation of field data occurs as the data are being collected by the field crews (e.g., are these data in the ballpark?). If the field personnel encounter situations where they question the validity of data they are collecting, they should immediately attempt to isolate and resolve the problem; if they are unable to do so, then they should describe the situation in writing on the appropriate data sheet then, as soon as possible, consult with their respective senior Field Coordinator or State Project Manager for corrective actions.

The next level of validation takes place as the State IM Coordinator consolidates and formats the field data for transfer to the Regional Information Node. Apparently, most crew will use hardcopy data sheets to record the bulk of field data, therefore, the data must be transcribed into the field computer system. As soon as possible, upon return from the field, all raw data forms should be xeroxed and the originals then placed in a secure file; the copies can then be used for entering the data. During the data entry process, the State IM Coordinator will screen the field data for missing or errant information. Any observed deficits should be notated in a bound logbook. If corrective actions are initiated (e.g., correcting a spelling error on the copied data form), the correction must be legible and the person who made the correction must document the alteration with their initial and date; a description of the correction should be noted in the bound log.

Once the field data are transmitted to the Regional Information Node, they will again be systematically scanned for outliers and checked for units and completeness.

#### Verification of Field Data

Where “data validation” is a determination that the collected data appear appropriate and are expressed in the correct format, “data verification” is more of a process to evaluate the level of data quality (e.g., representativeness, accuracy and precision). Verification of field data involves a more critical review of QC elements or acceptance criteria such as calibration success for hydrographic equipment, acceptability of sediment grabs, siting of a station, duration and number of fish trawls, etc. These types of evaluations can be and should be executed at each stage of the process from data collection to final review prior to data being posted to the public. However, there must be several structured check points where documented verifications are performed.

#### Transcription Errors

One of the first reviews field data are subjected to is an evaluation on the relative frequency of transcription errors enacted going from hardcopy into the electronic format. This evaluation will be performed at the state level under the direction the State IM Coordinator. To determine this, a randomly selected subset of at least 10% of the station packages (the entire set of field data sheets submitted for a given station) will be pulled and the data (primarily, measurements or numerical values) manually compared against the electronic version on a field - by-field basis. Any errors will be listed in the bound logbook (see above, Field Data Validation) and a final tally derived for the station. The total number of transcription errors for a complete set of data sheets should not exceed 5.



### Verification of Field Measurements

Measurements of water quality parameters taken directly in the field will be evaluated for accuracy by verifying the results of calibration and QC checks. These checks should be performed by the field crews on a daily basis and if the instruments are out of tolerance, they should be re-calibrated. At the conclusion of the summer sampling, copies of the field records for calibration and QC checks will be provided to the State QA Lead for further review. Any data that was collected, when the instruments were out of compliance, will be flagged with a qualifier code. The Regional QA Coordinator may, at any time, request access to or copies of state field calibration and QC records.

Other field collected data that will be evaluated on a randomly selected subset of the field data include penetration depth for benthic grabs, light-down/light-up comparisons, trawl times, and difference (distance) between intended site and actual site. These evaluations will be conducted by the Regional QA Coordinator in conjunction with the Regional IM Coordinator, but the states are encouraged to conduct similar verifications on their own.

### LABORATORY GENERATED DATA

All laboratory data generated for the C2000 will be systematically reviewed and evaluated. Laboratories that perform the analyses will conduct their internal QA/QC verifications prior to submitting the data to the State IM Coordinator. Laboratory data will be submitted in accord to the Standardized Data Transfer Protocols (STDP) specified in SCCWRP, 2000; the STDP stipulate that data be submitted in comma-delimited, ASCII format. The following discussion on data flow and verification is taken from the Section III.(Roles and Responsibilities) of the above IM plan.

Upon receipt of a data set, the State Information Management Coordinator (SIMC) will create a temporary file and initiate a series of error checks to ensure the data : 1) are within specified ranges appropriate to each parameter measured, 2) contain all required fields, 3) have encoded valid values from constrained look-up lists where specified, and 4) are in the correct format (text in text fields and values in numeric fields, etc.).

If the data emerge from the error check routine with no errors or suspected outliers, the SIMC will append the temporary table for that data type. If there are only a few, easily correctable errors, the SIMC will make the changes, with the consent of the submitting agency. If there are numerous errors or the corrections are difficult to implement, the SIMC will send the data back to the submitting agency with a list of necessary corrections. The submitting agency will make the corrections and resubmit the file to within one week to the SIMC who will subject the file to error checking again. Each of these paths will be documented by the SIMC as part of the submittal tracking process.

When all data for received for a particular laboratory function have been submitted, error checked, and corrected, the SIMC will certify that the file is consistent with the STDP format and complete. The completed data set can then be transmitted to the Regional IMC who also will assess that the file is complete and consistent with the SDTP format. If there are a few minor, correctable errors, then the Regional IMC will make the corrections and send a list of documenting the changes to the SIMC (who, in turn, will send them to the data generator). Changes will only be made with the

consent of the SIMC (who will be responsible for contacting the data generator). If there are extensive errors, the regional IMC will send the file back to the SIMC for the necessary corrective action.

Once a complete set of data are certified by the regional IMC, the Regional QA coordinator is notified that the data are ready for technical review. The review will involve plotting of data and examining interrelationships among individual parameter responses and will address more extensive data quality issues than can be accomplished by range checking alone. Any further corrections resulting from such review processes will be documented by the Regional IMC, who will determine whether he can make the changes or if the data must be returned to the submitting agency for correction and resubmittal.

The Regional QA Coordinators and Regional Project Managers will be responsible for conducting technical reviews of the data before the data are accepted for C2000 assessments; certain aspects of these reviews may be delegated to other staff with final approval through the above quality management personnel. Data quality of a specific data set will be assessed by a critical comparison of the submitted QA/QC results to the quality criteria or standards established by this QAPP for that analysis. If the evaluation indicates that the data, overall, meet the quality standards, with no or only minor deficiencies, then the data set will be acceptable for C2000 assessments without further qualification. If the data consistently fail one or more quality criteria, then the data set will be flagged with an appropriate data qualifier code. Depending on the degree of the deficiency, the data might still be used in certain C2000 assessments (provided that data clearly carry the appropriate qualifier code), or they may be dropped entirely from the accessible C2000 database.

Upon completion of technical review, corrected, qualified data sets will be finalized by the Regional IMC and a regional working database will be generated and available to all state and federal partners for use in preparing regional or subregional assessments, reports, and other publications. This working database will reside at the Regional Information Node. However, after additional formatting in accord to the protocols for EMAP, the Regional data will be transferred to the EMAP IM Center at EPA-AED, Narragansett, RI, for final reposition and ultimate posting on a public webpage.

### D3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

Coastal 2000 will serve multiple functions: to provide standardized data to characterize the environmental conditions in a regional ( e.g., U.S. Pacific Coast) or in a subregional (e..g., individual states: CA, OR, and WA) estuarine system, which, in turn, can be used as a component on a national scale; and, also, to evaluate the efficacy of the U.S. EPA's role as a steering element, responsible for the coordination of the monitoring activities conducted by state and other federal agencies, rather than implementing the project solely based on EPA support.

Coastal 2000 is in fact a demonstration program and, as such, the need to reconcile results from this first year of monitoring to the proposed project Data Quality Objectives (DQOs) is not totally germane. The project represents an experimental application that should not be bound by success/failure criteria, but rather an iterative success/revision approach. For these reasons, C2000 will use Method Quality Objectives (MQOs) to evaluate success on a component level, in addition to project DQOs as criteria for the overall sampling design.

The C2000 management team will be advised on the QC results for the individual monitoring and analytical activities as evaluated against the MQOs or quality goals established in this QAPP. Each activity for which QA/QC guidelines were described should submit a summary of those results along with their analytical results. If the data quality for a particular indicator is substandard, C2000 management will be charged with the decision to: 1) if consensus agreement is reached that existing criteria are overly stringent, revise the quality criteria to reflect the level of data quality attained and then use the data for environmental assessments; 2) totally reject the use of the data for environmental assessments; or, 3) flag the deficient data with qualifiers and use it conditionally for environmental assessments.

After a thorough assessment of the 2000 data, Coastal 2000 management will retain those indicators that appear to be efficacious for future monitoring projects in the following year of C2000 or of other subsequent EMAP-sponsored monitoring projects. Indicators that fail to produce acceptable data will be revamped or suspended.

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## APPENDIX A

### ANALYSIS OF CHEMICAL CONTAMINANTS IN SEDIMENTS AND FISH TISSUE <sup>1</sup>

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#### <sup>1</sup> IMPORTANT NOTE

The information contained in Appendix A was taken verbatim from “Section 5 - Analysis of Chemical Contaminants in Sediment and Fish Samples” as it appeared in EPA-EMAP-Estuaries QAPPs for 1990-1995. Section 5 was thorough and well written, particularly in reference to performance-based QA/QC. Since the National Coastal Assessment evolved directly from EMAP, most of the details in Section 5 remain application to the analytical processes associated with the National Coastal Assessment. However, Appendix A material is dated and slight discrepancies (e.g., analyte lists of MDLs) exist between the information in the appendix and that presented in the main body of the National Coastal Assessment QAPP. When such discrepancies are encountered, **the guidelines described in the main body of the QAPP take precedence.**



## ANALYSIS OF CHEMICAL CONTAMINANTS IN SEDIMENT AND FISH TISSUE SAMPLES

### 5.1 OVERVIEW

Quality assurance of chemical measurements has many diverse aspects. This section presents EMAP-Estaries QA/QC protocols and requirements covering a range of activities, from sample collection and laboratory analysis to final validation of the resultant data. Much of the guidance provided in this section is based on protocols developed for EPA's Puget Sound Estuary Program (U.S. EPA 1980), as well as those developed over many years on the National Oceanic and Atmospheric Administration's (NOAA) National Status and Trends (NS&T) Program. This guidance is applicable to low parts per billion analyses of both estuarine sediment and tissue samples unless otherwise noted.

The EMAP-E program measures a variety of organic and inorganic contaminants in estuarine sediment and fish tissue samples (Tables 5-1 and 5-2); these compounds are the same as those measured in the NOAA NS&T Program with a few additions. These contaminants are being measured for the purpose of environmental monitoring, with the understanding that the data will not be used for litigation purposes. Therefore, legal and contracting requirements as stringent as those used in the U.S. EPA Contract Laboratory Program, for example, have not been applied to EMAP-E. Rather, EMAP-E requires its laboratories to demonstrate comparability continuously through strict adherence to common QA/QC procedures, routine analysis of Certified Reference Materials<sup>1</sup>, and regular participation in an on-going series of QA intercomparison exercises (round-robins). This is a "performance-based" approach for quality assurance of low-level contaminant analyses, involving continuous laboratory evaluation through the use of accuracy-based materials (e.g., CRMs), laboratory fortified sample matrices, laboratory reagent blanks, calibration standards, and laboratory and field replicates. The definition and use of each of these types of quality control samples are explained in later sections.

No single analytical method has been approved officially for low-level (i.e., low parts per billion) analysis of organic and inorganic contaminants in estuarine sediments and fish tissue. Recommended methods for the EMAP-E program are those used in the NOAA NS&T Program (Lauenstein et al. 1993), as well as those documented in the EMAP-E Laboratory Methods Manual (U.S. EPA 1992, in revision). Under the EMAP-E performance-based chemistry QA program, laboratories are not required to use a single, standard analytical method for each type of

<sup>1</sup> Certified Reference Materials (CRMs) are samples in which chemical concentrations have been determined accurately using a variety of technically valid procedures; these samples are accompanied by a certificate or other documentation issued by a certifying body (e.g., agencies such as the National Research Council of Canada (NRCC), U.S. EPA, U.S. Geological Survey, etc.). Standard Reference Materials (SRMs) are CRMs issued by the National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards (NBS). A useful catalogue of marine science reference materials has been compiled by Cantillo (1992).

analysis, but rather are free to choose the best or most feasible method within the constraints of cost and equipment. Each laboratory must, however, continuously demonstrate proficiency and data comparability through routine analysis of accuracy-based performance evaluation samples and reference materials representing real-life matrices.

## **5.2 QUALITY CONTROL PROCEDURES: SAMPLE COLLECTION, PRESERVATION AND HOLDING**

Field personnel must strictly adhere to EMAP-E protocols to insure the collection of representative, uncontaminated sediment and fish tissue chemistry samples. These sample collection protocols are described in detail in the Louisianian Province Field Operations Manual (Macauley 1997). Briefly, the key aspects of quality control associated with chemistry sample collection are as follows: 1.) field personnel must be thoroughly trained in the proper use of sample collection gear and must be able to distinguish acceptable versus unacceptable sediment grab samples or fish trawls in accordance with pre-established criteria, 2.) field personnel must be thoroughly trained to recognize and avoid potential sources of sample contamination (e.g., engine exhaust, winch wires, deck surfaces, ice used for cooling), 3.) samplers and utensils which come in direct contact with the sample should be made of non-contaminating materials (e.g., glass, high-quality stainless steel and/or Teflon) and should be thoroughly cleaned between sampling stations (e.g., Alconox scrub followed by thorough rinse with ambient water), 4.) sample containers should be of the recommended type (Table 5-3) and must be free of contaminants (i.e., carefully pre-cleaned), 5.) recommendations for sample collection, preservation and holding times should be followed (Table 5-3).

**TABLE 5-1.** Chemicals to be measured in sediments by Estuaries Louisianian Province.

<b>Polynuclear Aromatic Hydrocarbons (PAHs)</b>		<b>21 PCB Congeners</b>	
Acenaphthene		<u>PCB No.</u>	<u>Compound Name</u>
Anthracene		8	2,4'-dichlorobiphenyl
Benz(a)anthracene		18	2,2',5-trichlorobiphenyl
Benzo(a)pyrene		28	2,4,4'-trichlorobiphenyl
Biphenyl		44	2,2',3,5'-tetrachlorobiphenyl
Chrysene		52	2,2',5,5'-tetrachlorobiphenyl
Dibenz(a,h)anthracene		66	2,3',4,4'-tetrachlorobiphenyl
Dibenzothiophene		101	2,2',4,5,5'-pentachlorobiphenyl
2,6-dimethylnaphthalene		105	2,3,3',4,4'-pentachlorobiphenyl
Fluoranthene		110/77	2,3,3',4',6-pentachlorobiphenyl
Fluorene			3,3',4,4'-tetrachlorobiphenyl
2-methylnaphthalene		118	2,3',4,4',5-pentachlorobiphenyl
1-methylnaphthalene		126	3,3',4,4',5-pentachlorobiphenyl
1-methylphenanthrene		128	2,2',3,3',4,4'-hexachlorobiphenyl
2,6-dimethylnaphthalene		138	2,2',3,4,4',5'-hexachlorobiphenyl
Naphthalene		153	2,2',4,4',5,5'-hexachlorobiphenyl
Pyrene		170	2,2',3,3',4,4',5-heptachlorobiphenyl
Benzo(b)fluoranthene		180	2,2',3,4,4',5,5'-heptachlorobiphenyl
Acenaphthylene		187	2,2',3,4',5,5',6-heptachlorobiphenyl
Benzo(k)fluoranthene		195	2,2',3,3',4,4',5,6-octachlorobiphenyl
Benzo(g,h,i)perylene		206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl
Indeno(1,2,3-c,d)pyrene		209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl
2,3,5-trimethylnaphthalene			
<b>DDT and its metabolites</b>		<b>Chlorinated pesticides other than DDT</b>	
2,4'-DDD		Aldrin	
4,4'-DDD		Alpha-Chlordane	
2,4'-DDE		Dieldrin	
4,4'-DDE		Endosulfan I	
2,4'-DDT		Endosulfan sulfate	
4,4'-DDT		Endrin	
		Heptachlor	
		Heptachlor epoxide	
		Hexachlorobenzene	
		Lindane (gamma-BHC)	
		Mirex	
		Toxaphene	
		Trans-Nonachlor	
		<b>Trace Elements</b>	
		Aluminum	
		Antimony (sediment, only)	
		Arsenic	
		Cadmium	
		Chromium	
		Copper	
		Iron	
		Lead	
		Manganese (sediment, only)	
		Mercury	
		Nickel	
		Selenium	
		Silver	
		Tin	
		Zinc	
		<b>Other Measurements</b>	
		Total organic carbon	

(REFER TO TABLE B5-2, PAGE 57 OF THIS DOCUMENT)

**TABLE 5.2** Chemicals to be measured in fish and shellfish tissue by EMAP-Estuaries Louisianian Province.**DDT and its metabolites**

2,4'-DDD  
 4,4'-DDD  
 2,4'-DDE  
 4,4'-DDE  
 2,4'-DDT  
 4,4'-DDT

**Trace Elements**

Aluminum  
 Arsenic Cadmium  
 Chromium  
 Copper  
 Iron  
 Lead  
 Mercury  
 Nickel  
 Selenium  
 Silver  
 Tin  
 Zinc

**Chlorinated pesticides other than DDT**

Aldrin  
 Alpha-Chlordane  
 Dieldrin  
 Endosulfan  
 Endrin  
 Heptachlor  
 Heptachlor epoxide  
 Hexachlorobenzene  
 Lindane (gamma-BHC)  
 Mirex  
 Toxaphene  
 Trans-Nonachlor

**(REFER TO TABLE B5-2, PAGE 57 OF THIS DOCUMENT)****21 PCB Congeners:****PCB**

No.	Compound Name
8	2,4'-dichlorobiphenyl
18	2,2'5-trichlorobiphenyl
28	2,4,4'-trichlorobiphenyl
44	2,2',3,5'-tetrachlorobiphenyl
52	2,2',5,5'-tetrachlorobiphenyl
66	2,3',4,4'-tetrachlorobiphenyl
101	2,2',4,5,5'-pentachlorobiphenyl
105	2,3,3',4,4'-pentachlorobiphenyl
110/77	2,2',4,5,5'-pentachlorobiphenyl 3,3',4,4'-tetrachlorobiphenyl
118	2,3',4,4',5-pentachlorobiphenyl
126	3,3',4,4',5-pentachlorobiphenyl
128	2,2',3,3',4,4'-hexachlorobiphenyl
138	2,2',3,4,4',5'-hexachlorobiphenyl
153	2,2',4,4',5,5'-hexachlorobiphenyl
170	2,2',3,3',4,4',5-heptachlorobiphenyl
180	2,2'3,4,4',5,5'-heptachlorobiphenyl
187	2,2',3,4',5,5',6-heptachlorobiphenyl
195	2,2',3,3',4,4',5,6-octachlorobiphenyl
206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl
209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl

**TABLE 5-3.** Summary of EMAP-E chemistry sample collection, preservation, and holding time requirements. (EPA criteria recommends maximum sample holding times of 2-4 weeks at 4°C for most of the parameters listed here. Currently, in the Louisianian Province, logistical constraints prevent sample turn around in the 2- 4 week recommended period. Therefore, unless stated otherwise, chemistry samples are held frozen for up to 1 year.)

Parameter	Container	Volume	Sample Size	Sample Preservation	Max. Sample Holding Time	Max. Extract Holding Time
Sediment Metals	125-ml HDPE wide-mouth bottle	100 to 150 ml	75 to 100 g (approx.)	Freeze (-18°)	1 Year	<sup>a</sup>
Sediment TOC	Glass jar	same as above	30-50 ml (approx.)	Cool, 4°	6 months	<sup>b</sup>
Sediment Organics (including butyltins)	500-ml-pre-cleaned glass	250 to 300 ml	300 g (approx.)	Freeze (-18°)	1 year	40 days
Sediment Acid Volatile Sulfide (AVS)	125-ml. poly-propylene wide-mouth bottle	125 ml <sup>b</sup>	100 g <sup>b</sup> (approx.)	Freeze (-18°)	6 months	36 hours
Fish Tissue (Organic and In-organics)	Whole fish individually wrapped in Al. foil, then placed in water-tight plastic bags	NA	NA	Freeze (-18°)	1 year	40 days
<sup>a</sup>	No EPA criteria exists. Every effort should be made to analyze sample as soon as possible following extraction, or in the case of metals, digestion.					
<sup>b</sup>	AVS containers should be filled near the top to minimize the head space; however, there should be small head space to allow for sample expansion during freezing; containers should be capped tightly and then frozen. Every effort should be made to minimize contact of the sediment with air to analyze these samples as soon as possible.					

## 5.3 QUALITY CONTROL PROCEDURES: LABORATORY OPERATIONS

### 5.3.1 Overview

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

The performance-based EMAP-E QA program for analytical chemistry laboratories consists of two basic elements: 1.) initial demonstration of laboratory capability (e.g., performance evaluation) and 2.) ongoing demonstration of capability. Prior to the analysis of samples, each laboratory must demonstrate proficiency in several ways: written protocols for the analytical methods to be employed for sample analysis must be submitted to the Program for review, method detection limits for each analyte must be calculated, an initial calibration curve must be established for all analytes, and acceptable performance must be shown on a known or blind accuracy-based material. Following a successful first phase, the laboratory must demonstrate its continued capabilities in several ways: participation in an on-going series of laboratory intercomparison exercises, repeated analysis of Certified Reference Materials, calibration checks, and analysis of laboratory reagent blanks and fortified samples. These steps are detailed in the following sections and summarized in Table 5-4. The sections are arranged to mirror the elements in Table 5-4 to provide easy cross-reference for the reader.

The results for the various QA/QC samples should be reviewed by laboratory personnel immediately following the analysis of each sample batch. These results then should be used to determine when warning and control limit criteria have not been met and corrective actions must be taken, before processing a subsequent sample batch. When warning limit criteria have not been met, the laboratory is not obligated to halt analyses, but the analyst(s) is advised to investigate the cause of the exceedance. When control limit criteria are not met, specific corrective actions are required before the analyses may proceed. Warning and control limit criteria and recommended frequency of analysis for each QA/QC element or sample type required in the EMAP-E program also are summarized in Table 5-4.



**TABLE 5-4.** Key elements of laboratory quality control for EMAP-Estuaries chemical analyses (see text for detailed explanations).

Element or Sample Type	Warning Limit Criteria	Control Limit Criteria	Frequency
1.) Initial Demonstration of Capability (Prior to Analysis of Samples: prior to analyzing			
- Instrument Calibration	NA	NA	Initial and then prior to analyzing each batch of samples
- Calculation of Method Detection Limits	Must be equal to or less than target values (see Table 5-5)		At least once each year
- Blind Analysis of Accuracy-Based Material	NA	NA	Initial
2.) On-going Demonstration of Capability:			
- Blind Analysis of Laboratory Inter-comparison Exercise Samples	NA	NA	Regular intervals throughout the year
- Continuing Calibration Checks using Calibration Standard Solutions	NA	should be within $\pm 15\%$ of initial calibration on average for all analytes, not to exceed $\pm 25\%$ for any one analyte	At a minimum, middle middle and end of each sample batch
- Analysis of Certified Reference Material (CRM) or Laboratory Control Material (LCM):			One with each batch of samples
Precision (see NOTE 1):	NA	Value obtained for each analyte should be within 3s control chart limits	Value plotted on control chart after each analysis of the CRM

TABLE 5-4. (Continued)

Element or Sample Type	Warning Limit Criteria	Control Limit Criteria	Frequency
Relative Accuracy (see NOTE 2):			
PAHs	Lab's value should be within $\pm 25\%$ of true value on average for all analytes; not to exceed $\pm 30\%$ of true value for more than 30% of individual analytes	Lab's value should be within $\pm 30\%$ of true value on average for all analytes; not to exceed $\pm 35\%$ of true value for more than 30% of individual analytes	
PCBs/pesticides	same as above	same as above	
Inorganic elements	Lab should be within $\pm 15\%$ of true value for each analyte	Lab should be within $\pm 20\%$ of true value for each analyte	
<p><b>NOTE 1:</b> The use of control charts to monitor precision for each analyte of interest should follow generally accepted practices (e.g., Taylor 1987 and section 3.2.5 of this document). Upper and lower control limits, based on 99% confidence intervals around the mean, should be updated at regular intervals.</p> <p><b>NOTE 2:</b> "True" values in CRMs may be either "certified" or "non-certified" (it is recognized that absolute accuracy can only be assessed using certified values, hence the term relative accuracy). Relative accuracy is computed by comparing the laboratory's value for each analyte against either end of the range of values (i.e., 95% confidence limits) reported by the certifying agency. The laboratory's value must be within <math>\pm 35\%</math> of either the upper or lower 95% confidence interval value. Accuracy control limit criteria only apply for analytes having CRM concentrations <math>\geq 10</math> times the laboratory's MDL.</p>			
- Laboratory Reagent Blank	Analysts should use best professional judgement if analytes are detected at $< 3$ times the MDL	No analyte should be detected at $> 3$ times the MDL	One with each batch of samples
- Laboratory Fortified Sample Matrix (Matrix Spike)	NA	Recovery should be within the range 50%-120% for at least 80% of the analytes	At least 5% of total number of samples

TABLE 5-4. (Continued)

Element or Sample Type	Warning Limit Criteria	Control Limit Criteria	Frequency
<p><b>NOTE:</b> Samples to be spiked should be chosen at random; matrix spike solutions should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be at least 10 times the calculated MDL.</p>			
- Laboratory Fortified Sample Matrix Duplicate (Matrix Spike Duplicate)	NA	RPD <sup>1</sup> must be ≤ 30 for each analyte	Same as matrix spike
- Field Duplicates (Field Splits)	NA	NA	5% of total number of samples
- Internal Standards (Surrogates)	NA	Recovery must be within the range 30% to 150%	Each sample
- Injection Internal Standards	Lab develops its own	Lab develops its own	Each sample

<sup>1</sup> RPD = Relative percent difference between matrix spike and matrix spike duplicate results (see appropriate section for equation).

### 5.3.2 Initial Demonstration of Capability

#### Instrument Calibration

Equipment should be calibrated prior to the analysis of each sample batch, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended control limit criteria (Table 5-4). All calibration standards should be traceable to a recognized organization for the preparation and certification of QA/QC materials (e.g., National Institute of Standards and Technology, U.S. Environmental Protection Agency, etc.). Calibration curves must be established for each element and batch analysis from a calibration blank and a minimum of three analytical standards of increasing concentration, covering the range of expected sample concentrations. The calibration curve should be well-characterized and must be established prior to the analysis of samples. Only data which results from quantification within the demonstrated working calibration range may be reported by the laboratory (i.e., quantification based on extrapolation is not acceptable). Samples outside the calibration range should be diluted or concentrated, as appropriate, and reanalyzed.

#### Initial Documentation of Method Detection Limits

Analytical chemists have coined a variety of terms to define “limits” of detectability; definitions for some of the more commonly-used terms are provided in Keith *et al.* (1983) and in Keith (1991). In the EMAP-E program, the Method Detection Limit (MDL) will be used to define the analytical limit of detectability. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition: “the MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.” Confidence in the apparent analyte concentration increases as the analyte signal increases above the MDL.

Each EMAP-E analytical laboratory must calculate and report an MDL for each analyte of interest in each matrix of interest (sediment or tissue) prior to the analysis of field samples for a given year. Each laboratory is required to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate MDLs for each analytical method employed. The matrix and the amount of sample (i.e., dry weight of sediment or tissue) used in calculating the MDL should match as closely as possible the matrix of the actual field samples and the amount of sample typically used. In order to ensure comparability of results among different laboratories, MDL target values have been established for the EMAP-E program (Table 5-5). The initial MDLs reported by each laboratory should be equal to or less than these specified target values before the analysis of field samples may proceed. Each laboratory must periodically (i.e., at least once each year) re-evaluate its MDLs for the analytical methods used and the sample matrices typically encountered.

**TABLE 5-5.** Target method detection limits for EMAP-Estuaries analytes.  
**(REFER TO TABLE A7-2, PAGE 22 OF THIS DOCUMENT)**

INORGANICS (NOTE: concentrations in µg/g (ppm), dry weight)

	Tissue	Sediments
Aluminum	10.0	1500
Antimony	not measured	0.2
Arsenic	2.0	1.5
Cadmium	0.2	0.05
Chromium	0.1	5.0
Copper	5.0	5.0
Iron	50.0	500
Lead	0.1	1.0
Manganese	not measured	1.0
Mercury	0.01	0.01
Nickel	0.5	1.0
Selenium	1.0	0.1
Silver	0.01	0.01
Tin	0.05	0.1
Zinc	50.0	2.0

ORGANICS (NOTE: concentrations in ng/g (ppb), dry weight)

	Tissue	Sediments
PAHs	20.0	10
PCB congeners	2.0	1.0
Chlorinated pesticides	2.0	1.0

Initial Blind Analysis of a Representative Sample

A representative sample matrix which is uncompromised, homogeneous and contains the analytes of interest at concentrations of interest will be provided to each analytical laboratory new to the EMAP-E program; this sample will be used to evaluate laboratory performance prior to the analysis of field samples. The sample used for this initial demonstration of laboratory capability typically will be distributed blind (i.e., the laboratory will not know the concentrations of the analytes of interest) as part of the laboratory QA intercomparison exercises. A laboratory's performance generally will be considered acceptable if its submitted values are within  $\pm 30\%$  (for organic analyses) and  $\pm 20\%$  (for inorganic analyses) of the known concentration of each analyte of interest in the sample. These criteria apply only for analyte concentrations equal to or greater than 10 times the MDL established by the laboratory. If the results for the initial analysis fail to meet these criteria, the laboratory will be required to repeat the analysis until the performance criteria are met, prior to the analysis of real samples.

### 5.3.3 On-going Demonstration of Capability

#### Laboratory Participation in Intercomparison Exercises

Through an interagency agreement, NOAA's NS&T Program and EPA's EMAP-E program jointly sponsor an on-going series of laboratory intercomparison exercises (round-robins). All EMAP-E analytical laboratories are required to participate in these exercises, which are conducted jointly by the National Institute of Standards and Technology (NIST) and the National Research Council of Canada (NRCC). These exercises provide a tool for continuous improvement of laboratory measurements by helping analysts identify and resolve problems in methodology and/or QA/QC. The results of these exercises also are used to evaluate both the individual and collective performance of the participating analytical laboratories on a continuous basis. The EMAP-E laboratories are required to initiate corrective actions if their performance in these intercomparison exercises falls below certain pre-determined minimal standards, described in later sections.

Typically, three or four different exercises are conducted over the course of a year. In a typical exercise, either NIST or NRCC will distribute performance evaluation samples in common to each laboratory, along with detailed instructions for analysis. A variety of performance evaluation samples have been utilized in the past, including accuracy-based solutions, sample extracts, and representative matrices (e.g., sediment or tissue samples). Laboratories are required to analyze the sample(s) "blind" and must submit their results in a timely manner both to the EMAP-E QA Coordinator, as well as to either NIST or NRCC (as instructed). Laboratories which fail to maintain acceptable performance may be required to provide an explanation and/or undertake appropriate corrective actions. At the end of each calendar year, coordinating personnel at NIST and NRCC hold a QA workshop to present and discuss the intercomparison exercise results. Representatives from each laboratory are expected to participate in the annual QA workshops, which provide a forum for discussion of analytical problems brought to light in the intercomparison exercises.

#### Routine Analysis of Certified Reference Materials or Laboratory Control Materials

Certified Reference Materials (CRMs) generally are considered the most useful QC samples for assessing the accuracy of a given analysis (i.e., the closeness of a measurement to the "true" value). Certified Reference Materials can be used to assess accuracy because they have "certified" concentrations of the analytes of interest, as determined through replicate analyses by a reputable certifying agency using two independent measurement techniques for verification. In addition, the certifying agency may provide "non-certified" or "informational" values for other analytes of interest. Such values are determined using a single measurement technique, which may introduce unrecognized bias. Therefore, non-certified values must be used with caution in evaluating the performance of a laboratory using a method which differs from the one used by the certifying agency. A list of reference materials commonly used by EMAP-E laboratories is presented in Table 5-6.

A Laboratory Control Material (LCM) is similar to a Certified Reference Material in that it is a homogeneous matrix which closely matches the samples being analyzed. A "true" LCM is one which is prepared (i.e., collected, homogenized and stored in a stable condition) strictly for use in-house by a single laboratory. Alternately, the material may be prepared by a central laboratory and distributed to others (so-called regional or program control materials). Unlike CRMs,



concentrations of the analytes of interest in LCMs are not certified but are based upon a statistically valid number of replicate analyses by one or several laboratories. In practice, this material can be used to assess the precision (i.e., consistency) of a single laboratory, as well as to determine the degree of comparability among different laboratories. If available, LCMs may be preferred for routine (i.e., day to day) analysis because CRMs are relatively expensive. However, CRMs still must be analyzed at regular intervals (e.g., monthly or quarterly) to provide a check on accuracy.

**Table 5-6.** Certified Reference Materials commonly used by EMAP-E laboratories. SRMs are available from NIST (phone 301-975-6776); all other reference materials listed are available from NRC (phone 613-993-2359).

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Calibration Solutions:

SRM 1491	Aromatic Hydrocarbons in Hexane/Toluene
SRM 1492	Chlorinated Pesticides in Hexane
SRM 1493	Chlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane
SRM 2260	Aromatic Hydrocarbons in Toluene
SRM 2261	Chlorinated Pesticides in Hexane
SRM 2262	Chlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane

Environmental Matrices (Organics):

SRM 1941a	Organics in Marine Sediment
SRM 1974	Organics in Mussel Tissue ( <i>Mytilus edulis</i> )

Environmental Matrices (Inorganics):

SRM 1646	Estuarine Sediment	BCSS-1	Marine Sediment
MESS-1	Estuarine Sediment	PACS-1	Harbor Sediment
BEST-1	Marine Sediment	DORM-1	Dogfish Muscle
DOLT-1	Dogfish Liver	SRM 1566a	Oyster Tissue

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Routine analysis of Certified Reference Materials or, when available, Laboratory Control Materials represents a particularly vital aspect of the “performance-based” EMAP-E QA philosophy. At least one CRM or LCM must be analyzed along with each batch of 25 or fewer samples (Table 5-4). For CRMs, both the certified and non-certified concentrations of the target analytes should be known to the analyst(s) and should be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs or LCMs (Table 5-4); these criteria are discussed in detail in the following paragraphs. If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM or LCM, the data for the entire batch of samples is suspect. Calculations and instruments should be checked; the CRM or LCM may have to be reanalyzed (*i.e.*, reinjected) to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample processing. The results of the CRM or LCM analysis should never be used by the laboratory to “correct” the data for a given sample batch.

Precision criteria: Each laboratory is expected to maintain control charts for use by analysts in monitoring the overall precision of the CRM or LCM analyses. Upper and lower control chart limits (*e.g.*, warning limits and control limits) should be updated at regular intervals; control limits based on 3 standard deviations of the mean generally are recommended (Taylor 1987). Following the analysis of all samples in a given year, an RSD (relative standard deviation, a.k.a. coefficient of variation) will be calculated for each analyte of interest in the CRM. For each analyte having a CRM concentration  $\geq 10$  times the laboratory’s MDL, an overall RSD of less than 30% will be considered acceptable precision. Failure to meet this goal will result in a thorough review of the laboratory’s control charting procedures and analytical methodology to determine if improvements in precision are possible.

Accuracy criteria: The “absolute” accuracy of an analytical method can be assessed using CRMs only when certified values are provided for the analytes of interest. However, the concentrations of many analytes of interest to EMAP-E are provided only as non-certified values in some of the more commonly-used CRMs. Therefore, control limit criteria are based on “relative accuracy”, which is evaluated for each analysis of the CRM or LCM by comparison of a given laboratory’s values relative to the “true” or “accepted” values in the LCM or CRM. In the case of CRMs, this includes both certified and noncertified values and encompasses the 95% confidence interval for each value as described in Table 5-4.

Accuracy control limit criteria have been established both for individual compounds and combined groups of compounds (Table 5-4). There are two combined groups of compounds for the purpose of evaluating relative accuracy for organic analyses: PAHs and PCBs/pesticides. The laboratory’s value should be within  $\pm 30\%$  of the true value on average for each combined group of organic compounds, and the laboratory’s value should be within  $\pm 35\%$  of either the upper or lower 95% confidence limit for at least 70% of the individual compounds in each group. For inorganic analyses, the laboratory’s value should be within  $\pm 20\%$  of either the upper or lower 95% confidence limit for each analyte of interest in the CRM. Due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy only apply to analytes having CRM true values which are  $\geq 10$  times the MDL established by the laboratory.

### Continuing Calibration Checks

The initial instrument calibration performed prior to the analysis of each batch of samples is checked through the analysis of calibration check samples (i.e., calibration standard solutions) inserted as part of the sample stream. Calibration standard solutions used for the continuing calibration checks should contain all the analytes of interest. At a minimum, analysis of the calibration check solution should occur somewhere in the middle and at the end of each sample batch. Analysts should use best professional judgement to determine if more frequent calibration checks are necessary or desirable.

If the control limit for analysis of the calibration check standard is not met (Table 5-4), the initial calibration will have to be repeated. If possible, the samples analyzed before the calibration check sample that failed the control limit criteria should be reanalyzed following the recalibration. The laboratory should begin by reanalyzing the last sample analyzed before the calibration standard which failed. If the relative percent difference (RPD) between the results of this reanalysis and the original analysis exceeds 30 percent, the instrument is assumed to have been out of control during the original analysis. If possible, reanalysis of samples should progress in reverse order until it is determined that there is less than 30 RPD between initial and reanalysis results. Only the re-analysis results should be reported by the laboratory. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control check) is suspect. In this case, the laboratory should prepare a narrative explanation to accompany the submitted data.

### Laboratory Reagent Blank

Laboratory reagent blanks (also called method blanks or procedural blanks) are used to assess laboratory contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one laboratory reagent blank should be run in every sample batch. The reagent blank should be processed through the entire analytical procedure in a manner identical to the samples. Warning and control limits for blanks (Table 5-4) are based on the laboratory's method detection limits as documented prior to the analysis of samples. A reagent blank concentration between the MDL and 3 times the MDL for one or more of the analytes of interest should serve as a warning limit requiring further investigation based on the best professional judgement of the analyst(s). A reagent blank concentration equal to or greater than 3 times the MDL for one or more of the analytes of interest requires definitive corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis.

### Internal Standards

Internal standards (commonly referred to as "surrogates", "surrogate spikes" or "surrogate compounds") are compounds chosen to simulate the analytes of interest in organic analyses. The internal standard represents a reference analyte against which the signal from the analytes of interest is compared directly for the purpose of quantification. Internal standards must be added to each sample, including QA/QC samples, prior to extraction. The reported concentration of each analyte should be adjusted to correct for the recovery of the internal standard, as is done in the NOAA National Status and Trends Program. The internal standard recovery data therefore should be carefully monitored; each laboratory must report the percent recovery of the internal standard(s) along with the target analyte data for each sample. If possible, isotopically-labeled analogs of the analytes should be used as internal standards.

Control limit criteria for internal standard recoveries are provided in Table 5-4. Each laboratory should set its own warning limit criteria based on the experience and best professional judgement of the analyst(s). It is the responsibility of the analyst(s) to demonstrate that the analytical process is always “in control” (i.e., highly variable internal standard recoveries are not acceptable for repeat analyses of the same certified reference material and for the matrix spike/matrix spike duplicate).

#### Injection Internal Standards

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

#### Matrix Spike and Matrix Spike Duplicate

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

Recovery data for the fortified compounds ultimately will provide a basis for determining the prevalence of matrix effects in the sediment samples analyzed during the project. If the percent recovery for any analyte in the MS or MSD is less than the recommended warning limit of 50 percent, the chromatograms and raw data quantitation reports should be reviewed. If an explanation for a low percent recovery value is not discovered, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interferences and further instrument response checks may not be warranted, especially if the low recovery occurs in both the MS and MSD and the other QC samples in the batch indicate that the analysis was “in control”. An explanation for low percent recovery values for MS/MSD results should be discussed in a cover letter accompanying the data package. Corrective actions taken and verification of acceptable

instrument response must be included.

Analysis of the MS/MSD also is useful for assessing laboratory precision. The relative percent difference (RPD) between the MS and MSD results should be less than 30 for each analyte of interest (see Table 5-4). The RPD is calculated as follows:

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

where: C1 is the larger of the duplicate results for a given analyte  
C2 is the smaller of the duplicate results for a given analyte

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

#### Field Duplicates and Field Splits

For the EMAP-E program, sediment will be collected at each station using a grab sampler. Each time the sampler is retrieved, the top 2 cm of sediment will be scraped off, placed in a large mixing container and homogenized, until a sufficient amount of material has been obtained. At approximately 5% of the stations, the homogenized material will be placed in four separate sample containers for subsequent chemical analysis. Two of the sample containers will be submitted as blind field duplicates to the primary analytical laboratory. The other two containers, also called field duplicates, will be sent blind to a second laboratory. Together, the two pairs of duplicates are called field splits. The analysis of the field duplicates will provide an assessment of single laboratory precision. The analysis of the field duplicates and field splits will provide an assessment of both inter- and intra-laboratory precision, as well as an assessment of the efficacy of the field homogenization technique.

### **5.4 OTHER SEDIMENT MEASUREMENTS**

The preceding sections presented QA/QC requirements covering laboratory analysis of sediment and fish tissue samples for organics (i.e., PAHs, PCBs and chlorinated pesticides) and inorganics (i.e., metals). In addition to these “conventional” contaminants, EMAP-E laboratories are required to measure several ancillary sediment parameters, such as total organic carbon (TOC), acid volatile sulfide (AVS), and tri-, di- and monobutyltin (TBT, DBT, MBT) concentrations. The laboratory QA/QC requirements associated with these “other sediment measurements” are presented in the following sections.

#### **5.4.1 Total Organic Carbon**

As a check on precision, each laboratory should analyze at least one total organic carbon (TOC) sample in duplicate for each batch of 25 or fewer samples. The relative percent difference



(RPD) between the two duplicate measurements should be less than 20%. If this control limit is exceeded, analysis of subsequent sample batches should stop until the source of the discrepancy is determined and the system corrected.

At least one certified reference material (CRM) or, if available, one laboratory control material (LCM) should be analyzed along with each batch of 25 or fewer TOC samples. Any one of several marine sediment CRMs distributed by the National Research Council of Canada's Marine Analytical Chemistry Standards Program (e.g., the CRMs named "BCSS-1", "MESS-1" and "PACS-1", see Table 5-6) have certified concentrations of total carbon and are recommended for this use. Prior to analysis of actual samples, it is recommended that each laboratory perform several total organic carbon analyses using a laboratory control material or one of the aforementioned CRMs to establish a control chart (the values obtained by the laboratory for total organic carbon should be slightly less than the certified value for total carbon in the CRM). The control chart then should be used to assess the laboratory's precision for subsequent analyses of the LCM or CRM with each sample batch. In addition, a method blank should be analyzed with each sample batch. Total organic carbon concentrations should be reported as g/g (ppm) dry weight of the unacidified sediment sample. Data reported for each sample batch should include QA/QC sample results (duplicates, CRMs or LCMs, and method blanks). Any factors that may have influenced data quality should be discussed in a cover letter accompanying the submitted data.

#### 5.4.2 Acid Volatile Sulfide

Quality control of acid volatile sulfide (AVS) measurements is achieved through the routine analysis of a variety of QA/QC samples. These are outlined in the following section and described in full detail in the EMAP-E Laboratory Methods Manual (U.S. EPA, in preparation). Prior to the analysis of samples, the laboratory must establish a calibration curve and determine a limit of reliable detection for sulfide for the analytical method being employed. Following this, laboratory performance will be assessed through routine analysis of laboratory duplicates, calibration check standards, laboratory fortified blanks (i.e., spiked blanks), and laboratory fortified sample matrices (i.e., matrix spikes).

One sample in every batch of 25 or fewer samples should be analyzed in duplicate as a check on laboratory precision. The relative percent difference (RPD) between the two analyses should be less than 20%. If the RPD exceeds 20%, a third analysis should be performed. If the relative standard deviation of the three determined concentrations exceeds 20%, the individual analyses should be examined to determine if non-random errors may have occurred. As previously discussed, field duplicates and splits also will be collected for AVS determination to assess both inter- and intra-laboratory precision.

Due to the instability of acid volatile sulfides to drying and handling in air, CRMs have not been developed for assessing overall measurement accuracy. Therefore, each laboratory must analyze at least one calibration check standard, one laboratory fortified blank and one laboratory fortified sample matrix in each batch of 25 or fewer samples as a way of determining the accuracy of each step entailed in performing the analysis. The concentration of sulfide in each of these three types of accuracy check samples will be known to the analyst; the calculated concentration of sulfide in each sample should be within  $\pm 15\%$  of the known concentration.

If the laboratory is not within  $\pm 15\%$  of the known concentration for the calibration check

solution, instruments used for AVS measurement must be recalibrated and/or the stock solutions redetermined by titration. If the laboratory fails to achieve the same accuracy (within  $\pm 15\%$  of the true value) for AVS in the laboratory fortified blank, sources of error (e.g., leaks, excessive gas flows, poor sample-acid slurry agitation) should be determined for the analytical system prior to continuing. If AVS recovery falls outside the 85% to 115% range for the matrix spike, the system should be evaluated for sources of error and the analysis should be repeated. If recovery remains unacceptable, it is possible that matrix interferences are occurring. If possible, the analysis should be repeated using smaller amounts of sample to reduce the interferant effects. Results for all QA/QC samples (duplicates, calibration check standards, spiked blanks and matrix spikes) should be submitted by the laboratory as part of the data package for each batch of samples, along with a narrative explanation for results outside control limits.

#### 5.4.3 Butyltins

Assessment of the distribution and environmental impact of butyltin species of interest to the EMAP-E program (tributyltin, dibutyltin and monobutyltin) requires their measurement in marine sediment and tissue samples at trace levels. Quality control of these measurements consists of checks on laboratory precision and accuracy. One laboratory reagent blank must be run with each batch of 25 or fewer samples. A reagent blank concentration between the MDL and 3 times the MDL should serve as a warning limit requiring further investigation based on the best judgement of the analyst(s). A reagent blank concentration equal to or greater than 3 times the MDL requires corrective action to identify and eliminate the source(s) of contamination, followed by reanalysis of the samples in the associated batch.

One laboratory fortified sample matrix (commonly called a matrix spike) or laboratory fortified blank (i.e., spiked blank) should be analyzed along with each batch of 25 or fewer samples to evaluate the recovery of the butyltin species of interest. The butyltins should be added at 5 to 10 times their MDLs as previously calculated by the laboratory. If the percent recovery for any of the butyltins in the matrix spike or spiked blank is outside the range 70 to 130 percent, analysis of subsequent sample batches should stop until the source of the discrepancy is determined and the system corrected.

The NRCC sediment reference material "PACS-1", which has certified concentrations of the three butyltin species of interest, also should be analyzed along with each batch of 25 or fewer sediment samples as a check on accuracy and reproducibility (i.e., batch-to-batch precision). If values obtained by the laboratory for butyltins in "PACS-1" are not within  $\pm 30\%$  of the certified values, the data for the entire batch of samples is suspect. Calculations and instruments should be checked; the CRM may have to be reanalyzed to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to determine the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample processing.

## 5.5 QUALITY CONTROL PROCEDURES: INFORMATION MANAGEMENT

### 5.5.1 Sample Tracking

EMAP-E information management personnel have developed a comprehensive system for barcode labeling of sample containers, recording sampling information in the field and tracking sample shipments. A complete description of this system is provided in the EMAP-E Information Management Plan (Adams et al. 1993) and also summarized in Section 11 of this plan. Each analytical laboratory must designate a sample custodian, authorized to check the condition of and sign for incoming field samples, obtain documents of shipment and verify sample custody records. This individual is required, upon receipt of samples, to record and transmit all tracking information to the Province Information Management Center. The use of barcode labels and readers provided by the Province will facilitate this process. Laboratory personnel should be aware of the required sample holding times and conditions (see Table 5-3), and there must be clearly-defined custody procedures for sample handling, storage, and disbursement in the laboratory.

### 5.5.2 Data Reporting Requirements

As previously indicated, laboratory personnel must verify that the measurement process was “in control” (i.e., all specified QA/QC requirements were met) for each batch of samples before proceeding with the analysis of a subsequent batch. In addition, each laboratory must establish a system for detecting and eliminating transcription and/or calculation errors prior to reporting data. It is recommended that an individual not involved directly in sample processing be designated as laboratory QA Officer to perform these verification checks independent of day-to-day laboratory operations.

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

- A cover letter providing a brief description of the procedures and instrumentation used (including the procedure(s) used to calculate MDLs), as well as a narrative explanation of analytical problems (if any), departures from protocols, or failure(s) to meet required quality control limits.
- Tabulated results in hard copy form, including sample size, wet weight, dry weight, and concentrations of the analytes of interest (reported in units identified to three significant figures unless otherwise justified). Concentration units should be ng/g or g/g (dry weight) for sediment or tissue. The results should be checked for accuracy and the report signed by the laboratory manager or designee.
- Tabulated results in computer-readable form (e.g., diskette) included in the same shipment as the hard copy data, but packaged in a diskette mailer to prevent damage. Presently, there are three acceptable formats for computer-readable data, descriptions of which are available upon request from the Province Information Manager: 1.) the EPA Standard Format specified in EPA Order 2180.2 (“Data Standards for the Electronic Transmission of Laboratory Measurement Results”), 2.) ASCII text files in a format specified by the Province Information Manager, or 3.) any format agreed

upon by the submitting laboratory and the Province Information Manager. If data is not delivered in one of these formats, the data package will be considered incomplete and will not be accepted.

- Tabulated method detection limits achieved for the samples.
- Results for all QA/QC samples (e.g., CRMs, calibration check samples, blanks, matrix spike/matrix spike duplicates, etc.) must be submitted by the laboratory as part of the data package for each batch of samples analyzed. The laboratory must provide a “batch number” as a way to link samples from a given batch or analytical set with their accompanying QA/QC samples. The laboratory should denote QA/QC samples using the codes (abbreviations) and reporting units specified in Table 5-7. Laboratories are responsible for assigning only two data qualifier codes or “flags” to the submitted data. If an analyte is not detected, the laboratory should report the result either as “ND” or else leave the “RESULT” field empty, followed by the letter “a” in the “QACODE” field and the method detection limit (MDL) in the “MDL” field. The “a” code has the following meaning: “The analyte was not detected. The detection limit (MDL) is reported as a separate variable.” If a quantifiable signal is observed, the laboratory should report a concentration for the analyte; the data qualifier code “b” then should be used to flag any reported values which are below the laboratory’s MDL. The “b” code has the following meaning: “The reported concentration is below or equal to the detection limit. The detection limit (MDL) is reported as a separate variable.”

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Code	Description	Unit of Measure
CLC	Continuing Calibration Check Sample	Percent recovery
LRB	Lab Reagent Blank	varies
LCM	Lab Control Material	g/g or ng/g dry wt.
LCMPR	Lab Control Material % Recovery	Percent Recovery
LF1	Lab Spiked Sample- 1st Member	g/g or ng/g dry wt.
LF1PR	Lab Spiked Sample- 1st Mem.	% Rec. Percent Recovery
LF2	Lab Spiked Sample- 2nd Member	g/g or ng/g dry wt.
LF2PR	Lab Spiked Sample- 2nd Mem. % Rec.	Percent Recovery
MSDRPD	Rel % Difference: LF1 to LF2	Percent
LFB	Lab Fortified Blank	Percent Recovery
LSFPR	Lab Spiked Sample % Rec.	Percent Recovery
LDRPD	Lab Duplicate Relative %	Diff. Percent

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There may be a limited number of situations where sample re-analysis is not possible or practical (i.e., minor exceedance of a single control limit criteria). The laboratory is expected to provide a detailed explanation of any factors affecting data quality or interpretation; this explanation should be in the form of a cover letter accompanying each submitted data package. The narrative explanation is in lieu of additional data qualifier codes supplied by the laboratory (other than the “a” and “b” codes). Over time, depending on the nature of these narrative explanations, the EMAP-E program expects to develop a limited list of codes for qualifying data in the database (in addition to the “a” and “b” codes).

### 5.5.3 Data Evaluation Procedures

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

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

- 1.) Checking data completeness (verification)
- 2.) Assessing data quality (validation)
- 3.) Assigning data qualifier codes
- 4.) Taking final actions

The specific activities required to complete each of these steps are illustrated in Figure 5-1 and described in the following sections, which are adopted in large part from the document "A Project Manager's Guide to Requesting and Evaluating Chemical Analyses" (EPA 1991).

#### Checking Data Completeness

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

- Province personnel should verify that the package contains the following: narrative explanations signed by the laboratory manager, hard copies of all results (including QA/QC results), and accompanying computer diskettes.
- The electronic data file(s) should be parsed and entered into the EMAP Province database to verify that the correct format has been supplied.
- Once the data has been entered into the Province database, automated checks should be run to verify that results have been reported for all expected samples and all analytes.



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

### Assessing Data Quality

Data validation, or the process of assessing data quality, can begin after Province personnel have determined that the data package is complete. Normally, the first major part of validation involves checking 100-percent of the data for any possible errors resulting from transcription of tabulated results, misidentification or miscalculations. However, EMAP-E laboratories are expected to submit data which already has been tabulated and checked 100% for accuracy, and the raw data reports needed by Province personnel to perform these checks (e.g., chromatograms, original quantitation reports) are not submitted as part of the data package. In addition, a 100-percent validation check is both cost-prohibitive and unnecessary on monitoring programs, like EMAP-E, which do not involve enforcement actions. Therefore, the first-step validation checks performed by Province personnel will be limited to the following: 1.) a check to verify that all reporting units and numbers of significant figures are correct; 2.) a check to verify that all of the laboratory's calculated percent recovery values (for calibration check samples, Laboratory Control Materials, and matrix spikes) and relative percent difference values (for duplicates) are correct; and 3.) a check to verify that the reported concentrations for each analyte fall within "environmentally-realistic" ranges, determined from previous studies and expert judgement. In addition, past studies indicate that the different compounds in each class of chemicals being measured for EMAP-E (e.g., PAHs, PCBs, DDTs and other chlorinated pesticides) typically occur in the environment in somewhat fixed ratios to one another. For example, the DDT breakdown products p,p DDD and p,p DDE typically can be expected to occur at higher concentrations than p,p DDT in estuarine sediments of the Gulf Coast. If anomalous departures from such expected ratios are found, it may indicate a problem in the measurement or data reduction process requiring further investigation.

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

- 1.) Results for QA/QC samples should be tabulated, summarized and evaluated. Specifically, a set of summary tables should be prepared from the Province database showing the percent recovery values and relative percent difference values (where applicable) for the following QA/QC samples: continuing calibration checks samples, laboratory control material(s), and matrix spike/matrix spike duplicate samples. The tables should indicate the percent recovery values for these samples for each individual batch of samples, as well as the average, standard deviation, coefficient of variation, and range for all batches combined.
- 2.) Similar summary tables should be prepared for the laboratory reagent blank QA/QC samples.
- 3.) The summary results, particularly those for the Laboratory Control Material (i.e., Certified Reference Material), should be evaluated by comparing them against the QA/QC warning and



control limit criteria for accuracy, precision, and blank contamination specified in Table 5-4.

4.) Method detection limits reported by the laboratory for each analyte should be tabulated and compared against the target values in Table 5-5.

There are several possible courses of action to be taken if the reported data is found to be deficient (i.e., warning and/or control limits exceeded) during the assessment of data quality:

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

2.) If only warning limits were exceeded, then it is appropriate for the laboratory to report the results. Minor exceedances of a limited number of control limits should result in all associated data being qualified as estimated values, as explained in the following section. Large exceedances of several action limits should result in rejection of the data because there is ample evidence that the analyses were out of control and unreliable. However, because EMAP-E laboratories must report only data meeting QA/QC criteria for acceptability, this type of data rejection is not anticipated.

#### Assigning Data Qualifier Codes

Data qualifier codes are notations used by laboratories and data reviewers to briefly describe, or qualify, data and the systems producing data. As previously indicated, EMAP-E laboratories are expected to assign only two data qualifier codes ("a" and "b") to data values before submitting them to the program. EMAP-E data reviewers, in turn, will assign an additional data qualifier code in situations where there are minor exceedances of a limited number of control limit criteria. The most typical situation is when a laboratory fails to meet the accuracy control limit criteria for a particular analyte in a Certified Reference Material or matrix spike sample. In these situations, the QA reviewer should verify that the laboratory did meet the control limit criteria for precision. If the lack of accuracy is found to be consistent (i.e., control limit criteria for precision were met), then it is likely that the laboratory experienced a true bias for that particular analyte. In these situations, all reported values for that particular analyte will be qualified with a "c" code. The "c" code has the following meaning: "The reported concentration is considered an estimate because control limits for this analyte were exceeded in one or more quality control samples."

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

#### Taking Final Action

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

begins after the QA review has been completed.

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

- Summary of overall data quality, including a description of data that were qualified.
- Brief descriptions of analytical methods and the method(s) used to determine detection limits.
- Description of data reporting, including any corrections made for transcription or other reporting errors, and description of data completeness relative to objectives stated in the QA plan.
- Descriptions of initial and ongoing calibration results, blank contamination, and precision and bias relative to QA plan objectives (including tabulated summary results for Certified Reference Materials and matrix spike/matrix spike duplicates).

The chemistry QA results will be presented in the Program Annual Quality Assurance Report and will also become a permanent part of the database documentation (i.e., metadata). The QA/QC data collected by the Program will be used not only to assess the accuracy and precision of individual laboratory measurements, but ultimately to assess the comparability of data generated by multiple laboratories.

## **APPENDIX B**

### **Coastal 2000 Information Management Plan**

#### **Coastal 2000**

#### **Information Management**

**DRAFT - May 10, 2000**

**U.S. Environmental Protection Agency  
Office of Research and Development  
National Health and Ecological Effects Research Laboratory  
Atlantic Ecology Division  
27 Tarzwell Drive  
Narragansett RI 02882**

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## **I. Introduction**

This document describes how data and information from Coastal 2000 will be managed by the Environmental Monitoring and Assessment Program (EMAP). It provides guidance on data and metadata file formats and transmittal procedures for regional Coastal 2000 groups providing data to the EMAP National Coastal Database. All data sent to the national database will have passed the quality assurance and control procedures established by the Coastal 2000 Quality Assurance Project Plan (Heitmuller 2000). In general, regional Coastal 2000 groups will be supplying the same type of field and results data for this project, but there will be differences based on geographic location. It is important that all regional groups follow the same basic guidelines to ensure that the field and results data can be loaded into a uniform database in a timely manner. The national database, available over the World Wide Web, will provide a uniform, well-documented source of data and information that can be used for regional and national assessments. Further details on EMAP information management are given in the EMAP Information Management Plan (Hale et al. 1999) and on the EMAP Web site (EMAP 2000).

## **II. Data Policy Statements**

The fundamental objectives of Coastal 2000 are dependent upon the cooperation of researchers from many locations. Our objectives require quantitative analysis of interdisciplinary data sets and therefore participants must exchange data on a timely basis. Precedent and perception have resulted in a disparity of data collection, storage, and archival methods. This makes the exchange of data difficult and may suppress dissemination of data. Coastal 2000 seeks to enhance the value of data collected within the study by providing a set of guidelines for the collection, storage, exchange, and archival of these data sets. These statements are given in Appendix A.

The overall purpose of these policy statements is to facilitate full and open access and use with confidence, both now and in the future, of the data and information that is used in and results from Coastal 2000 activities. These policies reflect the goals and policies of EMAP and incorporate federal laws, directives, and regulations regarding the maintenance and dissemination of data and information in the Federal Government. They apply to all participants in Coastal 2000, including federal, state, local, tribal, foreign, educational, non-government organizations and their private partners, and will be incorporated into the provisions of any acquisition or assistance agreements funded by Coastal 2000.

## **III. Information Management Standards**

The core data management and GIS standards for Coastal 2000 are set forth in Appendix B. This is not a comprehensive list of all federal or EPA standards. It is a list of information management standards that all participants in Coastal 2000 agree to follow.

The goal of these core standards is to maximize the ability to exchange data within the study and with other studies conducted under the monitoring framework of the Committee on Natural Resources and Environment (CENR 1997). The main standards are those of the Federal Geographic Data Committee (FGDC 1998), the National Spatial Data Infrastructure (NSDI 2000), and the

National Biological Information Infrastructure (NBII 2000).

#### **IV. Data Flow**

Coastal 2000 data will be collected by state agencies. Four regional centers coordinate Coastal 2000 activities. These are: (1) West Coast - the Southern California Coastal Water Research Project in Westminster, CA; (2) Gulf of Mexico - EPA's Gulf Ecology Division in Gulf Breeze, FL; (3) Southeast - NOAA in Charleston, SC; and (4) Northeast - EPA's Atlantic Ecology Division in Narragansett, RI.

The Atlantic Ecology Division (AED) also has responsibility for the national Coastal 2000 information management. AED operates the EMAP National Coastal Database and the EMAP Web site (EMAP 2000).

Data will flow from the states to the four regional data centers to the national database. Additionally, data from field samples (for example, sediment chemistry) will go from state labs or a national contract lab to the regional data centers. The state data collectors ensure that the data meet the quality assurance standards. The regional data centers have the responsibility of ensuring consistency among the states in their region and merging field samples data from the analytical labs. The national data center will load the data to a consistent national database and transmit a copy to STORET (STORET 2000) for long-term archival.

Because of regional differences and existing data management procedures, each regional data center has the flexibility to manage information the way they determine is best for them. Each will have its own information management plan and procedures. Some regions will use field computer systems, others will not. The only requirement for regional data centers is to bring all the data together to the consistent format specified in this document. How they choose to do that is entirely up to them.

Regional data centers will have and manage certain data that are not required by the national data center. These include such things as QA results data from sediment chemistry and other raw data. Those users that ask for raw data not available on the national Web site will be directed to a contact at the regional data centers.

#### **V. Data Format and Transmittal Procedures**

Standard data tables (Appendix C) and code tables (Appendix D) will ensure consistency. These must be used by all regional Coastal 2000 groups when sending data to the national data center. EMAP IM is currently synchronizing species and chemical names and codes with STORET because STORET will be used as a long-term archive for Coastal 2000 data. Chemical Abstracts Service (CAS 2000) conventions are used for chemical names and codes. Scientific names and species codes follow the Integrated Taxonomic Information System (ITIS 2000), which is an interagency effort that updates the old NODC names and codes. Regional data centers should provide both the scientific name (spelling as given in ITIS) and the ITIS Taxonomic Serial Number. If the species does not exist in ITIS, provide the scientific name, author, citation, and full taxonomic hierarchy. Code tables will be placed on the EMAP Web site ([www.epa.gov/emap](http://www.epa.gov/emap)) for downloading.



Data must be provided in electronic media. Data may be sent from the regional data centers to the national data center as SAS data sets or delimited ASCII files (comma or semi-colon).

Documentation must be provided for all data sets defining the required data elements. Code table information should be provided as separate data sets. Electronic files can be sent by email attachment, FTP, or mailed on diskettes or ZIP disk.

It is important to keep AED informed of the completeness of the data sets. Files may be sent periodically, but AED should be informed that more data sets are pending.

Please direct questions regarding data and metadata transmittal to Melissa M. Hughes, OAO Corporation, at (401) 782-3184 or [hughes.melissa@epa.gov](mailto:hughes.melissa@epa.gov).

## VI. Metadata

Metadata provide documentation about sample collection, methods, processing, analysis, and quality assurance and quality control procedures applied to the samples and data. Metadata are necessary so that others can understand and use Coastal 2000 data. EMAP metadata consists of the EMAP Data Directory and the EMAP Data Catalog. Together, these provide the metadata content standard required by FGDC (2000). Both of these can be viewed on the EMAP Web site ([www.epa.gov/emap](http://www.epa.gov/emap)). Since the start of EMAP monitoring in 1990, EMAP has used the Data Catalog format shown in Appendix E.

Data sets cannot be included in the EMAP National Coastal Database without metadata. All data sets must be accompanied by metadata which follows the example given in Appendix E. However, if a regional data center wishes to use one of the metadata-writing software packages now available (such as USGS's MetaMaker), that format will also be acceptable.

## VII. Data Access

Preliminary data from states and analytical labs will be available to all partners as the data are received at the regional data centers. The EMAP National Coastal Database will be accessible to all on the EMAP Web site ([www.epa.gov/emap](http://www.epa.gov/emap)). Coastal 2000 data will also be copied to STORET ([www.epa.gov/OWOW/STORET](http://www.epa.gov/OWOW/STORET)).

## References

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- FGDC 1998 Content standard for digital geospatial metadata, version 2.0. FGDC-STD-001-1998.

- Federal Geographic Data Committee, Washington, DC [<http://www.fgdc.gov>].
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- STORET. 2000. The STORET web site. [<http://www.epa.gov/OWOW/STORET>].

## Appendix A. Coastal 2000 Data Policy Statements

The fundamental objectives of the Coastal 2000 are dependent upon the cooperation of scientists from several disciplines. Our objectives require quantitative analysis of interdisciplinary data sets and therefore participants must exchange data on a timely basis. Precedent and perception have resulted in a disparity of data collection, storage, and archival methods. This makes the exchange of data difficult and may suppress dissemination of data. Coastal 2000 seeks to enhance the value of data collected within the study by providing a set of guidelines for the collection, storage, exchange, and archival of these data sets.

The overall purpose of these policy statements is to facilitate full and open access and use with confidence, both now and in the future, of the data and information that is used in and results from Coastal 2000 activities. These policies reflect the goals and policies of EMAP and incorporate federal laws, directives, and regulations regarding the maintenance and dissemination of data and information in the Federal Government. They apply to all participants in Coastal 2000, including federal, state, local, tribal, foreign, educational, non-government organizations and their private partners, and will be incorporated into the provisions of any acquisition or assistance agreements funded by Coastal 2000.

- The Environmental Monitoring and Assessment Program requires a continuing commitment to the establishment, maintenance, description, accessibility, and long-term availability of high-quality data and information.
- Full and open sharing of the full suite of data and published information produced by the study is a fundamental objective. Data and information will be available without restriction for no more than the cost of reproduction and distribution. Where possible, the access to the data will be via the World Wide Web to keep the cost of delivery to a minimum and to allow distribution to be as wide as possible. All data collected by this study will be publicly available following verification and validation of the datasets.
- Organizations and individuals participating in the study should make measurements that do not involve manual analysis available to other study participants within 6 months after collection. All other measurements should be made available to study participants within 15 months after collection. Data and metadata should be publicly available on the EMAP web site within 24 months after field collection.

- All data sets and published information used in the study will be identified with a citation; for data sets an indication of how the data may be accessed will be provided.
- All data sets generated as part of the study will be made available on the EMAP public web site. These data sets must be described and a quality assessment provided. All such data set descriptions will be made available for inclusion in the EMAP Data Directory/Data Catalog, accessible on the EMAP web site. In addition, steps will be taken to assure their continuing availability.
- Participants will adhere to the 'Core Information Management Standards for Coastal 2000'. National and international standards will be used to the greatest extent possible.
- Citation information for all the study's published reports will be provided to the EMAP Bibliography, accessible on the EMAP web site.
- Organizations participating in the study are encouraged to contribute to the Coastal 2000 web site to share information.
- To the extent feasible, Coastal 2000 data will be copied to STORET for long-term archival and use.
- Suggested Data Product Requirement for Grants, Cooperative Agreements, and Contracts:  
*Describe the plan to make available the data products produced, whether from observations or analyses, that contribute significantly to the <grant's> results. The data products will be made available to the <grant official/contracting officer> without restriction and be accompanied by comprehensive metadata documentation adequate for specialists and non-specialists alike to be able to not only understand both how and where the data products were obtained but adequate for them to be used with confidence for generations. The data products and their metadata will be provided in a <standard> exchange format no later than the <grant's> final report or the publication of the data product's associated results, whichever comes first.*

**Acknowledgment:** This Data Policy Statement was modified, with permission, from two sources: Data Management for Global Change Research. Policy Statements for the National Assessment Program. July 1998. U.S. Global Change Research Program. National Science Foundation, Washington, DC.

U. S. GLOBEC. 1994. U. S. GLOBEC Data Policy. U. S. Global Ocean Ecosystems Dynamics. Report No. 10. Woods Hole, MA. (<http://globec.who.edu>).

## Appendix B. Coastal 2000 Core Information Management Standards

The core data management and GIS standards for Coastal 2000 are set forth below. This is not a comprehensive list of all federal or EPA standards, nor is it a list of data standards. It is a list of standards pertaining to information management that all participants in Coastal 2000 agree to follow.

Further details on EMAP standards are given in the EMAP Information Management Plan (Hale et al. 1999).

The goal of these core standards is to maximize the ability to exchange data within the study and with other studies conducted under the monitoring framework of the Committee on Natural Resources and Environment (CENR 1997). The main standards are those of the Federal Geographic Data Committee (FGDC 1999), the National Spatial Data Infrastructure (NSDI 1999), and the National Biological Information Infrastructure (NBII 1999).

### **Metadata**

- Federal Geographic Data Committee, Content standard for digital geospatial metadata, version 2.0. FGDC-STD-001-1998 (FGDC 1998), including the Biological Data Profile and the Biological Names and Taxonomy Data Standards developed by the National Biological Information Infrastructure (NBII 1999).
- For tabular data, metadata that meet the FGDC content standard are contained by a combination of the EMAP Data Directory and the EMAP Data Catalog. For Arc/Info coverages, the metadata are in the .DOC file embedded in the coverage. This file stays with the coverage. When the coverage is moved to the EMAP public web site, it will be duplicated to an ASCII text file.

### **EMAP Data Directory**

- EMAP Data: EMAP Data Directory Oracle database. The guidelines are given in Frithsen and Strebel (1995), Frithsen (1996a,b), and USEPA (1996b).
- Other data: Environmental Information Management System (EIMS 1999). EMAP Directory entries are periodically uploaded to the EIMS. The EIMS will become EPA's node for the National Spatial Data Infrastructure and will make directory information available to other federal agencies through the Z39.50 protocol in accordance with the US Global Change Research Program (USGCRP 1998).

### **EMAP Data Catalog**

- EMAP Data Catalog standards are given in Strebel and Frithsen (1995b), Frithsen (1996a), and USEPA (1996c).

### **Data Formats**

- **Attribute data**
  - ASCII files: comma-separated values, or space-delimited, or fixed column.
  - SAS export files
  - Oracle
- **GIS data**
  - ARC/INFO export files; compressed .tar file of ARC/INFO workspace
  - Spatial Data Transfer Standard (SDTS; FGDC 1999) format available on request

## Parameter Formats

- **Sampling Site** (EPA Locational Data Policy; EPA 1991)
  - Latitude and Longitude in decimal degrees ( +/- 7.4)
  - Negative longitude values (west of the prime meridian).
  - NAD83
- **Date:** YYYYMMDD (year,month,day).
- **Hour:** HHMMSS (hour,minute,second)
  - Greenwich mean time
  - Local time

Data loaded to STORET will take on the STORET formats upon loading.

## Standard Coding Systems

- **Chemical Compounds:** Chemical Abstracts Service (CAS 1999)
- **Species Codes:** Integrated Taxonomic Information System (ITIS 1999).
- **Land cover/land use codes:** Mutli-Resolution Land Characteristics (MRLC 1999)

## EMAP Web Site and Data Distribution

- EMAP-funded data, directory entry and catalog files must be made available to the EMAP public web site (EMAP 1999).
- Guidelines for making data available on this site are given in Strebel and Frithsen (1995a) and (USEPA 1997).
- Data and metadata files are posted to the internal EMAP web site for review by the contributor before moving to the EMAP public web site (EMAP 1999).
- Data on the internal web site may have not gone through full Quality Control/Quality Assurance (QA/QC), but data to be placed on the public web site must have undergone QA/QC according to an approved Quality Assurance Project Plan.
- No data sent to EMAP public web site without approval from data source.
- No data sets are distributed publicly without the accompanying metadata.
- Web site design must follow EPA standards [<http://www.epa.gov/epahome/webguide/guide.htm>].
- Very large files may be distributed by CD-ROM or DVD (free or for no more than the cost of reproduction)

## EMAP Bibliography

- EMAP Bibliography, an Oracle database (EMAP 1999).
- 'Guide to Submitting Information to the EMAP Bibliographic Database' (EMAP 1999).
- The citation format for the EMAP Bibliography is the Council of Biology Editors Manual (CBE 1994).



## Data Stewardship and Responsibility

- Data collectors are responsible for the preparation of data, directory, and catalog files.
- Data stewardship, maintenance and implementation of data Quality Control/Quality Assurance procedures lies primarily with the data collectors.

## Long-term Archival

- EMAP web data: EMAP Archival Plan (Hale et al. 1999; USEPA 1996a).
- Low-level data not transferred to EMAP web site: relevant EPA and Division archival policies.
- Water quality data will be archived to the extent feasible in STORET (STORET 1999).

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## Appendix C. Data Set Contents

This appendix provides data set and code file contents. These give attribute formats and descriptions. Groups are requested to provide all data sets and attributes within a data set that are relevant. Attributes listed in **bold** are mandatory fields. Code table information should be provided as separate data sets.

For questions, contact Melissa M. Hughes, OAO Corp. at (401) 782-3184 or by E-mail at [hughes.melissa@epa.gov](mailto:hughes.melissa@epa.gov)

### Geographic Location Data

- Stations
- Sampling Visits
- Observed Objects

### Water Measurements

- Physical, Chemical and Nutrients
- Vertical Profile Information

### Benthic Macroinvertebrate Data

- Benthic Grab Replicates
- Replicate Abundance Data
- Replicate Biomass Data
- Summary Abundance Data by Taxon by Station
- Summary Abundance and Physical Data by Station
- Benthic Indices

### Sediment Measurements

- Chemical Analyses
- Grain Size
- Toxicity: Sediment/Microtox Test

### Netted Organisms

- Collection Information and Replicate Abundance
- Trawl Abundance Summary Data by Taxon by Station
- Trawl Abundance Summary Data by Station
- Tissue Analyte Measurements
- Fish Pathology

Stations

Geographic location and statistical information appear in the station location data set. Latitude and longitude are required for each station. Other geographic information aids in subsetting and analyzing the data. Statistical data (station area, strata) are also useful for statistical analyses. There is only one record for each station. Descriptions followed by (code) should refer to the GEOGRAPHIC CODE data sets for examples.

Data Set Name: STA_LOC		Station location data			Variables: 17
#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier designated by sampling group
2	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling (code)
3	<b>SAMPYEAR</b>	Num	8	4.	Year of sampling
4	<b>REG_CODE</b>	Char	4	\$4.	EPA region code (code)
5	<b>SYS_CODE</b>	Char	6	\$6.	Large water body where station located (code)
6	<b>STATE</b>	Char	2	\$2	FIPS State code (code)
7	CLASCODE	Char	18	\$18.	Station class-determines sampling regime (code)
8	STRATA	Char	6	\$6.	Design strata:large/small/tidal river (code)
9	ESTUARY	Char	50	\$50.	Small water body where station located
10	STA_AREA	Num	8	7.2	Statistical area (sq. km.) of station
11	<b>LNGITUDE</b>	Num	8	9.3	Longitude of station
12	<b>LATITUDE</b>	Num	8	9.3	Latitude of station
13	RESOURCE	Char	20	\$20.	Project conducting sampling (code)
14	EMAPSTAT	Char	20	\$20.	EMAP station name
15	SEGMENT	Char	20	\$20.	Segment in which station is located
16	MAIASTAT	Char	20	\$20.	MAIA station name
17	PROVINCE	Char	4	\$4.	EMAP Province (code)

Sorted by: SAMPYEAR STATION

## Sampling Visits

Each visit to a station is recorded in the sampling visits data set. One station may have multiple records with a unique sample collection date and visit number. All other data sets must have a station and date combination that matches one in sampling visits.

Data Set Name: SAMP\_VIS      Sampling Visit Information      Variables: 7

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
4	<b>SAMPYEAR</b>	Num	8	4.	Year during which data were collected
5	<b>DEPTH</b>	Num	8	5.1	Depth at station at time of sampling
6	<b>D_UNITS</b>	Char	4	\$4.	Depth units (m, ft)
7	<b>VISNUM</b>	Num	8	2.	Number of visit to station

Sortedby: STATION VST\_DATE VISNUM

## Observed Objects

Data groups have recorded the presence of 'man\_made' or 'natural' objects in trawls and visually from the sampling boats. Objects are recorded as present/absent (Y/N) in OBJ\_PRES either seen from the working platform (visually) or collected in a trawl (trawl) in OBS\_MADE. Man\_made objects include balls, cans, bottles, metal, paper, man-made wood, among others and could be considered as 'trash'. Natural material include objects like natural wood, algae or dead organisms. All information should be condensed to one record/station per visit or per trawl and are not considered quantitative.

Data Set Name: OBS\_OBJ      Objects (man-made/natural) observed      Variables: 9

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Date of sample collection
3	<b>DATA_GRP</b>	Char	4	\$4.	Group collecting data
4	<b>SAMPYEAR</b>	Num	4	4.	Year in which data were collected
5	<b>REPNUM</b>	Num	3	3.	Trawl replicate or visit number
6	<b>OBS_MADE</b>	Char	20	\$20.	Object observed from: report as 'trawl' or 'visually'
7	<b>OBJ_PRES</b>	Char	3	\$3.	Object present: Y/N
8	<b>OBJ1</b>	Char	20	\$20.	Man-made object or natural material
9	<b>OBJ2</b>	Char	20	\$20.	Man-made object or natural material

——Sort Information——

Sortedby: STATION EVNTDATE REPNUM

## Benthic Macroinvertebrate Data

Results and field data from benthic samples can be provided at several levels: replicate abundance and biomass measurements, abundance data summarized by taxon and station or abundance and physical measurements summarized at the station level. Latin names should be abbreviated to an 8 letter code used consistently throughout all data sets. A code lookup table is detailed later. Replicate results data are related to a benthic grab data set (BENGRABS) which provides one record for each replicate sample collected at a station. Even if sediment data are not available for each replicate, collection and gear information should be reported.

Data Set Name: BENGRABS      Benthic grab replicate information      Variables: 12

#	Variable	Type	Len	Format	Label
1	<b>DATA_GRP</b>	Char	4	\$4.	Group collecting data
2	<b>SAMPYEAR</b>	Num	4	4.	The year sampling occurred
3	<b>STATION</b>	Char	12	\$12.	Station identifier
4	<b>VST_DATE</b>	Num	8	DATE8.	Date samples was conducted
5	<b>REP_NUM</b>	Num	8	2.	Benthic grab replicate number
6	<b>BENDEPTH</b>	Num	8	4.	Depth of grab penetration (mm)
7	<b>SILTCLAY</b>	Num	8	6.3	Silt-clay content (%)
8	<b>MOISTURE</b>	Num	8	5.2	Moisture content (%)
9	<b>RPDDEPTH</b>	Num	8	3.	Redox potential discontinuity depth (mm) by replicate
10	<b>GRABAREA</b>	Num	8	6.2	Area sampled by benthic grab
11	<b>AREAUNTS</b>	Char	8	\$8.	Units of area sampled
12	<b>COL_GEAR</b>	Char	250	\$30.	Name of benthic sampling gear

Sortedby: STATION VST\_DATE REP\_NUM

## Replicate Abundance Data

The benthic replicate abundance measurements should be provided with one record for each taxon found in a replicate for each station visit. Sieve size may further subset the data, but is not mandatory. Codes for SPEC\_IGN are resolved in Appendix A.

Data Set Name: BEN\_ABUN      Benthic Abundance by replicate      Variables: 9

#	Variable	Type	Len	Format	Label
1	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
2	<b>SAMPYEAR</b>	Num	4	4.	Year during which data were collected
3	<b>STATION</b>	Char	12	\$12.	Station identifier
4	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
5	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
6	<b>REP_ABN</b>	Num	8	6.	Taxon abundance (# / sample)
7	<b>SPEC_IGN</b>	Char	1	\$1.	Flag: if 1 ignore taxon for # taxon
8	<b>REP_NUM</b>	Num	8	1.	Replicate number
9	<b>SIEVE_MM</b>	Num	8	5.2	Sieve size (mm)

Sortedby: STATION VST\_DATE REP\_NUM TSN SIEVE\_MM

### Replicate Biomass Data

Benthic biomass measurements should be provided as one record for each taxonomic group weighed per sample. Sieve size may be a factor, but is not mandatory. Each station, visit date, replicate number combination should have a record in BENGRABS.

Data Set Name: BIOMASS      Benthic biomass data by replicate      Variables: 9

#	Variable	Type	Len	Format	Label
1	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
2	<b>SAMPYEAR</b>	Num	4	4.	Year during which data were collected
3	<b>STATION</b>	Char	2	\$12.	Station identifier
4	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
5	<b>REP_NUM</b>	Num	3	3.	Sample replicate number
6	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
7	<b>SIEVE_MM</b>	Num	8	5.2	Sieve size (mm)
8	<b>BIOMASS</b>	Num	8	7.5	Biomass (g / Sample)
9	<b>BIOM_ABN</b>	Num	4	4.	Count (#) of organisms. in biomass sample

Sortedby: STATION VST\_DATE REP\_NUM TSN SIEVE\_MM

### Summary Abundance Data by Taxon by Station

The benthic station abundance values should be provided with one record for each taxon found per station. Mean abundance is calculated across 'n' grabs collected at a station.

Data Set Name: BEN\_SPEC      Benthic Species by taxon and station      Variables: 8

#	Variable	Type	Len	Format	Label
1	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
2	<b>SAMPYEAR</b>	Num	4	4.	Year of sample collection
3	<b>STATION</b>	Char	12	\$12.	Station identifier
4	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
5	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
6	<b>BSPECABN</b>	Num	8	6.	Organisms of the taxon:total #
7	<b>BSPEC_MA</b>	Num	8	6.2	Organisms of the taxon:mean #/grab
8	<b>BSPECSTD</b>	Num	8	6.2	Organisms of the taxon:SD of mean/grab

Sortedby: STATION VST\_DATE TSN



## Summary Abundance and Physical Data by Station

The benthic station summary values should be provided with one record for each station. Values are calculated across all grabs and all or a subset of taxa collected at a station.

Data Set Name: BENTHOS      Benthic Summary by Station      Variables:      23

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
4	<b>SAMPYEAR</b>	Num	8	4.	Year sampling conducted
5	<b>N_ABUN</b>	Num	8	3.	# grabs analyzed, abundance data
6	<b>BSP_TOT</b>	Num	8	6.	Total # benthic taxa in 'n' grabs
7	<b>TNSP_INF</b>	Num	8	4.	Total number of infauna taxa
8	<b>TNSP_EPI</b>	Num	8	4.	Total number of epifauna taxa
9	<b>BSP_MEAN</b>	Num	8	7.2	Mean # benthic taxa in 'n' grabs
10	<b>MNSP_INF</b>	Num	8	7.2	Mean number of infauna taxa per grab
11	<b>MNSP_EPI</b>	Num	8	7.2	Mean number of epifauna taxa per grab
12	<b>BSP_TABN</b>	Num	8	6.	Total abundance per grab, all organisms
13	<b>INF_TABN</b>	Num	8	6.	Total abundance per grab, all infauna
14	<b>EPI_TABN</b>	Num	8	6.	Total abundance per grab, all epifauna
15	<b>BSP_MABN</b>	Num	8	7.2	Total abundance per grab, all organisms
16	<b>INF_MABN</b>	Num	8	7.2	Mean abundance per grab, all infauna
17	<b>EPI_MABN</b>	Num	8	7.2	Mean abundance per grab, all epifauna
18	<b>BMAS_MN</b>	Num	8	6.4	Mean biomass per grab, all species
19	<b>BMAS_TOT</b>	Num	8	6.4	Total biomass per grab, all species
20	<b>SICL_B_M</b>	Num	8	6.3	Mean silt/clay content (%) in 'n' cores
21	<b>MOIS_M</b>	Num	8	5.2	Mean moisture content (%) in 'n' cores
22	<b>GRAB_PEN</b>	Num	8	4.	Grab penetration: mean depth (mm)
23	<b>RPD_MDEP</b>	Num	8	3.	Redox potential discontinuity:(RPD) mean depth (mm)
24	<b>H_DIV_IND</b>	Num	8	6.3	Mean infaunal H prime diversity per grab

Sortedby:      STATION VST\_DATE SAMPYEAR

## Benthic Index Data

Some groups have established an algorithm to estimate if a station is considered in degraded or non-degraded condition. The values are presented by station and date. Each data group would have a separate table since algorithms would be different.

Data Set Name: B\_INDEX      Benthic Index Data      Variables:      5

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group collecting data
4	<b>SAMPYEAR</b>	Num	4	4.	Year during which data were collected
5	<b>B_INDEX</b>	Num	8	9.5	Benthic index: VA94 algorithm

Sortedby:      STATION VST\_DATE

## Water Measurements

Physical, chemical and nutrient measurements taken with instruments or under ambient conditions are presented in water measurements. Each measurement taken is defined under WM\_NAME. The location in the water column where the sample was taken (COL\_LOC) is recorded as well as the method. QA codes can be associated with individual measurements. A list of currently used Water measurement names appears in Appendix A.

Data Set Name: WTR\_MEAS      Water measurement data ( physical, nutrient)      Variables:      13

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
4	<b>SAMPYEAR</b>	Num	4	4.	Year of sample collection
5	<b>WM_UNITS</b>	Char	10	\$10.	Measurement units
6	<b>WM_NAME</b>	Char	25	\$25.	Measurement name
7	<b>MEASURE</b>	Num	8	13.4	Measurement or concentration
8	<b>COL_LOC</b>	Char	10	\$10.	Collection location (Surface, mid, bottom, varies)
9	<b>MEAS_DEP</b>	Num	8	5.1	Measurement depth
10	<b>DEP_UNIT</b>	Char	2	\$2.	Depth units (m, ft)
11	<b>COL_PROP</b>	Char	25	\$25.	Collection property: vertical profile/ambient
12	<b>METHOD</b>	Char	25	\$25.	Analysis method
13	<b>QA_CODE</b>	Char	15	\$15.	Quality assurance code related to water measurement (code)

Sortedby:      STATION VST\_DATE WM\_NAME COL\_LOC

Alternatively, water quality data can be submitted in a more conventional manner with each parameter as an attribute in the data set. Nutrient data could also appear in this format.

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	8	8.	Station identifier
2	<b>VST_DATE</b>	Num	8	YYMMDD6.	The date the sample was collected
3	<b>SRF_DO</b>	Num	8	5.1	Dissolved oxygen (mg/l) at the surface
4	<b>SRF_TEMP</b>	Num	8	5.2	Temperature (C) at the surface
5	<b>SRF_SAL</b>	Num	8	5.2	Salinity (ppt) at the surface
6	SRF_PH	Num	8	5.1	pH (units) at the surface
7	SRF_PAR	Num	8	5.	PAR (mE/m2/s) at the surface
8	SRF_TRNS	Num	8	4.	Transmissivity (%) at the surface
9	SRF_FLR	Num	8	4.	Fluorescence at the surface
10	SRF_DENS	Num	8	5.2	Density (Sigma T) at the surface
11	<b>BTM_DO</b>	Num	8	5.1	Dissolved Oxygen (mg/l) at the bottom
12	<b>BTM_TEMP</b>	Num	8	5.2	Temperature (C) at the bottom
13	<b>BTM_SAL</b>	Num	8	5.2	Salinity (ppt) at the bottom
14	BTM_PH	Num	8	5.1	pH (units) at the bottom
15	BTM_PAR	Num	8	5.	PAR (mE/m2/s) at the bottom
16	BTM_TRNS	Num	8	4.	Transmissivity (%) at the bottom
17	BTM_FLR	Num	8	4.	Fluorescence at the bttom
18	BTM_DENS	Num	8	5.2	Density (Sigma T) at the bottom
19	MAX_FLR	Num	8	4.	Maximum fluorescence measured in VP file
20	K_PAR	Num	8	7.3	Rate of light extinction
21	AVG_K	Num	8	7.3	Average rate of light extinction
22	COMP_PAR	Num	8	5.1	Depth where PAR = 1 % of SRF PAR
23	TRNS_1MT	Num	8	4.	Transmissivity (%) at 1 meter
24	QA_CODE	Char	30	\$30.	Quality Assurance code for data
25	SS_CONC	Num	8	7.1	Total suspended solids conc. (mg/l)
26	SECCHI	Num	8	6.1	Secchi depth (m)

——Sort Information——

Sortedby: STATION VST\_DATE

## Sediment

## Chemical Analyses

Results of sediment chemical analyses should be reported in a single file. It should contain one record for each analyte measured in a sample (multiple records per sample). Only one result (CONC) should be reported for each analyte for each sample. A value for the MDL (method detection limit) must be provided in the DETLIMIT field for every sample where the analyte is not detected or is detected at or below the detection limit.

Data Set Name: SED\_CHEM      Sediment Chemistry analyte concentrations      Variables: 10

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
4	<b>SAMPYEAR</b>	Num	4	4.	Year of sample collection
5	<b>ANALYTE</b>	Char	8	\$8.	Code for analyte measured
6	<b>CONC</b>	Num	8	13.6	Concentration of analyte in sample
7	<b>UNITS</b>	Char	15	\$15.	Concentration units of measure
8	<b>MDL</b>	Num	8	13.6	Method detection limit
9	<b>TOT_ANAL</b>	Num	8	3.	Analytes (#) included in summed conc.
10	<b>QACODE</b>	Char	15	\$15.	Quality assurance code related to sediment analyte (code)

Sortedby: STATION VST\_DATE ANALYTE

## Grain Size

Grain size measurements associated with a sediment chemistry sample should be provided in a data set with one record for each sample.

Data Set Name: SEDGRAIN      Sediment Grain Data      Variables:      16

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
4	<b>SAMPYEAR</b>	Num	4	4.	Year of sample collection
5	Q1_PHI	Num	8	5.1	25% Quartile diameter (Phi)
6	SKEWNESS	Num	8	5.1	Phi Quartile skewness (Folk 1974)
7	SILT_PC	Num	8	5.1	Silt content (%)
8	SICL_PC	Num	8	5.1	Silt-clay content (%)
9	SAND_PC	Num	8	5.1	Sand content (%)
10	CLAY_PC	Num	8	5.1	Clay content (%)
11	Q3_PHI	Num	8	5.1	75% Quartile diameter (Phi)
12	MED_DIAM	Num	8	5.1	Median diameter (Phi)
13	QUARDVTN	Num	8	5.1	Phi Quartile deviation (Folk 1974)
14	MOISTURE	Num	8	5.1	Moisture content (%)
15	TOC	Num	8	6.3	Total organic carbon (TOC) amount
16	TOC_UNITS	Num	8	6.3	Total organic carbon (TOC) units

Sortedby:      STATION VST\_DATE

## Toxicity: Sediment/Microtox Test

Results of all toxicity tests should be reported in the toxicity test data set. These include sediment and Microtox tests and may be conducted on one or more organisms. Mortality or growth data can be summarized several ways.

Data Set Name: TOXICITY      Toxicity Test Data      Variables: 14

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
4	<b>SAMPYEAR</b>	Num	4	4.	Sample collection year
5	<b>TESTSPEC</b>	Char	60	\$60.	Species (Latin name) used in test
6	<b>TESTTYPE</b>	Char	10	\$10.	Type of test - sediment, Microtox
7	<b>RSLTMEAS</b>	Char	15	\$40.	Unit of result (growth/survival/EC50)
8	<b>RESULT</b>	Num	8	5.1	Result value
9	STATCODE	Char	3	\$3.	Sig diff from control (Y/N); toxic, non-toxic, etc.
10	MOISTURE	Num	8	11.1	Moisture content (%)
11	<b>TESTNUM</b>	Num	8	2.	Number of test if replicate of same species
12	P_VALUE	Num	8	7.4	P-value for statistical test
13	PW_UNAM	Num	8	8.3	Un-Ionized ammonia (mg/L) in pore water
14	QACODE	Char	15	\$15.	Quality assurance code(s)

Sortedby: STATION VST\_DATE TESTTYPE TESTSPEC

Alternatively, toxicity data from different tests can be submitted in a more conventional manner with each test parameter as an attribute in the data set.

#	Variable	Type	Len	Format	Label
1	STATION	Char	12	12.	The Station identifier
2	VST_DATE	Num	8	YYMMDD6.	The date the sample was collected
3	LAT_NAME	Char	8	\$8.	Latin name
4	SURVIVAL	Num	8	5.1	Ampelisca % survival (samp nean as % of control)
5	SIG_CONT	Char	8	\$3.	Ampelisca sig diff from control(samp x % mortal'y)
6	EC50_MC	Num	8	12.3	Microtox corrected mean EC50 (%)
7	MTOX_SIG	Char	1	1	Microtox test significance
8	QACODE	Char	15	\$15.	Quality assurance code(s)

——Sort Information——

Sortedby: STATION VST\_DATE



## Netted Organisms

Field data from trawl and seine samples can be provided at several levels: replicate abundance and length measurements, abundance and length data summarized by taxon and station or abundance measurements summarized at the station level. Latin names should be abbreviated to an 8 letter code used consistently throughout all data sets. A code lookup table is detailed later.

## Replicate Abundance and Collection Information

Replicate trawl or seine data are presented as one record for each taxon collected in each replicate trawl or seine conducted at a station. Length can be reported as a mean for all organisms of a taxon or as multiple size classes for a taxon. The taxon information should be resolved in a code table. Gear description and type collection information are also reported.

Data Set Name: NET\_ORG

Abundance of organisms collected by trawl/seine

Member Type: DATA

Variables: 13

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station identifier
2	<b>DATA_GRP</b>	Char	4	\$4.	Group collecting data
3	<b>SAMPYEAR</b>	Num	4	4.	Year during which data were collected
4	<b>VST_DATE</b>	Num	8	DATE8.	Date of sample collection
5	<b>REP_NUM</b>	Num	3	3.	Replicate number
6	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
7	<b>FSPECNUM</b>	Num	8	6.	Total # of organisms in replicate
8	<b>FSPEC_ML</b>	Num	8	6.1	Mean length of organisms
9	<b>FSPEC_SD</b>	Num	8	6.1	Standard dev. length
10	<b>LEN_UNITS</b>	Char	8	\$8.	Length units (mm, cm)
11	<b>NUM_LENS</b>	Num	8	3.	# organisms measured
12	<b>COL_TYPE</b>	Char	5	\$5.	Type of collection:trawl/seine
13	<b>GEARTYPE</b>	Char	250	\$250.	Gear type description
14	<b>SIZECLAS</b>	Num	8	4.	Size class length of organism

Sortedby: STATION VST\_DATE REP\_NUM TSN

### Trawl Abundance Summary Data by Taxon by Station

Trawl abundance data by taxon and station are presented as one record for each taxon collected in each trawl conducted at a station. The taxon information should be resolved in a code table. Codes for measurement types are resolved in Appendix A.

Data Set Name: TRWLTSUM      Trawl Taxon summary      Variables:      9

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	11	\$11.	Station identifier
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group collecting data
4	<b>SAMPYEAR</b>	Num	4	4.	Year during which data were collected
5	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
6	<b>T_ABN</b>	Num	8	5.	Total taxon abundance in 'n' trawls
7	<b>M_LEN</b>	Num	8	5.2	Mean length of taxon in 'n' trawls
8	<b>SDLEN</b>	Num	8	5.2	SD length of taxon in 'n' trawls
9	<b>MEASTYPE</b>	Char	3	\$3.	Code for measurement type

Sortedby:      STATION VST\_DATE TSN

### Trawl Abundance Summary Data by Station

Trawl abundance data by station are presented as one record for all trawls conducted at a station.

Data Set Name: TRWL\_SUM      Trawl Summary data by station      Variables:      11

#	Variable	Type	Len	Format	Label
1	<b>STATION</b>	Char	12	\$12.	Station name
2	<b>VST_DATE</b>	Num	8	DATE8.	Sample collection date
3	<b>DATA_GRP</b>	Char	4	\$4.	Group collecting data
4	<b>SAMPYEAR</b>	Num	4	4.	Year during which data were collected
5	<b>COL_TYPE</b>	Char	5	\$5.	Type of collection - trawl or seine
6	<b>TOT_TRWL</b>	Num	3	2.	Number of trawls/seines conducted
7	<b>F_TOTAL</b>	Num	8	5.	Total organisms (#) trawl
8	<b>FSPECCNT</b>	Num	8	5.	Total taxa (#) in trawl
9	<b>FSPMABN</b>	Num	8	5.1	Mean # organisms in 'n' trawls at a station
10	<b>F_MTOT</b>	Num	8	5.1	Mean taxa (species) in 'n' trawls at a station
11	<b>GEARTYPE</b>	Char	250	\$250.	Type of gear used

Sortedby:      STATION VST\_DATE

## Tissues

## Chemistry Concentrations

## Tissue Analyte Measurements

Results of tissue (fish, shrimp, crab) chemical analyses should be reported as one record for each analyte measured in a sample (multiple records per sample). Either a concentration or detection limit should appear in a record. It is important to include all relevant fields that identify a unique sample, such as: sample number, composite, sample type, tissue type, local name.

Data Set Name: TISUCHEM    Tissue Chemistry Analyses    Variables: 18

#	Variable	Type	Len	Format	Label
1	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
2	<b>SAMPYEAR</b>	Num	4	4.	Year sampling was conducted
3	<b>STATION</b>	Char	12	\$12.	Station identifier
4	<b>VST_DATE</b>	Num	8	DATE8.	Date samples were collected
5	<b>SAMP_NUM</b>	Num	8	3.	Sample number assigned to distinguish samples of the same species at a station
6	<b>COMPOSIT</b>	Char	1	\$1.	Composite code (Y/N). Is this sample a composite?
7	<b>SAMPTYPE</b>	Char	10	\$10.	Nature of sample material (Fish, Shrimp, Crab)
8	<b>TISUTYPE</b>	Char	10	\$10.	Type of tissue sampled (cascass, muscle)
9	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
10	<b>ANALYTE</b>	Char	8	\$8.	Analyte code
11	<b>CONC</b>	Num	8	13.6	Concentration of analyte in sample
12	<b>UNITS</b>	Char	15	\$15.	Concentration units
13	<b>MDL</b>	Num	8	13.6	Method detection limit for analyte
14	<b>TOT_ANAL</b>	Num	3	3.	Number of analytes in total measure
15	<b>NUM_CMPT</b>	Num	3	3.	Number of organisms/composite
16	<b>FSPEC_MM</b>	Num	8	6.1	Mean length (mm) of organisms in sample
17	<b>FSPEC_SD</b>	Num	8	6.1	SD of length (mm) of organisms in sample
18	<b>QACODE</b>	Char	15	\$15.	Quality assurance code(s)

**FISH PATHOLOGY**

Pathology data from organisms collected in trawls/seines may be presented as presence/absence or as counts. These data may be submitted at the replicate level or summarized by Latin name and station.

Data Set Name: FISHPATH    Fish Pathology Observations    Variables: 11

#	Variable	Type	Len	Format	Label
1	<b>DATA_GRP</b>	Char	4	\$4.	Group conducting sampling
2	<b>SAMPYEAR</b>	Num	4	4.	Year sampling was conducted
3	<b>STATION</b>	Char	12	\$12.	Station identifier
4	<b>VST_DATE</b>	Num	8	DATE8.	Date samples were collected
5	<b>REP_NUM</b>	Num	8	2.	Nekton trawl replicate number
6	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
7	<b>PATHPRES</b>	Char	2	\$2.	Y/N - pathology present
8	<b>PATH_CNT</b>	Num	8	3.	Count (#) of pathologies present
9	<b>PATH_LOC</b>	Char	30	\$30.	Area on fish where pathology observed - eyes, mouth, gills,body
10	<b>TYPEPATH</b>	Char	30	\$30.	Pathology description - ulcers, lumps,growths,finrot
11	<b>QACODE</b>	Char	15	\$15.	Quality assurance code(s)

## Appendix D. Code Tables

### Geographic/Statistical Codes

Data Set Name: REG\_CODE      EPA Region code information      Variables: 2

#	Variable	Type	Len	Format	Label
1	<b>REG_CODE</b>	Num	3	2.	EPA Region code where station located
2	<b>DESCR</b>	Char	25	25.	Name of region

Below is the list of REGION codes

<b>REG_CODE</b>	<b>DESCR</b>
1	EPA Region 1
2	EPA Region 2
3	EPA Region 3
4	EPA Region 4
5	EPA Region 5
6	EPA Region 6
7	EPA Region 7
8	EPA Region 8
9	EPA Region 9
10	EPA Region 10

Data Set Name: STRATA      Statistical design strata information      Variables: 2

#	Variable	Type	Len	Format	Label
1	<b>STRATA</b>	Char	6	6.	Design strata: large, Small or tidal river
2	<b>DESCR</b>	Char	40	40.	Strata name

Below is the list of currently used STRATA codes

<b>STRATA</b>	<b>DESCR</b>
L	Large Estuary
O	Small Estuary or Tidal River
TR	Large Tidal River
RR	Large Tidal River
SR	Small Estuary
LR	Large Estuary
SP	Small Estuary Replicate
RP	Large Tidal River Replicate
LE	Large Estuary/Tidal River
S	Small estuary site:random/intensive

Data Set Name: SYS\_CODE      Large water body system code      Variables:    2

#	Variable	Type	Len	Format	Label
1	<b>SYS_CODE</b>	Char	4	4.	Large water body code of sta. location
2	<b>DESCR</b>	Char	60	60.	Name of system

<b>SYS_CODE</b>	<b>DESCR</b>	<b>SYS_CODE</b>	<b>DESCR</b>
SB	Sinepuxent Bay	MS	Mississippi Sound
DCN	Dead-End Canal	PB	Pensacola Bay
IRB	Indian River Bay	PH	Panhandle
BB	Buzzards Bay	SAB	San Antonio Bay
BIS	Block Island Sound	SANB	St. Andrew Bay
CB	Chesapeake Bay	SAS	St. Andrew Sound
DB	Delaware Bay	SGS	St. Georges Sound
DEC	DE Coast-Indian River Basin	SJB	St. Josephs Bay
ELI	Eastern Long Island	SL	Sabine Lake
HR	Hudson River	TB	Terrebone Bay
LIC	Long Island Coast	VB	Vermilion Bay
LIS	Long Island Sound	WFC	West Florida Coast
MDC	Maryland Coast	CHS	Chandeleur Sound
NB	Narragansett Bay	BR	Brazos River
NJC	New Jersey Coast	RG	Rio Grande
NS	Nantucket Sound	COR	Colorado River
VAC	Virginia Coast	SRS	Santa Rosa Sound
AB	Apalachee Bay	FRH	Freeport Harbor
AFB	Atchafalaya Bay	SBR	San Bernard River
APB	Apalachicola Bay	BA	Bight Apex
BRB	Barataria Bay	JB	Jamaica Bay
CCB	Corpus Christi Bay	WLS	Western Long Island Sound
CHB	Choctawhatchee Bay	NKB	Newark Bay
CL	Calcasieu Lake	RB	Raritan Bay
GB	Galveston Bay	UH	Upper New York Harbor
LB	Lake Borgne	UIR	Upper Indian River
LC	Louisiana Coast	AWB	Assawoman Bay
LM	Laguna Madre	TCNB	Trappe Creek/Newport Bay
LP	Lake Pontchartrain	LIR	Lower Indian River
LS	Lake Salvador	SMR	St. Martin River
LW	Lake Wimico	RHB	Rehoboth Bay
MB	Matagorda Bay	LAB	Lower Assawoman Bay
MBB	Mobile Bay	CTB	Chincoteague Bay
MR	Mississippi River	CD	Coastal Delaware
AP	Albemarle/Pamlico Sound	SCB	Southern California Bight
ATL	Atlantic Ocean		

Data Set Name: STATE      State code resolution      Variables: 3

#	Variable	Type	Len	Format	Label
1	<b>STATE</b>	Char	2	2.	Code for state
2	<b>DESCR</b>	Char	15	25.	Name of state
	<b>STATE</b>	<b>DESCR</b>			
	AK	Alaska			
	AL	Alabama			
	CA	California			
	CT	Connecticut			
	DC	D. of Columbia			
	DE	Delaware			
	FL	Florida			
	GA	Georgia			
	HI	Hawaii			
	LA	Louisiana			
	MA	Massachusetts			
	MD	Maryland			
	ME	Maine			
	MS	Mississippi			
	NC	North Carolina			
	NH	New Hampshire			
	NJ	New Jersey			
	NY	New York			
	OR	Oregon			
	PA	Pennsylvania			
	PR	Puerto Rico			
	RI	Rhode Island			
	SC	South Carolina			
	TX	Texas			
	VA	Virginia			
	WA	Washington			



Data Set Name: CLASCODE Station Classification information Variables: 2

#	Variable	Type	Len	Format	Label
1	<b>CLASCODE</b>	Char	18	18.	Station class-determines sampling regime
2	<b>DESCR</b>	Char	80	80.	Name of station class

Below is a list of currently used Station Classification codes

<b>CLASCODE</b>	<b>Description</b>
BASE	Base Sampling Site
BASE/ITE	Base Sampling/Indicator Testing and Evaluation Site
BASE/ITE/LTDO	Base Sampling/Indicator Testing and Evaluation/Long Term Dissolved Oxygen Site
BASE/LTDO	Base Sampling/Long Term Dissolved Oxygen Site
REP	Spatial Replicate Station
SUPPLEMENT	Supplement
RANDOM-BASE	Random-Base
INTENSIVE	Intensive
REVISIT	Revisit
REFERENCE	Reference
ITE	Indicator Testing and Evaluation Site
SUPP	Supplemental
LTS	Long Term Spatial
LTT	Long Term Trend
IND	Index Stations
OTH	Other
LTDO	Long Term Dissolved Oxygen Site
BSS/LTT	Base Sampling/Long Term Trend
REP-92	Replicate 1992
REP-93	Replicate 1993
REP-94	Replicate 1994
Random	Random Site
Non-Random	Non-Random Site
CBP-BNT	Chesapeake Bay Program benthic monitoring site
CBP-WTR	Chesapeake Bay Program water monitoring site
INT	Spatially intensive sampling site
SE	Randomly selected small estuary site
MS	Mainstem site: Chesapeake, Delaware, Chincoteague Bays
INT/SE	Spatially intensive/Randomly selected small estuary site
QA/QC	Quality Assurance/Quality Control site

#### Taxonomic codes:

A table should identify each taxon code found in the benthic and netted organism abundance data. One record should be provided for each ITIS code. Data in all taxonomic abundance and biomass data sets should be summarized to the next highest taxonomic level if the lower taxonomic level cannot be identified. For example, if there are several species of *Ampelisca* present, but the species can't be identified, the data should be summarized to the genus level and not transmitted as *Ampelisca sp. A*, *Ampelisca sp. B*, etc. The ITIS Taxonomic Serial Number (TSN) is very important. The TSN's for most taxon can be extracted from the ITIS database, found at: <http://www.itis.usda.gov/itis/>. Minimally, the information in bold should be provided. If the taxon is not in ITIS, then EMAP IM will provide a surrogate ITIS code. Complete taxonomic information, to Phylum, must be provided by the transmitter, as well as the original species citation information to verify the name. Once this is provided, the taxon will be submitted to ITIS for an official serial number.

A list of current ITIS codes and taxonomic names can be obtained by sending email to [hughes.melissa@epa.gov](mailto:hughes.melissa@epa.gov). Data groups can match taxon names to this list to find an ITIS code. All taxon names not having a match in this list should be queried against the ITIS database (URL above). Only codes lists incorporating ITIS codes will be accepted.

Data Set Name: TAXONOMY    Benthic/Fish/Invertebrate Taxon Information    Variables: 9

#	Variable	Type	Len	Format	Label
1	<b>TSN</b>	Char	8	\$8.	ITIS Taxonomic Serial Number
2	<b>LAT_NAME</b>	Char	80	\$80.	Latin name of taxon
3	COMNAME	Char	20	\$30.	Common name of taxon
4	KINGDOM	Char	20	\$30.	Kingdom level of taxon
5	PHYLUM	Char	20	\$30.	Phylum level of taxon
6	CLASS	Char	20	\$30.	Class level of taxon
7	ORDER	Char	20	\$30.	Order level of taxon
8	FAMILY	Char	20	\$30.	Family level of taxon
9	GENUS	Char	20	\$30.	Genus level of taxon
10	SPECIES	Char	25	\$30.	Species level of taxon

If the taxon name is not present in ITIS, then a surrogate code number will be assigned by EMAP IM. The above information becomes mandatory and the information below must also be provided in order to submit the names to ITIS:

#	Variable	Type	Len	Format	Label
1	<b>TSN</b>	Char	8	8.	ITIS Taxonomic Serial Number
2	<b>LAT_NAME</b>	Char	80	\$80.	Latin name of taxon
3	<b>COMNAME</b>	Char	20	\$30.	Common name of taxon
4	<b>KINGDOM</b>	Char	20	\$30.	Kingdom level of taxon
5	<b>PHYLUM</b>	Char	20	\$30.	Phylum level of taxon
6	<b>CLASS</b>	Char	20	\$30.	Class level of taxon
7	<b>ORDER</b>	Char	20	\$30.	Order level of taxon
8	<b>FAMILY</b>	Char	20	\$30.	Family level of taxon
9	<b>GENUS</b>	Char	20	\$30.	Genus level of taxon
10	<b>SPECIES</b>	Char	25	\$30.	Species level of taxon
11	<b>AUTHOR</b>	Char	40	\$60.	Author of publication originally naming taxon
12	<b>DATE</b>	DATE	8	DATE8.	Date of publication
13	<b>CITATION</b>	Char	200	\$200.	Citation of paper originally naming taxon

#### Chemical Codes

A table should identify each analyte (ANALYTE) code found in the sediment analyte and tissue chemistry concentration data. The analyte code for each analyte is an 8-letter code for the official chemical name of a compound. A list of current codes is provided as a separate file (chemcomp.asc). Only codes listed in this file should be used. Analytes not listed should be submitted to AED for code assignment. For this reason the CAS Number (CAS\_NUM) is very important to define an official chemical name. The CAS number for most chemical names can be extracted from EPA's Chemical Registry System found at: [http://www.epa.gov:6706/crsdcd/owa/chemqry\\$.startup](http://www.epa.gov:6706/crsdcd/owa/chemqry$.startup). The information in bold should be provided.

Data Set Name: CHEMCOMP      Chemical compound information      Variables: 4

#	Variable	Type	Len	Format	Label
1	<b>ANALYTE</b>	Char	8	\$8.	Analyte code
2	<b>CAS_NUM</b>	Char	12	12.	CAS number
3	<b>CHEMNAME</b>	Char	80	80.	Full chemical name
4	<b>DESCR</b>	Char	20	20.	Description of code, i.e., organic, inorganic compound

## Analyte Codes

ANALYTE;CAS\_NUM;CHEMNAME;

6CLBNZ;118741;HEXACHLOROBENZENE;  
ABHC;319846;ALPHA-HEXACHLOROCYCLOHEXANE;  
ACENTHE;83329;ACENAPHTHENE;  
ACENTHY;208968;ACENAPHTHYLENE;  
AG;7440224;SILVER;  
AL;7429905;ALUMINUM;  
ALDRIN;309002;ALDRIN;  
ALKANE\_T;.;TOTAL ALKANES;  
ALPHACHL;5103719;ALPHA-CHLORDANE;  
ANTHRA;120127;ANTHRACENE;  
AS;7440382;ARSENIC;  
AVS;18496258;ACID VOLATILE SULFIDES;  
BA;7440393;BARIUM;  
BBHC;319857;BETA-HEXACHLOROCYCLOHEXANE;  
BE;7440417;BERYLLIUM;  
BENANTH;56553;BENZ(A)ANTHRACENE;  
BENAPY;50328;BENZO(A)PYRENE;  
BENEPY;192972;BENZO(E)PYRENE;  
BENZOBFL;205992;BENZO(B)FLUORANTHENE;  
BENZOF.;.;BENZO(B+K)FLUORANTHENE;  
BENZOKFL;207089;BENZO(K)FLUORANTHENE;  
BENZOP;191242;BENZO(G,H,I)PERYLENE;  
BHC\_TOT;.;SUM OF BHC (HEXACHLOROCYCLOHEXANE) COMPOUNDS;  
BIPHENYL;92524;BIPHENYL;  
BT\_TOT;.;TOTAL BUTYLINS;  
C10\_ALKA;124185;C10-ALKANE (N-DECANE ALIPHATIC HYDROCARBON);  
C11\_ALKA;1120214;C11-ALKANE (N-UNDECANE ALIPHATIC HYDROCARBON);  
C12\_ALKA;112403;C12-ALKANE (N-DODECANE ALIPHATIC HYDROCARBON);  
C13\_ALKA;629505;C13-ALKANE (N-TRIDECANE ALIPHATIC HYDROCARBON);  
C14\_ALKA;629594;C14-ALKANE (N-TETRADECANE ALIPHATIC HYDROCARBON);  
C15\_ALKA;629629;C15-ALKANE (N-PENTADECANE ALIPHATIC HYDROCARBON);  
C16\_ALKA;544763;C16-ALKANE (N-HEXADECANE ALIPHATIC HYDROCARBON);  
C17\_ALKA;629787;C17-ALKANE (N-HEPTADECANE ALIPHATIC HYDROCARBON);  
C18\_ALKA;593453;C18-ALKANE (N-OCTADECANE ALIPHATIC HYDROCARBON);  
C19\_ALKA;629925;C19-ALKANE (N-NONADECANE ALIPHATIC HYDROCARBON);  
C1CHRY.;.;C1-CHRYSENE;  
C1DIBENZ;.;C1-DIBENZOTHIOPHENES;  
C1FLRAN;.;C1-FLUORANTHENES + PYRENES;  
C1FLUOR;.;C1-FLUORENES;  
C1NAPH;.;C1-NAPHTHALENES;  
C1PHENAN;.;C1-PHENANTHRENE;  
C20\_ALKA;112958;C20-ALKANE (N-EICOSANE ALIPHATIC HYDROCARBON);  
C21\_ALKA;629947;C21-ALKANE (N-HENEICOSANE ALIPHATIC HYDROCARBON);  
C22\_ALKA;629970;C22-ALKANE (N-DOCOSANE ALIPHATIC HYDROCARBON);  
C23\_ALKA;638675;C23-ALKANE (N-TRICOSANE ALIPHATIC HYDROCARBON);  
C24\_ALKA;646311;C24-ALKANE (N-TETRACOSANE ALIPHATIC HYDROCARBON);  
C25\_ALKA;629992;C25-ALKANE (N-PENTACOSANE ALIPHATIC HYDROCARBON);  
C26\_ALKA;630013;C26-ALKANE (N-HEXACOSANE ALIPHATIC HYDROCARBON);  
C27\_ALKA;593497;C27-ALKANE (N-HEPTACOSANE ALIPHATIC HYDROCARBON);  
C28\_ALKA;630024;C28-ALKANE (N-OCTACOSANE ALIPHATIC HYDROCARBON);  
C29\_ALKA;630035;C29-ALKANE (N-NONACOSANE ALIPHATIC HYDROCARBON);  
C2CHRY.;.;C2-CHRYSENE;  
C2DIBENZ;.;C2-DIBENZOTHIOPHENES;

C2FLUOR;.;C2-FLUORENES;  
C2NAPH;.;C2-NAPHTHALENES;  
C2PHENAN;.;C2-PHENANTHRENES;  
C30\_ALKA;638686;C30-ALKANE (N-TRIACONTANE ALIPHATIC HYDROCARBON);  
C31\_ALKA;630046;C31-ALKANE (N-HENTRIACONTANE ALIPHATIC HYDROCARBON);  
C32\_ALKA;544854;C32-ALKANE (N-DOTRIACONTANE ALIPHATIC HYDROCARBON);  
C33\_ALKA;630057;C33-ALKANE (N-TRITRIACONTANE ALIPHATIC HYDROCARBON);  
C34\_ALKA;14167590;C34-ALKANE (N-TETRATRIACONTANE ALIPHATIC HYDROCARBON);  
C3CHRYS;.;C3-CHRYSENES;  
C3DIBENZ;.;C3-DIBENZOTHIOPHENES;  
C3FLUOR;.;C3-FLUORENES;  
C3NAPH;.;C3-NAPHTHALENES;  
C3PHENAN;.;C3-PHENANTHRENES;  
C4CHRYS;.;C4-CHRYSENES;  
C4FLUOR;.;C4-FLUORENES;  
C4NAPH;.;C4-NAPHTHALENES;  
C4PHENAN;.;C4-PHENANTHRENES;  
CA;7440702;CALCIUM;  
CARBOFEN;786196;CARBOPHENOTHION;  
CD;7440439;CADMIUM;  
CHL\_TOT;.;SUM OF CHLORDANE COMPOUNDS;  
CHLA\_FL;.;CHLOROPHYLL\_A CONC (FLUOROMETRIC METHOD);  
CHLA\_HP;.;CHLOROPHYLL\_A CONC (HPLC METHOD);  
CHRYSENE;218019;CHRYSENE;  
CISNONA;5103731;CIS-NONACHLOR;  
CLOSTR;.;CLOSTRIDIUM;  
CO;7440484;COBALT;  
CR;7440473;CHROMIUM;  
CU;7440508;COPPER;  
DBHC;319868;DELTA-HEXACHLOROCYCLOHEXANE;  
DBT;.;DIBUTYLTIN;  
DDD\_TOT;.;2,4'-DDD + 4,4'-DDD;  
DDE\_TOT;.;2,4'-DDE + 4,4'-DDE;  
DDT\_TOT;.;2,4'-DDT + 4,4'-DDT;  
DIAZINON;333415;DIAZINON;  
DIBENZA;53703;DIBENZ[A,H]ANTHRACENE;  
DIBENZO;132650;DIBENZOTHIOPHENE;  
DICOVOL;115322;DICOVOL;  
DIELDRIN;60571;DIELDRIN;  
DIMETH;581420;2,6-DIMETHYLNAPHTHALENE;  
DISULFOT;298044;DISULFOTON;  
DURBAN;2921882;CHLORPYRIFOS;  
ENDOSLFT;1031078;ENDOSULFAN SULFATE;  
ENDOSUI;959988;ALPHA-ENDOSULFAN;  
ENDOSUII;33213659;BETA-ENDOSULFAN;  
ENDOSULF;115297;ENDOSULFAN;  
ENDRIN;72208;ENDRIN;  
ENDRIN\_A;7421934;ENDRIN ALDEHYDE;  
ENDRIN\_K;53494705;ENDRIN KETONE;  
ETHION;563122;ETHION;  
FE;7439896;IRON;  
FLUORANT;206440;FLUORANTHENE;  
FLUORENE;86737;FLUORENE (9H-FLUORENE);  
GAMMACHL;5566347;GAMMA-CHLORDANE;  
HEPT\_TOT;.;HEPTACHLOR + HEPTACHLOR EPOXIDE;  
HEPTACHL;76448;HEPTACHLOR;  
HEPTAEPO;1024573;HEPTACHLOR EPOXIDE;

HG;7439976;MERCURY;  
INDENO;193395;INDENO(1,2,3-C,D)PYRENE;  
ISOPRN\_T;.;TOTAL ISOPRENOIDS;  
LINDANE;58899;LINDANE;  
LIPID;.;LIPID;  
MBT;.;MONOBUTYLTIN;  
MENAP1;90120;1-METHYLNAPHTHALENE;  
MENAP2;91576;2-METHYLNAPHTHALENE;  
MEPHEN1;31711532;1-METHYLPHENANTHRENE;  
MG;7439954;MAGNESIUM;  
MIREX;2385855;MIREX;  
MN;7439965;MANGANESE;  
MOISTURE;.;MOISTURE;  
MTLS\_TOT;.;TOTAL METALS;  
NAPH;91203;NAPHTHALENE;  
NI;7440020;NICKEL;  
OPDDD;53190;2,4'-DDD;  
OPDDE;3424826;2,4'-DDE;  
OPDDT;789026;2,4'-DDT;  
OXYCHL;27304138;OXYCHLORDANE;  
OXYFL;42874033;OXYFLUORFEN;  
P;7723140;PHOSPHORUS;  
PAH\_HMW;.;HIGH MOLECULAR WEIGHT PAHS;  
PAH\_LMW;.;LOW MOLECULAR WEIGHT PAHS;  
PAH\_TOT;.;TOTAL PAHS;  
PB;7439921;LEAD;  
PCB\_TOT;.;TOTAL PCBS;  
PCB101;.;PCB CONGENER 101/90;  
PCB105;32598144;2,3,3',4,4'-PENTACHLOROBIPHENYL;  
PCB110;.;PCB 110/77;  
PCB118;.;PCB CONGENER 118/108/149;  
PCB126;57465288;3,3',4,4',5-PENTACHLOROBIPHENYL;  
PCB128;38380073;2,2',3,3',4,4'-HEXACHLOROBIPHENYL;  
PCB138;35065282;2,2',3,4,4',5'-HEXACHLOROBIPHENYL;  
PCB138a;.;PCB CONGENER 138/160;  
PCB153;35065271;2,2',4,4',5,5'-HEXACHLOROBIPHENYL;  
PCB153a;.;PCB CONGENER 153/132;  
PCB170;.;PCB CONGENER 170/190;  
PCB18;37680652;2,2',5-TRICHLOROBIPHENYL;  
PCB18\_17;.;PCB CONGENER 18/17;  
PCB180;35065293;2,2',3,4,4',5,5'-HEPTACHLOROBIPHENYL;  
PCB187;.;PCB CONGENER 187/182/159;  
PCB195;.;PCB CONGENER 195/208;  
PCB200;52663737;2,2',3,3',4,5,6'-OCTACHLOROBIPHENYL;  
PCB206;40186729;2,2',3,3',4,4',5,5',6-NONACHLOROBIPHENYL;  
PCB209;2051243;DECACHLOROBIPHENYL;  
PCB28;7012375;2,4,4'-TRICHLOROBIPHENYL;  
PCB29;15862074;2,4,5-TRICHLOROBIPHENYL;  
PCB44;41464395;2,2',3,5'-TETRACHLOROBIPHENYL;  
PCB52;35693993;2,2',5,5'-TETRACHLOROBIPHENYL;  
PCB66;32598100;2,3',4,4'-TETRACHLOROBIPHENYL;  
PCB77;32598133;3,3',4,4'-TETRACHLOROBIPHENYL;  
PCB8;.;PCB CONGENER 8/5;  
PCB87;38380028;2,2',3,4,5'-PENTACHLOROBIPHENYL;  
PCB99;38380017;2,2',4,4',5-PENTACHLOROBIPHENYL;  
PERYLENE;198550;PERYLENE;  
PEST\_TOT;.;TOTAL CHLORINATED PESTICIDES;



PHENANTH;85018;PHENANTHRENE;  
PHYTANE;638368;PHYTANE;  
PPDDD;72548;4,4'-DDD;  
PPDDE;72559;4,4'-DDE;  
PPDDT;50293;4,4'-DDT;  
PRISTANE;1921706;PRISTANE;  
PYRENE;129000;PYRENE;  
S;7704349;SULFUR;  
SB;7440360;ANTIMONY;  
SE;7782492;SELENIUM;  
SEM\_CD;.;SEM- CADMIUM;  
SEM\_CU;.;SEM- COPPER;  
SEM\_NI;.;SEM- NICKEL;  
SEM\_PB;.;SEM- LEAD;  
SEM\_ZN;.;SEM- ZINC;  
SI;7440213;SILICON;  
SN;7440315;TIN;  
SR;7440246;STRONTIUM;  
T2PAHC;.;CONC. OF TOTAL 2-RING PAHS;  
T3PAHC;.;CONC. OF TOTAL 3-RING PAHS;  
T4PAHC;.;CONC. OF TOTAL 4-RING PAHS;  
T5PAHC;.;CONC. OF TOTAL 5-RING PAHS;  
T6PAHC;.;CONC. OF TOTAL 6-RING PAHS;  
TBT;.;TRIBUTYLTIN;  
TBT4;1461252;TETRABUTYLTIN;  
TCMX;877098;2,4,5,6-TETRACHLORO-m-XYLENE;  
TERBUFOS;13071799;TERBUFOS;  
TL;7440280;THALLIUM;  
TNONCHL;39765805;TRANS-NONACHLOR;  
TOC;.;TOTAL ORGANIC CARBON;  
TOT\_DDT;.;SUM OF DDTS;  
TOXAPHEN;8001352;TOXAPHENE;  
TRIMETH;2245387;2,3,5-TRIMETHYLNAPHTHALENE;  
V;7440622;VANADIUM;  
ZN;7440666;ZINC;

# Chemical Units

In many data sets unit codes are assigned. Below is a list of units to use.

Data Set Name: CHMUNITS

Chemical units information Variables: 2

#	Variable	Type	Len	Format	Label
1	<b>UNITS</b>	Char	15	15.	Concentration/measurement units
2	<b>DESCR</b>	Char	25	25.	Description of code
	<b>UNITS</b>				<b>DESCR</b>
	mE/sec/m2				micro-Einsteins/second/meter squared
	%				per cent
	ng/g				nannograms/gram
	ug/g				micrograms/gram
	umoles/g				micromoles/gram
	m				meters
	deg C				degrees Celsius
	flr units				Fluorescence units
	kg/m**3				kilograms/cubic meter
	mE/m2/s				milliEinsteins/meter squared/ second
	mg/L				milligrams/Liter
	pH units				pH units
	ppt				parts per thousand
	ug/L				micrograms/Liter
	ppm				parts per million
	uMol				microMoles
	NTU				NTU
	ppb				parts per billion
	cm2				centimeters squared
	ft				feetmS/cm @25C milliSiemens/centimeter @25C
	ng Sn/g				nannograms of tin per gram
	#/gm				number/gram
	mmol				micromolar
	cm				centimeters
	mm				millimeters
	ng/g dry wt				nannograms per gram dry wt
	ug/g dry wt				micrograms per gram dry wt
	mg/L as Si				milligrams/liter as silica
	mg/L as C				milligrams/liter as carbon
	mg/L as P				milligrams/liter as phosphorus
	mg/L as N				milligrams/liter as nitrogen
	% saturation				per cent saturation
	psu				practical salinity units
	% light				per cent light
	Kg				kilogram
	ng/wet g				nannograms per gram wet wt
	% wet				per cent wet
	Siemens/m				Siemens/meter

## Quality Assurance codes

Data values, at times, have to be qualified in order for the values to be understood or used in the appropriate manner. Data groups should define all codes listed in the data files submitted. A list of current codes and descriptions is provided. This list should be reviewed so that Quality Assurance (QA) codes and definitions listed will be used and not duplicated. QA codes should be listed in the appropriate data set to link it to the correct value.

Data Set Name: QA\_CODES    Quality Assurance Code Resolution    Variables: 3

#	Variable	Type	Len	Format	Label
1	<b>QACODE</b>	Char	6	\$15.	Quality Assurance code related to value
2	<b>QA_DESC</b>	Char	200	\$200.	Quality Assurance code description
3	<b>QA_USE</b>	Char	60	\$60.	QA code related sample type

### QA      QA      QA Code Description Code Use

#### QA Use: Sediment Toxicity Test Code (ST)

ST-A	ST	More than 20 animals inoculated into replicate.
ST-B	ST	Fewer than 4 replicates were tested.
ST-C	ST	Fewer than 5 replicates were tested.
ST-D	ST	Mean control survival was < 85 %.
ST-E	ST	Sample held for >30 days prior to testing.
ST-F	ST	Sediment too coarse to sieve through 0.5 mm mesh, therefore making it difficult to recover clams.
ST-G	ST	No reference toxicant test was run.
ST-H	ST	Hardness and alkalinity not measured.
ST-I	ST	Control survival in one replicate was <80%.
ST-J	ST	Physical parameters were out of bounds.
ST-K	ST	<20 animals used per replicate.
ST-L	ST	Not used in Province assessment.
ST-M	ST	Reduced number of replicates used.
ST-N	ST	Minor deviation in test conditions.
ST-O	ST	Control performance criteria not met.
ST-P	ST	Folly River control sediment not used. Note that this occurred only once. Sediments from Breach Inlet were used.
ST-Q	ST	Statistical analysis not run because the mean growth rate was >100% of the mean control growth rate.
ST-R	ST	Unable to calculate an EC50 value for this sample due to an insignificant decrease in luminescence or an increase in luminescence (i.e., little or no toxic effects)
ST-S	ST	Very high to complete mortality of clams in sample (i.e., sample is toxic).
ST-T	ST	Fewer than 3 replicates were tested (cadmium exposures only).
ST-U	ST	Samples were processed within 14 days of sampling.
ST-V	ST	Sample held for > 10 days prior to testing.
ST-W	ST	Unable to calculate an EC50 value for this sample due to an insignificant decrease in luminescence or an increase in luminescence (i.e., little or no toxic effects).
ST-X	ST	Calculated EC50 result was greater than the highest test concentration of 10%. Because the accuracy of an EC50 value above 10% is unknown, EC50 values greater than 10% have been reported as 10.000%.
ST-Y	ST	Hit/Miss result could not be determined due to missing silt-clay data.

**QA Use: Water Quality Measurement Code (WQ)**

WQ-A	WQ	Values estimated from another data source
WQ-B	WQ	DO value possibly as much as 0.32 low
WQ-C	WQ	DO value possibly as much as 0.54 low
WQ-D	WQ	DO value possibly as much as 0.85 low
WQ-E	WQ	DO value possibly as much as 1.3 low
WQ-F	WQ	DO value possibly as much as 1.6 low
WQ-G	WQ	DO value possibly as much as 1.5 low
WQ-H	WQ	Only surface measures taken, depth <1 m
WQ-I	WQ	Depth values questionable.
WQ-J	WQ	Fluorescence off-scale.
WQ-K	WQ	Shallow station: surface and bottom values equal. Bottom file used for both.
WQ-L	WQ	One sample was collected mid-depth due to shallow water (<3m); measurement values were reported identically for both surface and bottom layers.
WQ-M	WQ	The calculated salinity range was -0.1 to 0.1 ppt. The value is reported as zero.

**QA Use: Trawl Abundance/Biomass or Acceptability Codes (FT)**

FT-A	FT	Abundance count based on calculation of aliquot.
FT-B	FT	The species was present in the trawl, but not counted.
FT-C	FT	The species group was not weighed.
FT-D	FT	The species, taxon or group was weighed, but the weight was not detected at the minimum level of 0.1 kg; therefore, the group weighed less than 0.1 kg.
FT-E	FT	Trawl was marginally acceptable because its duration was less than the planned 10 min. As a result, observations flagged with the FT-E Trawl QC code may not truly represent the demersal community at a station, and may result in underestimation of abundance or diversity for that trawl. Data from trawls flagged with this code should be used with discretion.
FT-F	FT	Trawl was unacceptable due to reasons such as: trawl filled with algae, trawl twisted or not properly opened, large object caught in trawl, trawl fouled on bottom. These situations generally resulted in the trawl being aborted well before its planned duration was reached. Due to the problems mentioned above, any observations flagged with the FT-F Trawl QC code should not be used in data analyses.

**QA Use: Chemical Analyte Codes - Sediment and Tissue (CH)**

CH-A	CH	The CH-A code indicates that an analyte was not detected. When the CH-A code is used, the concentration field is left blank and the method detection limit for the analyte in that particular sample is reported under Detection Limit Concentration.
CH-B	CH	It is sometimes possible for a laboratory to detect an analyte and report its concentration at a level which is below the calculated method detection limit for the sample. In these situations the analyst is confident that the analyte was present in the sample, but there is a high degree of uncertainty in the reported concentration. The CH-B code is used to flag reported values which are below the calculated method detection limit for the sample. Such values are considered estimates only and should be used with discretion.
CH-C	CH	The CH-C code indicates that the laboratory experienced minor deficiencies meeting the QC requirements, but the overall data quality is judged to be reliable for EMAP assessments.
CH-D	CH	The CH-D code indicates that there was insufficient tissue in a given sample for analysis of all chemical components. In this case, only one or two groups of analytes were measured (usually metals or TBT).
CH-E	CH	Estimated quantity below reported detection limit.
CH-F	CH	Algae Present - Indicates that the presence of algae in the sample prevented accurate measurement of TOC. Samples with the CH-F code will have a missing value for TOC.
CH-G	CH	Blank Interference - Indicates that there was an interference detected in the blank which would interfere with the accurate determination of an analytes concentration. Results for observations with the CH-G code should be considered questionable and used with discretion.
CH-H	CH	Concentration is undetectable; user to decide regarding interpretation.
CH-I	CH	Some analytes are difficult to quantify because they co-elute with other closely related analytes. This phenomenon is called matrix interference. When this occurs the suspect analyte(s) are given a CH-I code and concentration is left blank.
CH-J	CH	Failed QA criteria.
CH-K	CH	A laboratory may elect to cease reporting some analytes. EMAP protocol only requires that the

laboratory analyze a given list of chemicals; when additional chemicals are analyzed and reported, they may be included in the data. The CH-K code indicates that an analyte has been excluded from a given set of data. Only unflagged or CH-E coded values are considered valid and useful for most assessment purposes.

CH-L	CH	Some of the analytes listed represent the sum of concentrations of similar analytes (e.g. PCB_TOT is the sum of the concentrations of all PCB congeners). In the event that the concentrations for all of the individual analytes included in the sum are non-detects (have CH-A code) the sum is missing. This is not technically a non-detect, but a sum of non-detects hence the CH-L code.
CH-M	CH	Dilution Required - Indicates the sample required dilution prior to analysis. This has no effect on reported concentrations and is not a problem. Values with this code can be used with no further qualification.
CH-N	CH	Field QA sample
CH-O	CH	Just Detected - Indicates that an analyte was detected in the sample, but at a concentration below the method detection limit for the sample. In these cases, you can be confident that the analyte is present in the sample, but there is a high degree of uncertainty in the reported concentration. Therefore, values flagged with the CH-O QA code should be considered estimates only, and used with discretion.
CH-P	CH	CONC is less than or equal to the MDL, but is detectable; value uncertainty.
CH-Q	CH	Matrix Interference - Indicates that the reported concentration is questionable due to interference from other compounds in the sample. Therefore, values flagged with the CH-Q QA code should be used with discretion.
CH-R	CH	Non Detect - Indicates that the concentration of an analyte was too low to detect. In these cases, the QA code of CH-R is used, and the concentration is reported as 0. Although the actual concentration is unknown (but likely very low to none), reporting a concentration of 0 serves as a place holder.
CH-S	CH	Not detected.
CH-T	CH	QA problem - Indicates cases where required quality assurance guidelines were not met by the lab. If no concentration is reported, then the QC problem was judged to be severe enough to invalidate the result for that analyte. If however a concentration is reported for an analyte with a CH-T code, then the overall data quality was judged to be reliable enough to be used with discretion.
CH-U	CH	No QA/QC samples (i.e. Certified Reference Material) exist for evaluation of accuracy of this parameter. No apparent sample corruption was evident; caution is expressed for those who wish to convert to a dry weight basis.
CH-V	CH	The reported concentration is considered an estimate because control limits for this analyte were exceeded in one or more quality control samples.
CH-W	CH	In GC_ECD dual column confirmation results from the primary and secondary columns differed by more than a factor of 3. The lower of the two is reported.

#### Water Measurement Names

Ammonium NH<sub>4</sub>  
 Chlorophyll a  
 Conductivity  
 Density  
 Depth where PAR=1% of surface PAR  
 Dissolved oxygen  
 Dissolved oxygen (saturation)  
 Fluorescence  
 Fluorescence (maximum)  
 Light extinction rate  
 Light extinction rate (avg)  
 Nitrate and nitrite  
 Orthophosphate PO<sub>4</sub>  
 Phaeophytin  
 Photosynthetically active radiation

Salinity  
 Secchi depth  
 Specific conductance  
 Temperature  
 Total dissolved nitrogen  
 Total dissolved phosphorus  
 Total particulate carbon  
 Total particulate nitrogen  
 Total particulate phosphorus  
 Total suspended solids  
 Transmissivity  
 Transmissivity @ 1m depth  
 Turbidity  
 pH

### Species Ignore Code Resolution

Ignore Code	Ignore Code Description
1	An Ignored Taxon Code of “1” identifies observations where taxon should be excluded from the calculation of taxonomic richness (total number of taxa) at a station, but not excluded from calculations of abundance. Refer to the associated metadata for a more complete discussion.
2	An Ignored Taxon Code of “2” indicates organisms that, although captured in the benthic grab, are not typically considered members of the infaunal community. Refer to the associated metadata for a more complete discussion.

### Measurement Type Code Resolution

Measurement Type	Measurement Description
F	Fork length (finfish)
T	Total length (finfish)
B	Standard length (finfish)
S	Shell length - rostrum to telson (shrimp)
C	Greatest carapace width (crabs)
D	Disk width (skates and rays)
M	Mantle length (squid)



## Appendix E. Example of Metadata File

### DRAFT

CATALOG DOCUMENTATION

MAIA-ESTUARIES SUMMARY DATABASE

1997 and 1998 STATIONS

BENTHIC SUMMARY DATA: "BENSUMRY"

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### 1. DATASET IDENTIFICATION

- 1.1 Title of Catalog document  
MAIA-Estuaries Summary Database  
1997 and 1998 Stations  
Benthic Summary Data
- 1.2 Authors of the Catalog entry  
John Kiddon, U.S. EPA NHEERL-AED  
Harry Buffum, OAO Corp.
- 1.3 Catalog revision date  
April 15, 2000
- 1.4 Dataset name  
BENSUMRY
- 1.5 Task Group  
MAIA Estuaries
- 1.6 Dataset identification code  
011
- 1.7 Version  
001
- 1.8 Request for Acknowledgment  
EMAP requests that all individuals who download EMAP data acknowledge the source of these data in any reports, papers, or presentations. If you publish these data, please include a statement similar to: "Some or all of the data described in this article were produced by the U. S. Environmental Protection Agency through its Environmental Monitoring and Assessment Program (EMAP)".

## 2. INVESTIGATOR INFORMATION (for full addresses see Section 13)

### 2.1 Principal Investigators

John Paul, U.S. Environmental Protection Agency, NHEERL-Atlantic Ecology Division (AED)  
Charles Strobel, U.S. Environmental Protection Agency, NHEERL-Atlantic Ecology Division (AED)

### 2.2 Sample Collection Investigators

Charles Strobel, U.S. Environmental Protection Agency, NHEERL-Atlantic Ecology Division (AED)  
John Macauley, U.S. Environmental Protection Agency, Gulf Ecology Division (GED)  
Jeffrey L. Hyland, National Oceanographic and Atmospheric Admin.-Carolinian Province (NOAA-DB)  
Michelle Harmon, National Oceanographic and Atmospheric Admin.-Delaware Bay (NOAA-DB)  
Carl Zimmerman, National Park Service (NPS)  
Dan Dauer, Chesapeake Bay Program, Old Dominion University (CBP-ODU)  
J. Ananda Ranasinghe, Chesapeake Bay Program, Versar, Inc. (CBP-VER)

### 2.3 Sample Processing Investigators

J. Ananda Ranasinghe, Chesapeake Bay Program, Versar, Inc. (CBP-VER)

## 3. DATASET ABSTRACT

### 3.1 Abstract of the Dataset

The BENSUMRY file presents a summary of selected benthic abundance and biomass data that was collected in MAIA estuaries during the Summers of 1997 and 1998. Seventeen summary parameters are reported for each sampling event at a station. The parameters include the mean abundances per grab of infaunal species, epifaunal species, spionid polychaetes, and tubificid oligochaetes (calculated separately); the mean biomass per grab of all species; the total and mean numbers per grab of infaunal species and epifaunal species (calculated separately); and three indices characterizing the environmental condition at the site: the Shannon-Weiner, Gleason's D, and EMAP VA Province Benthic indices. One record is presented for each site visit. The complete records of benthic abundance and biomass data are contained in the BEN\_ABUN and BEN\_BIOM files, respectively.

### 3.2 Keywords for the Dataset

Benthic species, invertebrates, epifaunal, infaunal, spionid polychaetes, tubificid oligochaetes, Shannon-Weiner, Gleason's D, EMAP VA Province Benthic Index, mean abundance per grab, mean biomass per grab

## 4. OBJECTIVES AND INTRODUCTION

### 4.1 Program Objective

The main objectives of the MAIA-Estuaries program are: (1) to evaluate the ecological condition of the Mid-Atlantic estuaries by measuring key properties of the water, sediment, and the community of organisms; (2) to focus attention on small estuaries in order to develop better monitoring approaches for these critical systems; and (3) to develop partnerships among federal and state environmental organizations.

The Environmental Monitoring and Assessment Program (EMAP) is an EPA research and monitoring program designed to provide unbiased assessments of the condition of selected resources over a wide region. A key feature of the program is a probabilistic sampling strategy that randomly selects sampling sites and assigns weighting factors based on area to all measured results. EMAP's strategy was adopted by the Mid-Atlantic Integrated Assessment (MAIA) program, which was designed to assess the conditions of the estuaries, forests, streams and lakes, and agricultural lands in the seven-state Mid-Atlantic region. This file contains data measured in MAIA estuaries during the Summers of 1997 and 1998. Samples were collected for water and sediment analyses primarily in 1997, with a few additional sites sampled in 1998. Fish samples were collected from comparable but not identical sites in 1998. Thus, although data were collected from two years, the dataset does not characterize 97/98 interannual variability. Several estuaries were designated as intensive sites and were subjected to a spatially intensive sampling scheme (see STATIONS file).

The partners in MAIA-Estuaries program are: (1) The U.S. Environmental Protection Agency (USEPA), including both the Atlantic Ecology Division (AED) and the Gulf Ecology Division (GED); (2) National Park Service (NPS) under their project “Maryland Coastal Bays Monitoring”; (3) National Oceanographic and Atmospheric Administration (NOAA) which conducts sampling both in the Delaware Bay (DB) under their “National Status and Trends Program” and in the Carolinian Province (CP); and (4) The Chesapeake Bay Program (CBP), which is a consortium of federal, state, and local governments and nongovernmental organizations. Each partner was responsible for collecting, processing, and reviewing data. The USEPA Atlantic Ecology Division was responsible for final assembly and review of all data. Laboratories contracted to process samples are specified by the parameter LABCODE included in all data files (Section 4.4). Details regarding use of partner and LABCODE information and are presented in the EVENTS metadata file.

#### 4.2 Dataset Objective

This file presents summary parameters and indices calculated from benthic abundance and biomass data collected in MAIA estuaries in during the Summers of 1997 and 1998.

#### 4.3 Dataset Background Discussion

The data files BEN\_ABUN and BEN\_BIOM contain extensive records reporting the abundance and biomass of benthic invertebrate organisms in MAIA sediments. However, it is often useful to summarize some of this information to aid in its interpretation. This file reports several simple averages of abundance and biomass data, as well as three indices that express the diversity or richness of species in a community. Infaunal species refer to organisms living within the sediments. Epifaunal organisms live at the sediment/water interface. The summary parameters include the mean abundances per grab of infaunal species, epifaunal species, spionid polychaetes, and tubificid oligochaetes (calculated separately); the mean biomass per grab of all species; and the total and mean numbers per grab of infaunal species and epifaunal species (calculated separately). The three indices are the Shannon-Weiner index, Gleason’s D index, and the EMAP VA Province Benthic index. The expressions used to calculate these indices are presented in Section 6.2.

The Shannon-Weiner index,  $H'$ , is a standard measure of species diversity that ranges from zero to positive values, representing progressively increasing diversity (Krebs, 1989). Gleason’s D index is an expression of species richness, also ranging from near zero to positive values, with larger values signifying greater richness. The EMAP Virginian Province Benthic Index is a combination of three metrics into a single index (the metrics are: salinity-adjusted Gleason’s index, the salinity-adjusted abundance of tubificids, and the abundance of spionids). This Benthic Index was developed with data compiled during the 1990-1993 EMAP effort in the Virginian Province (Paul *et al.* 1999). The majority of values range from -5 to +5, with positive values signifying healthy conditions and negative values indicating probable impairment.

#### 4.4 Summary of Dataset Parameters

*STATION	Station name
*EVNTDATE	Event date
A_SAMPS	Number of grabs with abundance data
INF_ABU	Mean abundance per grab, all infauna
EPI_ABU	Mean abundance per grab, all epifauna
SPIONID	Spionid polychaetes (infaunal species only), mean abundance/grab
TUBIFIC	Tubificid oligochaetes, mean abundance/grab
B_SAMPS	Number of grabs with biomass data
MN_BIOM	Mean biomass per grab, all species
TSINFCNT	Total number of infaunal species
TSEPICNT	Total number of epifaunal species
MSINFCNT	Mean number of infaunal species per grab
MSEPICNT	Mean number of epifaunal species per grab
SHANNON3	Shannon-Wiener Index - all species
GLEASON3	Gleason’s D - all species
BOT_SAL	Bottom salinity used in calculating benthic index. Some missing values in Delaware Bay were interpolated from data at neighboring sites. The interpolated data are denoted with a

	QACODE = BI-A.
PEXP_GL3	Percent expected Gleason's D
PEXP_TUB	Expected tubificid abundance
B_IND94B	EMAP VA province benthic index
QACODE	QA qualifier
	<blank> No qualification
BI-A	Salinity values used in calculating Benthic Index are interpolated
LABCODE	Contract / lab identifier
BEN-1	USEPA contractor: Versar, Inc.
BEN-2	NOAA Carolinian Province contractor
BEN-3	Chesapeake Bay Program contractor: Versar, Inc.
BEN-4	NOAA Delaware Bay contractor
YEAR	Year of Sampling: 1997 or 1998

\* denotes parameters that should be used as key fields when merging data files

## 5. DATA ACQUISITION AND PROCESSING METHODS

All values in this data file were calculated from data presented in the BEN\_ABUN and BEN\_BIOM data files. Refer to the metadata for those files for details regarding sampling and processing methods.

## 6. DATA ANALYSIS AND MANIPULATIONS

### 6.1 Name of New or Modified Values

SHANNON3	Shannon-Wiener Index - all species
GLEASON3	Gleason's D - all species
PEXP_GL3	Percent expected Gleason's D
PEXP_TUB	Expected tubificid abundance
B_IND94B	EMAP VA province benthic index

### 6.2 Data Manipulation Description

The Shannon-Wiener Index, SHANNON3, was calculated as:

$$H' = -\sum P_i \log_{10}(P_i)$$

where  $P_i$  is the fraction of the total abundance attributed to the  $i$ th species, and  $\log_{10}$  denotes log base 10. All species reported at a station (infaunal and epifaunal) were included.

The Gleason's D Index, GLEASON3, for infaunal and epifaunal species was calculated as:

$$D = (\text{total \# species}) / (\text{natural log of total abundance})$$

All species reported at a station were included.

The salinity-normalized Gleason's Index was calculated as the ratio of the *measured* and *expected* Gleason's D indices, reported as a percent. The expected index is calculated with a polynomial expression describing the of the upper boundary (90th percentile) of index values vs salinity data (Paul *et al.*, 1999):

$$\text{PEXP\_GL3} = \text{GLEASON3} / (4.283 - 0.498 * \text{sal} + 0.0542 * \text{sal}^2 - 0.00103 * \text{sal}^3) * 100$$

where 'sal' is the bottom water salinity.

The salinity-adjusted tubificid abundance was calculated as:

$$\text{PEXP\_TUB} = \text{measured tubificid abundance} - 500 * \exp(-15 * \text{sal})$$

where 'sal' is the bottom water salinity.

The EMAP VA Province Benthic Index, B\_IND94B, was developed as described by Paul *et al.*, (1999).

The coefficients of the expression differ depending on the number of grabs analyzed at a station. Where one grab sample was analyzed, the benthic index (BI) was calculated as:

$$\text{BI} = 1.389 * (\text{PEXP\_GL3} - 51.5) / 28.4 - 0.651 * (\text{PEXP\_TUB} - 28.2) / 119.5 - .375 * (\text{spionid abundance} - 20.0) / 45.4;$$

Where either 2 or 3 grabs samples were analyzed, the BI was calculated as :

$$\text{BI} = 1.246 * (\text{PEXP\_GL3} - 40.5) / 25.3 - 0.555 * (\text{PEXP\_TUB} - 29.1) / 124.7 - .344 * (\text{spionid abundance} - 20.0) / 52.0;$$

PEXP\_GL3 is the salinity-normalized Gleason's D index, and PEXP\_TUB is the salinity-adjusted tubificid abundance.

## 7. DATA DESCRIPTION

### 7.1 Description of Parameters

#### 7.1.1 Components of the Dataset

STATION	Station name
EVNTDATE	Event date
A_SAMPS	Number of grabs with abundance data
INF_ABU	Mean abundance per grab, all infauna
EPI_ABU	Mean abundance per grab, all epifauna
SPIONID	Spionid polychaetes (infaunal species only), mean abundance/grab
TUBIFIC	Tubificid oligochaetes, mean abundance/grab
B_SAMPS	Number of grabs with biomass data
MN_BIOM	Mean biomass per grab, all species
TSINFCNT	Total number of infaunal species
TSEPICNT	Total number of epifaunal species
MSINFCNT	Mean number of infaunal species per grab
MSEPICNT	Mean number of epifaunal species per grab
SHANNON3	Shannon-Wiener Index - all species
GLEASON3	Gleason's D - all species
BOT_SAL	Bottom water salinity
PEXP_GL3	Percent expected Gleason's D
PEXP_TUB	Expected tubificid abundance
B_IND94B	EMAP VA province benthic index
QACODE	QA qualifier
LABCODE	Contract / lab identifier
YEAR	Year of sampling

#### 7.1.2 Precision to which values are reported

PARAMETER	PRECISION	MIN	MAX	UNITS
A_SAMPS	unit	1	3	number of grabs
INF_ABU	0.1	0	2720	organisms per grab
EPI_ABU	0.1	0	928	organisms per grab
SPIONID	0.1	0	296	organisms per grab
TUBIFIC	0.1	0	1860	organisms per grab
B_SAMPS	unit	1	3	number of grabs
MN_BIOM	0.0001	0	10.8	gram per grab
TSINFCNT	unit	0	55	number of species
TSEPICNT	unit	0	33	number of species
MSINFCNT	0.1	0	35	species per grab
MSEPICNT	0.1	0	19.5	species per grab
SHANNON3	0.001	0	1.42	no units
GLEASON3	0.01	0	11.5	number of species
BOT_SAL	0.1	0	35	ppt
PEXP_GL3	0.1	0	124	percent
PEXP_TUB	0.1	-493	1860	organisms per grab
B_IND94B	0.01	-8.87	5.01	no units

7.1.3 Minimum Value in Dataset  
See Section 7.1.2.

7.1.4 Maximum Value in Dataset  
See Section 7.1.2.

7.2 Data Record Example

7.2.1 Column Names for Example Record  
See Section 7.2.2.

7.2.2 Example Data Records

STATION	EVNTDATE	A_SAMPS	INF_ABU	EPI_ABU	SPIONID	TUBIFIC
MA97-0001	8/25/97	2	141.5	1.0	52.0	0.0
MA97-0003	8/26/97	2	58.0	6.5	11.5	0.0
MA97-0004	8/26/97	2	59.5	4.5	7.5	0.0
MA97-0005	8/27/97	2	136.0	6.0	10.5	0.0

B_SAMPS	MN_BIOM	TSINFCNT	TSEPICNT	MSINFCNT	MSEPICNT	SHANNON3
2	0.1051	16	1	12.0	0.5	0.788
2	0.0623	26	6	16.5	3.0	1.305
2	0.0307	14	1	10.5	1.0	0.944
2	0.0445	27	6	18.5	3.0	1.034

GLEASON3	BOT_SAL	PEXP_GL3	PEXP_TUB	B_IND94B	QACODE	YEAR
3.01	30.1	29.2	0.0	-1.20		1997
6.58	27.2	65.1	0.0	0.89		1997
3.09	26.0	31.3	0.0	-0.73		1997
5.84	28.3	57.0	0.0	0.50		1997



## 8. GEOGRAPHIC AND SPATIAL INFORMATION

8.1 Minimum Longitude (Westernmost)  
-77.4339 decimal degrees

8.2 Maximum Longitude (Easternmost)  
-74.7230 decimal degrees

8.3 Minimum Latitude (Southernmost)  
34.9670 decimal degrees

8.4 Maximum Latitude (Northernmost)  
40.1470 decimal degrees

8.5 Name of area or region  
MAIA estuary region, consisting of Delaware Bay, Chesapeake Bay, the Delmarva coastal bays, Albemarle-Pamlico Sound, and contiguous estuaries.

## 9. QUALITY CONTROL AND QUALITY ASSURANCE

All values in this data file were calculated from data presented in the BEN\_ABUN and BEN\_BIOM data files. Refer to the metadata for those files for details regarding sampling and processing methods.

9.1 Measurement Quality Objectives  
Not applicable

9.2 Data Quality Assurance Procedures  
Not applicable

9.3 Actual Measurement Quality  
Not applicable

## 10. DATA ACCESS

10.1 Data Access Procedures  
Data can be downloaded from the web

10.2 Data Access Restrictions  
None

10.3 Data Access Contact Persons  
John Paul, Principal Investigator  
U.S. EPA NHEERL-AED  
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10.4 Dataset Format  
ASCII (CSV) and SAS Export files

10.5 Information Concerning Anonymous FTP  
Not available

10.6 Information Concerning WWW  
No gopher access, see Section 10.1 for WWW access

10.7 EMAP CD-ROM Containing the Dataset  
Data not available on CD-ROM

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Strobel, C.J. 1998. Mid Atlantic Integrated Assessment / Environmental Monitoring and Assessment Program - Estuaries: Virginian Province Quality Assurance Project Plan. U.S. EPA, Office of Research and Development, NHEERL-AED, Narragansett, RI. June 1998.

## 12. TABLE OF ACRONYMS

AED	Atlantic Ecology Division
BI	Benthic Index
CP	Carolinian Province
CBP	Chesapeake Bay Program
D	Gleason's D Index
DB	Delaware Bay
EMAP	Environmental Monitoring and Assessment Program
EPA	U.S. Environmental Protection Agency
GED	Gulf Ecology Division
GERG	Geochemical and Environmental Research Group
H'	Shannons-Weiner Index
MAIA	Mid-Atlantic Integrated Assessment
NHEERL	National Health and Environmental Effects Research Laboratory
NOAA	National Oceanic and Atmospheric Administration
NOS	National Ocean Service
NPS	National Park Service
ODU	Old Dominion University
ORCA	Office of Ocean Resources Conservation and Assessment
ORD	Office of Research and Development
QA/QC	Quality Assurance/Quality Control
TAMU	Texas A&M University
TOC	Total Organic Carbon
USEPA	United States Environmental Protection Agency
VER	Versar, Inc.
WWW	World Wide Web

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## APPENDIX C

### West EMAP Information Management Plan For 2000

(Available in electronic format upon request; contact Larry Cooper: [larryc@sccwrp.org](mailto:larryc@sccwrp.org))

## APPENDIX D

### Methods: Non-Acidification Analysis for Chlorophyll *a*



## TURNER DESIGNS

845 W Maude Ave., Sunnyvale, CA 94086  
(408) 749-0994 FAX (408) 749-0998

### USING THE TURNER DESIGNS MODEL 10 ANALOG, THE 10-AU DIGITAL, OR THE TD-700 FLUOROMETER WITH EPA METHOD 445.0:

#### **“In Vitro Determination of Chlorophyll *a* and Pheophytin *a* in Marine and Freshwater Phytoplankton by Fluorescence”<sup>1</sup>**

by Elizabeth J. Arar & Gary B. Collins

The United States Environmental Protection Agency (EPA) has recently published a chlorophyll method, Method 445.0. Method 445.0 describes the use of a Turner Designs Model 10 Series Fluorometer (Section 6.1).<sup>2</sup> This Fluorometer has been redesigned to make it easier to use. It is now called the Turner Designs Model 1-AU Fluorometer. The Model 10-AU is digital and is capable of performing calculations formerly done by the user. The TD-700, Turner Design's newest fluorometer, is also capable of performing these calculations.

In addition, there is a new method<sup>3</sup> for measuring chlorophyll *a* in the presence of chlorophyll *b* and pheopigments, which does NOT require the acidification step of conventional fluorescence techniques. Conventional fluorescence methods for measuring chlorophyll *a* require samples to be measured twice; once before acidification and once afterwards. Under the most extreme ratio of chlorophyll *a*/chlorophyll *b* likely to occur in nature (1:1 molar), conventional acidification techniques results in approximately a 60% underestimate of chlorophyll *a*. In these conditions the new method yields only a 10% overestimate of true chlorophyll *a*. It requires only a single fluorescence reading and is sensitive enough for estimates of euphotic zone chlorophyll *a* in all marine and freshwater ecosystems.

**CONFIGURING THE TURNER DESIGNS MODEL 10-AU DIGITAL OR THE MODEL 10 ANALOG FLUOROMETER FOR METHOD 445.0.** Your fluorometer should be equipped with the following Turner Designs optical filter kit (or equivalent):

Optical Kit:	PN: 10-037 or 10-037R
Lamp:	10-045 Daylight White Lamp
Excitation filter:	10-050 or 10-050R color specification 5-60
Emission filter:	10-051 or 10-051R color specification 2-64
Reference filter:	10-032 1 neutral density (1 ND), <u>or</u> the 10-035 2 neutral density (2 ND), <u>or</u> the 10-052 color specification 3-66

**CONFIGURING THE TURNER DESIGNS MODEL TD-700 FLUOROMETER FOR METHOD 445.0.** Your fluorometer should be equipped with the following Turner Designs optical filter kit (or equivalent):

Optical Kit:	PN: 7000-961
Lamp:	10-045 Daylight White Lamp
Excitation filter:	10-050R color specification 5-60
Emission filter:	10-051R color specification 2-64

## USING YOUR TURNER DESIGNS FLUOROMETER WITH METHOD 445.0.

Section 10.1 (Calibration and Standardization) of Method 445.0: If you are using a digital fluorometer such as the 10-AU or the TD-700, you no longer need to calculate  $F_s$ . the calibration instructions in your fluorometer user's manual and the instrument will give you direct readout of the concentration of the standard and samples without the need for compensation for the various sensitivity settings. If you are using the model 10 analog, you must perform the calculations in this section.

In Section 12.0 (Data Analysis and Calculations), when the Model 10-AU and the TD-700 are properly calibrated with a known standard,  $F_s$  always equals 1 (in the formulas in Section 12.1). The Model 10-AU and the TD-700 do the range and sensitivity setting calculations for you, so it is not necessary to calculate  $F_s$ . If you are using the model 10 analog, you must perform the calculations in this section.

## CONFIGURING THE TURNER DESIGNS MODEL 10-AU DIGITAL OR THE MODEL 10 ANALOG FLUOROMETER FOR THE NEW CHLOROPHYLL *a* METHOD.\*\*

Optical Kit:	PN: 10-040 or 10-040R
Lamp:	10-045 Daylight White Lamp
Excitation filter:	10-113 (436 nm)
Emission filter:	10-115 (680 nm)
Reference filter:	10-035 2 neutral density (2 ND)

## CONFIGURING THE TURNER DESIGNS MODEL TD-700 FLUOROMETER FOR THE NEW CHLOROPHYLL *a* METHOD\*\*.

Optical Kit:	PN: 7000-962
Lamp:	10-045 Daylight White Lamp
Excitation filter:	10-113 (436 nm)
Emission filter:	10-115 (680 nm)

\*\*The new chlorophyll *a* method requires that your fluorometer be equipped with a special optical filter kit, which will read chlorophyll *a* in the presence of chlorophyll *b* and pheopigments. Your fluorometer should be equipped with the above Turner Designs optical filter kit (or equivalent).

## USING YOUR TURNER DESIGNS FLUOROMETER WITH THE NEW CHLOROPHYLL *a* METHOD.

For this procedure, follow the instructions in the Turner Designs procedure (P/N 998-9000), "Measuring Extracted Chlorophyll *a* Free from the Errors Associated with Chlorophyll *b* and Pheopigments." Procedures in Method 445.0 apply generally, **EXCEPT** you must **NOT** acidify your samples as set forth in Method 445.0, section 11.2.2. It is not necessary, as the special optical filter set up is designed to read **ONLY** chlorophyll *a* and **NOT** chlorophyll *b* and the pheopigments. It is not necessary to perform calculations set forth in Section 12.1 of Method 445.0. When properly calibrated with a known concentration of pure chlorophyll *a*, the sample reading without acidification represents the actual proportion of chlorophyll *a* relative to the standard.

## References

1. To obtain copies of the complete EPA standard methods book, *Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Samples*, call the EPA in Cincinnati, Ohio at (513)569-7562. The book contains Method 445.0 and several other useful procedures. Ask for item EPA/600/R-92/121.

2. A note about optics. For chlorophyll studies according to Method 445.0, the Turner Designs Model 10-AU comes equipped with an excitation filter equivalent to the CS 5-60 excitation filter and an emission filter equivalent to the CS 2-64 emission filter (see Section 6.1 of Method 445.0). We supply the F4T5D daylight white lamp.
3. The method was developed by Dr. Nicholas A. Welschmeyer of Moss Landing Marine Laboratories, Moss Landing, California. A paper by Dr. Welschmeyer, Fluorometer Analysis of Chlorophyll *a* in the presence of Chlorophyll *b* and Pheopigments, can be found in *Limnology and Oceanography* (1994) **39**: 1985-1992.

**METHOD 445.0****IN VITRO DETERMINATION OF CHLOROPHYLL *a* AND PHEOPHYTIN *a* IN MARINE  
AND FRESHWATER PHYTOPLANKTON BY FLUORESCENCE**

Adapted by  
Elizabeth J. Arar  
and  
Gary B. Collins

VERSION 1.1  
November 1992

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY  
OFFICE OF RESEARCH AND DEVELOPMENT  
U.S. ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OH 45268

Method 445.0 was reprinted from USEPA *Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Samples*. To obtain copies of the complete EPA standard methods book, call the EPA in Cincinnati, Ohio at (513)569-7562. The book contains Method 445.0 and several other useful procedures. Ask for item EPA/600/R-92/121.

**METHOD 445.0****IN VITRO DETERMINATION OF CHLOROPHYLL *a* AND PHEOPHYTIN *a* IN MARINE  
AND FRESHWATER PHYTOPLANKTON BY FLUORESCENCE****TABLE OF CONTENTS**

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## METHOD 445.0

### IN VITRO DETERMINATION OF CHLOROPHYLL *a* AND PHEOPHYTIN *a* IN MARINE AND FRESHWATER PHYTOPLANKTON BY FLUORESCENCE

#### 1.0 SCOPE AND APPLICATION

- 1.1 This method provides a procedure for determination of low level chlorophyll *a* (chl *a*) and its magnesium-free derivative, pheophytin (pheog *a*), in marine and freshwater phytoplankton using fluorescence detection.<sup>(1,2)</sup> Pheophorbides present in the sample are determined collectively as pheophytin *a*.

<u>ANALYTE</u>	<u>CHEMICAL ABSTRACTS SERVICE REGISTRY NUMBER (CASRN)</u>
Chlorophyll <i>a</i>	479-61-8

- 1.2 Instrumental detection limits of .05  $\mu\text{g}$  chl *a*/L and .06  $\mu\text{g}$  pheog *a*/L in a solution of 90% acetone were determined by this laboratory. Method detection limits using mixed assemblages of algae provide little information because of interference of other pigments in the fluorescence of chlorophyll *a* and pheophytin *a*.<sup>(3)</sup> An estimated detection limit for chlorophyll *a* was determined to be 0.11  $\mu\text{g}$ /L in 10 mL of final extraction solution. The upper limit of the linear dynamic range for the instrumentation used in this method evaluation was 250  $\mu\text{g}$  chl *a*/L.
- 1.3 This method uses 90% acetone as the extraction solvent because of its efficiency for most types of algae. There is evidence that certain chlorophylls and carotenoids are more thoroughly extracted with methanol(4-6) or dimethyl sulfoxide.(7) Bowles, et al. (6) found that for chlorophyll *a*, however, 90% acetone was an effective extractant when the extraction period was optimized for the dominant species present in the sample.
- 1.4 Depending on the type of algae under investigation, this method can have uncorrectable interferences (Sect. 4.0). In cases where taxonomic classification is unavailable, a spectrophotometric or high performance liquid chromatographic (HPLC) method may provide more accurate data for chlorophyll *a* and pheophytin *a*.
- 1.5 This method is for use by analysts experienced in the handling of photosynthetic pigments and in the operation of fluorescence detectors or by analysts under the close supervision of such qualified persons.

#### 2.0 SUMMARY OF METHOD

- 2.1 Chlorophyll-containing phytoplankton in a measured volume of sample water are concentrated by filtering at low vacuum through a glass fiber filter. The pigments are extracted from the phytoplankton in 0% acetone with the aid of a mechanical tissue grinder and allowed to steep for a minimum of 2 h, but not to exceed 24 h, to ensure thorough extraction of the chlorophyll *a*. The filter slurry is centrifuged at 675 g for 15 min. (or at 1000 g for 5 min) to clarify the solution. An aliquot of the supernatant is transferred to a glass cuvette and



fluorescence is measured before and after acidification to 0.003 N HCl with 0.1 N HCl. Sensitivity calibration factors, which have been previously determined on solutions of pure chlorophyll *a* of known concentration, are used to calculate the concentration of chlorophyll *a* and pheophytin *a* in the sample extract. The concentration in the natural water sample is reported in  $\mu\text{g/L}$ .

### 3.0 DEFINITIONS

- 3.1 ESTIMATED DETECTION LIMIT (EDL) – The minimum concentration of an analyte that yields a fluorescence 3X the fluorescence of blank filters which have been extracted according to this method.
- 3.2 LINEAR DYNAMIC RANGE (LDR) – The absolute quantity or concentration range over which the instrument response to an analyte is linear.
- 3.3 INSTRUMENT DETECTION LIMIT (IDL) – The minimum quantity of analyte or the concentration equivalent which gives an analyte signal equal to three times the standard deviation of the background signal at the selected wavelength, mass, retention time, absorbance line, etc. For this method the background is a solution of 90% acetone.
- 3.4 STOCK STANDARD SOLUTION (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.
- 3.5 PRIMARY DILUTION STANDARD SOLUTION (PDS) – A solution of the analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 3.6 CALIBRATION STANDARD (CAL) – A solution prepared from the primary dilution standard solution or stock standard solutions containing the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.7 RESPONSE FACTOR (RF) – The ratio of the response of the instrument to a known amount of analyte.
- 3.8 LABORATORY REAGENT BLANK (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.
- 3.9 FIELD DUPLICATES (FD1 AND FD2) – Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 3.10 QUALITY CONTROL SAMPLE (QCS) – A solution of method analytes of known concentrations which is used to fortify and aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

- 3.11 MATERIAL SAFETY DATA SHEET (MSDS) – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

#### 4.0 INTERFERENCES

- 4.1 Any substance extracted from the filter or acquired from laboratory contamination that fluoresces in the red region of the spectrum may interfere in the accurate measurement of both chlorophyll *a* and pheophytin *a*.
- 4.2 The relative amounts of chlorophylls *a*, *b*, and *c* vary with the taxonomic composition of the phytoplankton. Chlorophylls *b* and *c* may significantly interfere with chlorophyll *a* measurements depending on the amount present. Due to the spectral overlap of chlorophyll *b* with pheophytin *a* and chlorophyll *a*, underestimation of chlorophyll *a* occurs accompanied by overestimation of pheophytin *a* when chlorophyll *b* is present in the sample. The degree of interference depends upon the ratio of *a*:*b*. This laboratory found that at a ratio of 5:1, using the acidification procedure to correct for pheophytin *a*, chlorophyll *a* was underestimated by approximately 5%. Loftis and Carpenter<sup>(8)</sup> reported an underestimation of 16% when the *a*:*b* ratio was 2.5:1. A ratio of 2:1 is the highest ratio likely to occur in nature. They also reported overestimation of chlorophyll *a* in the presence of chlorophyll *c* of as much as 10% when the *a*:*c* ratio was 1:1 (the theoretical maximum likely to occur in nature). The presence of chlorophyll *c* also causes the underestimation of pheophytin *a*. The effect of chlorophyll *c* is not as severe as the effect of chlorophyll *b* on the measurement of chlorophyll *a* and pheophytin *a*. Knowledge of the taxonomy of the algae under consideration will aid in determining if the spectrophotometric method using trichromatic equations to determine chlorophyll *a*, *b*, and *c* or an HPLC method would be more appropriate.<sup>(9-14)</sup>
- 4.3 Quenching effects are observed in highly concentrated solutions or in the presence of high concentrations of other chlorophylls or carotenoids. Minimum sensitivity settings on the fluorometer should be avoided; samples should be diluted instead.
- 4.4 Fluorescence is temperature dependent with higher sensitivity occurring at lower temperatures. Samples, standards, LRBs, and QCSs, must be at the same temperature to prevent errors and/or low precision. Analyses of samples at ambient temperature is recommended in this method. Ambient temperature should not fluctuate more than  $\pm 3^{\circ}\text{C}$  between calibrations or recalibration of the fluorometer will be necessary.
- 4.5 Samples must be clarified by centrifugation prior to analysis.
- 4.6 All photosynthetic pigments are light and temperature sensitive. Work must be performed in subdued light and all standards, QC materials and filter samples must be stored in the dark at  $-20^{\circ}\text{C}$  to prevent degradation.

#### 5.0 SAFETY

- 5.1 The toxicity or carcinogenicity of the chemicals used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and handled with caution and respect. Each laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method.<sup>(15-18)</sup> A file of MSDS should also be made

available to all personnel involved in the chemical analysis.

- 5.2 The grinding of filters during the extraction step of this method should be conducted in a fume hood due to the volatilization of acetone by the tissue grinder.

## 6.0 APPARATUS AND EQUIPMENT

- 6.1 Fluorometer – Equipped with a high intensity F4TS blue lamp, red-sensitive photomultiplier, and filters for excitation (CS-5-60) and emission (CS-2-64), or equivalent. (The F4T5D daylight white lamp would be an acceptable substitute for the F4T5 blue lamp.) A Turner Designs Model 10 Series fluorometer was used in the evaluation of this method.
- 6.2 Centrifuge, capable of 675 g.
- 6.3 Tissue grinder, Teflon pestle (50 mm x 20 mm) with grooves in the tip with ¼” stainless steel rod long enough to chuck onto a suitable drive motor and 30-mL capacity glass grinding tube.
- 6.4 Precombusted filters, glass fiber, 47-mm, nominal pore size of 0.45 or 0.7  $\mu$ m. Whatman GF/F filters were used in this work.
- 6.5 Petri dishes, plastic, 50 x 9-mm, or some other solid container for transporting and storing sampled filters.
- 6.6 Aluminum foil.
- 6.7 Laboratory tissues.
- 6.8 Tweezers or flat-tipped forceps.
- 6.9 Vacuum pump or source capable of maintaining a vacuum up to 6 in. Hg.
- 6.10 Room thermometer.
- 6.11 LABWARE – All reusable labware (glass, polyethylene, Teflon, etc.) that comes in contact with chlorophyll solutions should be clean and acid free. An acceptable cleaning procedure is soaking for 4 h in laboratory grade detergent and water, rinsing with tap water, distilled deionized water and acetone.
- 6.11.1 Assorted Class A calibrated pipets.
- 6.11.2 Graduated cylinders, 500-mL and 1-L.
- 6.11.3 Volumetric flasks, Class A calibrated, 25-mL, 50-mL, 100-mL and 1-L capacity.
- 6.11.4 Glass rods.
- 6.11.5 Pasteur Type pipet or medicine dropper.
- 6.11.6 Disposable glass cuvettes for the fluorometer.
- 6.11.7 Filtration apparatus consisting of 1 or 2-L filtration flask, 47-mm fritted glass disk

base and a glass filter tower.

6.11.8 Centrifuge tubes, polypropylene or glass, 15-mL capacity with nonpigmented screw-caps.

6.11.9 Polyethylene squirt bottles.

## 7.0 REAGENTS AND STANDARDS

- 7.1 Acetone, HPLC grade, (CASRN 67-6401).
- 7.2 Hydrochloric acid (HCl), concentrated (sp. gr. 1.19), (CASRN 7647-01-0).
- 7.3 Magnesium carbonate ( $\text{MgCO}_3$ ), light powder (CASRN 39409-82-0).
- 7.4 Chlorophyll *a* free of chlorophyll *b*. May be obtained from a commercial supplier such as Sigma Chemical (St. Louis, MO).
- 7.5 WATER – ASTM Type I water (ASTM D1193) is required. Suitable water may be obtained by passing distilled water through a mixed bed of anion and cation exchange resins.
- 7.6 0.1 N HCl SOLUTION – Add 8.5 mL of concentrated HCl to approximately 500 mL water and dilute to 1 L.
- 7.7 SATURATED MAGNESIUM CARBONATE SOLUTION – Add 10 g  $\text{MgCO}_3$  powder to a 1-L flask and dilute to volume with water (Sect. 7.5). Cap the flask and invert it several times. Let the suspended powder settle before using the solution in subsequent work.
- 7.8 AQUEOUS ACETONE SOLUTION – 90% acetone/10% saturated magnesium carbonate solution. Carefully measure 100 mL of the saturated magnesium carbonate solution into the 1-L graduated cylinder. Transfer to a 1-L flask or storage bottle. Measure 900 mL of acetone into the graduated cylinder and transfer to the flask or bottle containing the saturated magnesium carbonate solution. Mix, label and store.
- 7.9 CHLOROPHYLL STOCK STANDARD SOLUTION (SSS) – Chlorophyll *a* from a commercial supplier will be shipped in an amber glass ampoule which has been flame sealed. This dry standard should be stored at  $-20^\circ\text{C}$  in the dark and the SSS prepared just prior to use. Tap the ampoule until all the dried chlorophyll is in the bottom of the ampoule. In subdued light, carefully break the tip of the ampoule. Weight the ampoule and its contents to the nearest .1 mg. Transfer the entire contents of the ampoule into a 50-mL volumetric flask and reweigh the empty ampoule. Determine by difference the mass of chlorophyll *a* added to the flask. Dilute to volume with 90% acetone, determine the concentration in mg/L (1 mg in 50 mL = 20 mg/L), label the flask and wrap with aluminum foil to protect from light. The concentration of the solution must be confirmed spectrophotometrically using a multi-wavelength spectrophotometer. (9) When stored at  $-20^\circ\text{C}$ , the SSS is stable for months. However, confirmation of the SSS chlorophyll *a* concentration spectrophotometrically is required each time dilutions are made from the SSS.
- 7.10 LABORATORY REAGENT BLANK (LRB) – A blank filter which is extracted and analyzed just as a sample filter. The LRB should be the last filter extracted of a sample set. It is used to assess possible contamination of the reagents or apparatus.

- 7.11 CHLOROPHYLL *a* PRIMARY DILUTION STANDARD SOLUTION (PDS) – Add 1 mL of the SSS (Sect. 7.8) to a clean 100-mL flask and dilute to volume with the aqueous acetone solution (Sect. 7.7). If exactly 1 mg of pure chlorophyll *a* was used to prepare the SSS, the concentration of the PDS is 200  $\mu\text{g/L}$ . Prepare fresh just prior to use.
- 7.12 QUALITY CONTROL SAMPLE (QCS) – Chlorophyll *a* QCSs can be obtained from the Quality Assurance Research Division, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. QCSs are supplied with a calibration solution.

## 8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 Water Sample Collection – Water may be obtained by a pump or grab sampler. Data quality objectives will determine the depth at which samples are taken. Healthy phytoplankton, however, are generally obtained from the photic zone (depth at which the illumination level is 1% of surface illumination). Enough water should be collected to concentrate phytoplankton on at least three filters. Filtration volume size will depend on the particulate load of the water. Four liters may be required for open ocean water where phytoplankton density is usually low, whereas 1 L or less is generally sufficient for lake, bay or estuary water. All apparatus should be clean and acid free. Filtering should be performed in subdued light as soon as possible after sampling. Aboard ship filtration is highly recommended.

Assemble the filtration apparatus and attach the vacuum source with vacuum gauge and regulator. Vacuum filtration should not exceed 5 in. Hg (20 kPa). Higher filtration pressures may damage cells and result in loss of chlorophyll.

Prior to drawing a subsample from the water sample container, thoroughly shake the container to suspend the particulates. Pour the subsample into a graduated cylinder and accurately measure the volume. Pour the subsample into the filter tower of the filtration apparatus and apply a vacuum (not to exceed 20 kPa). A sufficient volume has been filtered when a visible green or brown color is apparent on the filter. Do not suck the filter dry with the vacuum; instead slowly release the vacuum as the final volume approaches the level of the filter and completely release the vacuum as the last bit of water is pulled through the filter. Remove the filter from the fritted base with tweezers, fold once with the particulate matter inside, lightly blot the filter with a tissue to remove excess moisture and place it in the petri dish or other suitable container. If the filter will not be immediately extracted, wrap the container with aluminum foil to protect the phytoplankton from light and store the filter at  $-20^{\circ}\text{C}$ . Short term storage (2 to 4 h) on ice is acceptable, but samples should be stored at  $-20^{\circ}\text{C}$  as soon as possible.

- 8.2 Preservation – Sampled filters should be stored frozen ( $-20^{\circ}\text{C}$  or  $-70^{\circ}\text{C}$ ) in the dark until extraction.
- 8.3 Holding Time – Filters can be stored frozen for as long as 3½ weeks without significant loss of chlorophyll *a*<sup>(19)</sup>.

## 9.0 QUALITY CONTROL

- 9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of



laboratory capability and the continued analysis of laboratory reagent blanks, field duplicates and quality control samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data thus generated.

## 9.2 INITIAL DEMONSTRATION OF PERFORMANCE (MANDATORY)

9.2.1 The initial demonstration of performance is used to characterize instrument performance (instrumental detection limits, linear dynamic range and EDLs) and laboratory performance (analyses of QCSs) prior to sample analyses.

9.2.2 Linear Dynamic Range (LDR) – The LDR should be determined by analyzing a minimum of 5 calibration standards ranging in concentration from 0.2  $\mu\text{g/L}$  to 200  $\mu\text{g chl } a/\text{L}$  across all sensitivity settings of the fluorometer. Normalize responses by dividing the response by the sensitivity setting multiplier. Perform the linear regression of normalized response vs. concentration and obtain the constants  $m$  and  $b$ , where  $m$  is the slope and  $b$  is the y-intercept. Incrementally analyze standards of higher concentration until the measured fluorescence response,  $R$ , of a standard no longer yields a calculated concentration,  $C_c$ , that is  $\pm 10\%$  of the known concentration,  $C$ , where  $C_c = (R - b)/m$ . That concentration defines the upper limit of the LDR for your instrument. Should samples be encountered that have a concentration which is 90% of the upper limit of the LDR, these samples must be diluted and reanalyzed.

9.2.3 Instrumental Detection Limit (IDL) – Zero the fluorometer with a solution of 90% acetone on the maximum sensitivity setting. Pure chlorophyll  $a$  in 90% acetone should be serially diluted until it is no longer detected by the fluorometer on a maximum sensitivity setting.

9.2.4 Estimated Detection Limit (EDL) – Several blank filters should be extracted according to the procedure in Sect. 11, using clean glassware and apparatus, and the fluorescence measured. A solution of pure chlorophyll  $a$  in 90% acetone should be serially diluted until it yields a response which is 3X the average response of the blank filters.

9.2.5 Quality Control Sample (QCS) – When beginning to use this method, on a quarterly basis or as required to meet data quality needs, verify the calibration standards and acceptable instrument performance with the analysis of a QCS (Sect. 7.12). If the determined value is not within the confidence interval provided with the reference value, then the determinative step of this method is unacceptable. The source of the problem must be identified and corrected before continuing analyses.

9.2.6 Extraction Proficiency – Personnel performing this method for the first time should demonstrate proficiency in the extraction of sampled filters (Sect. 11.1). Twenty to thirty natural samples should be obtained using the procedure outlined in Sect. 8.1 of this method. Sets of 10 samples or more should be extracted and analyzed according to Sect. 11.2. The percent relative standard deviation (%RSD) of uncorrected values of chlorophyll  $a$  should not exceed 15% for samples that are approximately 10X the IDL. RSD for pheophytin  $a$  might typically range from 10 to 50%.

## 9.3 ASSESSING LABORATORY PERFORMANCE (MANDATORY)

9.3.1 Laboratory Reagent Blank (LRB) – The laboratory must analyze at least one blank filter with each sample batch. The LRB should be the last filter extracted. LRB data are used



to assess contamination from the laboratory environment. LRB values that exceed the IDL indicate contamination from the laboratory environment. When LRB values constitute 10% or more of the analyte level determined for a sample, fresh samples or field duplicates must be analyzed after the contamination has been corrected and acceptable LRB values have been obtained.

## 10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Calibration – Calibration should be performed bimonthly or when there has been an adjustment made to the instrument, such as replacement of lamp, filters or photomultiplier. Prepare 0.2, 2, 5, 20, and 200  $\mu\text{g chl } a/\text{L}$  calibration standards from the PDS (Sect. 7.11). Alternately, a calibration solution can be obtained from the address listed in Sec. 7.12. Allow the instrument to warm up for at least 15 min. Measure the fluorescence of each standard at sensitivity settings that provide midscale readings. Obtain response factors for chlorophyll *a* for each sensitivity setting as follows:

$$F_s = C_a/R_s$$

where:

$F_s$  = response factor for sensitivity setting, S.

$R_s$  = fluorometer reading for sensitivity setting, S.

$C_a$  = concentration of chlorophyll *a*.

If pheophytin *a* determinations will be made, it will be necessary to obtain before-to-after acidification response ratios of the chlorophyll *a* calibration standards as follows: (1) measure the fluorescence of the standard, (2) remove the cuvette from the fluorometer, (3) acidify the solution to 0.003 N HCl<sup>(4)</sup> with the 0.1 N HCl solution, (4) wait 90 sec and measure the fluorescence of the standard solution again. Addition of the acid may be made using a medicine dropper. It will be necessary to know how many drops are equal to 1 mL of acid. For a cuvette that holds 5 mL of extraction solution, it will be necessary to add 0.15 mL of 0.1 N HCl to reach a final acid concentration of 0.003 N in the 5 mL. Calculate the ratio, *r*, as follows:

$$r = R_b/R_a$$

where:

$R_b$  = fluorescence of pure chlorophyll *a* standard solution before acidification.

$R_a$  = fluorescence of pure chlorophyll *a* standard solution after acidification.

## 11.0 PROCEDURE

### 11.1 EXTRACTION OF FILTER SAMPLES

11.1.1 If sampled filters have been frozen, remove them from the freezer but keep them in the dark. Set up the tissue grinder and have on hand tissues and squirt bottles containing water and acetone. Workspace lighting should be the minimum that is necessary to read

instructions and operate instrumentation. Remove a filter from its container and place it in the glass grinding tube. Push it to the bottom of the tube with a glass rod. With a volumetric pipet, add 4 mL of the aqueous acetone solution (Sect. 7.8) to the grinding tube. After the filter has been converted to a slurry, grind the filter for approximately 1 min at 500 rpm. Pour the slurry into a 15-mL screw-cap centrifuge tube and, using a 6-mL volumetric pipet, rinse the pestle and the grinding tube with 90% acetone. Add the rinse to the centrifuge tube containing the filter slurry. Cap the tube and shake it vigorously. Place it in the dark before proceeding to the next filter extraction. Before placing another filter in the grinding tube, use the acetone and water squirt bottles to thoroughly rinse the pestle, grinding tube and glass rod. The last rinse should be with acetone. Use a clean tissue to remove any filter residue that adheres to the pestle or to the steel rod of the pestle. Proceed to the next filter and repeat the steps above. The entire extraction with transferring and rinsing steps takes 5 min. Approximately 500 mL of acetone and water waste are generated per 20 samples from the rinsing of glassware and apparatus.

11.1.2 Shake each tube vigorously before placing them to steep in the dark at 4°C. Samples should be allowed to steep for a minimum of 2 h but not to exceed 24 h. Tubes should be shaken at least once during the steeping period or placed horizontally to allow the extraction solution to have maximum contact with the filter slurry.

11.1.3 After steeping is complete, centrifuge samples for 15 min at 675 g or for 5 min at 1000 g. Samples should be allowed to come to ambient temperature before analysis. This can be done by placing the tubes in a constant temperature water bath or by letting them stand at room temperature for 30 min. Recalibrate the fluorometer if the room temperature fluctuated  $\pm 3^\circ\text{C}$  from the last calibration date.

## 11.2 SAMPLE ANALYSIS

11.2.1 After the fluorometer has warmed up for at least 15 min, use the 90% acetone solution to zero the instrument on the sensitivity setting that will be used for sample analysis.

11.2.2 Pour or pipet the supernatant of the extracted sample into a sample cuvette. The volume of sample required in your instrument's cuvette should be known so that the correct amount of acid can be added in the pheophytin *a* determinative step. For a cuvette that holds 5 mL of extraction solution, 0.15 mL of the 0.1 N HCl solution should be used. Choose a sensitivity setting that yields a midscale reading when possible and avoid the minimum sensitivity setting. If the concentration of chlorophyll *a* in the sample is  $\geq 90\%$  of the upper limit of the LDR, then dilute the sample with the 90% acetone solution and reanalyze. Record the fluorescence measurement and sensitivity setting used for the sample. Remove the cuvette from the fluorometer and acidify the extract to a final concentration of 0.003 N HCl using the 0.1 HCl solution. Wait 90 sec before measuring fluorescence again. Twenty-five to thirty-five samples can be extracted and analyzed in one 8 hr day.

## 12.0 DATA ANALYSIS AND CALCULATIONS

12.1 "Uncorrected" chlorophyll *a* may be determined in a sample extract by multiplying the fluorescence response of the sample by the appropriate response factors determined in Sect. 10.1. Determine the "corrected" chlorophyll *a* concentration in the sample extract and the pheophytin *a* concentration in  $\mu\text{g/L}$  as follows:

$$\text{Chlorophyll } a, \mu\text{g/L} = F_s (r/r - 1 - (R_b - R_a))$$

$$\text{Pheophytin } a, \mu\text{g/L} = F_s (r/r-1) (rR_a - R_b)$$

where:

- $F_s$  = response factor for the sensitivity setting used.
- $R_b$  = fluorescence of sample extract before acidification.
- $R_a$  = fluorescence of sample extract after acidification.
- $r$  = the before-to-after acidification ratio of a pure chlorophyll *a* solution (Sect. 10.1).

12.2 The concentration of chlorophyll *a* and pheophytin *a* in the natural water sample is calculated by multiplying the results obtained in Sect. 12.1 by 10 mL (the extraction volume) and dividing by the volume (mL) of natural water sample that was filtered. Any other dilution or concentration factors should be incorporated accordingly.

12.3 LRB and QCS data should be reported with each sample data set.

### 13.0 METHOD PERFORMANCE

13.1 EDL for the instrument used in the evaluation of this method was 0.05  $\mu\text{g/L}$  for chlorophyll *a* and 0.06  $\mu\text{g/L}$  pheophytin *a*.

13.2 The precision (%RSD) for chlorophyll *a* in mostly blue-green and green phytoplankton natural sampled which were steeped for 2 h vs 24 h is reported in Table 1. Although the means were the same, precision was better for samples which were allowed to steep for 24 h prior to analysis. Since pheophytin *a* was found in the samples, the chlorophyll *a* values are “corrected” (Sect. 12.1). Table 2 contains precision data for pheophytin *a*. A statistical analysis of the pheophytin *a* data indicated a significant difference at the 0.05 significance level in the mean values obtain. The cause of the lower pheophytin *a* values in samples extracted for 24 h is not known.

13.3 Three QCS ampoules obtained from the USEPA were analyzed and compared to the reported confidence limits in Table 3. The reference values for QCS obtained from the USEPA are periodically updated and new confidence limits established.

### 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generate (e.g., Sect. 11.1.1). When wastes cannot be feasibly reduced as the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult *Less is better: Laboratory Chemical Management for Waste*

*Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16<sup>th</sup> Street N.W., Washington D.C. 20036, (202) 872-4477.

## 15.0 WASTE MANAGEMENT

- 15.1 The Environmental Protection agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult *The Waste Management for Laboratory Personnel*, available from the American Chemical Society at the address listed in Sect. 14.2.

## 16.0 REFERENCES

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## 17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE 1. COMPARISON OF PRECISION OF TWO EXTRACTION PERIODS

CORRECTED CHLOROPHYLL *a*

	Sample A <sup>(1)</sup>		Sample B <sup>(2)</sup>	
	2 h <sup>(3)</sup>	24 h <sup>(3)</sup>	2 h <sup>(3)</sup>	24 h <sup>(3)</sup>
Mean Concentration ( $\mu\text{g/L}$ )	49.6	52.9	78.6	78.8
Standard Deviation ( $\mu\text{g/L}$ )	4.89	2.64	6.21	2.77
Relative Standard Deviation(%)	9.9	5.0	7.9	3.5

<sup>1</sup> Values reported are the mean measured concentrations (n=6) of chlorophyll *a* in the natural water based on a 100-mL filtration volume.

<sup>2</sup> Values reported are the mean measured concentrations (n=9) of the extraction solution. Sample filtration volume was 300 mL.

<sup>3</sup> The length of time that the filters steeped after they were ground.



**TABLE 2. COMPARISON OF PRECISION OF TWO EXTRACTION PERIODS FOR PHEOPHYTIN *a***

<b>PHEOPHYTIN <i>a</i></b>				
Sample A <sup>(1)</sup>		Sample B <sup>(2)</sup>		
2 h <sup>(3)</sup>	24 h <sup>(3)</sup>	2 h <sup>(3)</sup>	24 h <sup>(3)</sup>	
Mean Concentration ( $\mu\text{g/L}$ )				
9.22	8.19	13.10	10.61	
Standard Deviation ( $\mu\text{g/L}$ )				
2.36	3.55	3.86	2.29	
Relative Standard Deviation(%)				
25.6	43.2	29.5	21.6	

<sup>1</sup> Values reported are the mean measured concentrations (n=6) of pheophytin *a* in the natural water based on a 100-mL filtration volume.

<sup>2</sup> Values reported are the mean measured concentrations (n=9) of pheophytin *a* the extraction solution. Sample filtration volume was 300 mL.

<sup>3</sup> The length of time that the filters steeped after they were ground.

**TABLE 3. ANALYSES OF USEPA QC SAMPLES**

<b>ANALYTE</b>	<b>REFERENCE VALUE</b>	<b>CONFIDENCE LIMITS</b>
Chlorophyll <i>a</i>	2.1 $\mu\text{g/L}$	0.5 to 3.7 $\mu\text{g/L}$
Pheophytin <i>a</i>	0.3 $\mu\text{g/L}$	-0.2 to 0.8 $\mu\text{g/L}$
<b>ANALYTE</b>	<b>MEAN MEASURED VALUE</b>	<b>% Relative Standard Deviation</b>
Chlorophyll <i>a</i>	2.8 $\mu\text{g/L}$	1.5
Pheophytin <i>a</i>	0.3 $\mu\text{g/L}$	33

## A PROCEDURE FOR MEASURING EXTRACTED CHLOROPHYLL *a* FREE FROM THE ERRORS ASSOCIATED WITH CHLOROPHYLL *b* AND PHEOPIGMENTS

(Without Acidification - Using 13 mm Test Tubes)

Instrument set-up: Model TD-700 Fluorometer equipped with:

1. 13 mm cuvette holder (included with the TD-700 Fluorometer)
2. Optical Filter Kit PN 7000-962, which includes:  
     PN 10.113 (436 nm) Excitation Filter  
     PN 10-115 (680 nm) Emission Filter;
3. PN 10-089 Blue Lamp (F4T4.5B2 equivalent).

### SUMMARY OF THE METHOD

Conventional fluorescence methods for measuring chlorophyll *a* require samples to be measured twice; once before acidification and once afterwards. Under the most extreme ratio of chlorophyll *a*/chlorophyll *b* likely to occur in nature (1:1 molar), conventional acidification techniques result in approximately 60% underestimate of true chlorophyll *a*.

Under these conditions, the new method<sup>1</sup> described in these pages yields at most a 10% overestimate of true chlorophyll *a*. In addition, it requires a single fluorescence determination and is sensitive enough for estimates of euphotic zone chlorophyll *a* in all marine and freshwater ecosystems. Filtration of less than 200 mL of water provides adequate sensitivity even in the most oligotrophic environments.

The method requires:

1. Sample preparation.
2. Calibration of the Fluorometer
3. Reading samples.

### SAMPLE PREPARATION

Detailed instructions for extracting chlorophyll *a* and measuring with the Turner Designs analog fluorometer can be found in United States Environmental Protection Agency (EPA) Method 445.0 "In Vitro Determination of Chlorophyll *a* and Pheophytin *a* in Marine and Freshwater Phytoplankton by Fluorescence." A copy is enclosed for your convenience. Method 445.0 sets forth the conventional fluorescence procedure, requiring two readings for each sample—before and after acidification. (Method 445.0 can be found in the EPA standard methods book, *Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Samples*.)

**PLEASE NOTE** that the procedure described in these instructions is without acidification. The Model TD-700 must be configured with special optical filters and lamp which read chlorophyll *a* in the presence of chlorophyll *b* and pheopigments. These optical filters and lamp should be installed according to instructions in your Model TD-700 User's Manual Sections III and IV (Optical Filter Installation and Removal, and Lamp Installation and Removal). **USING THIS METHOD**, you must NOT acidify your samples as set forth in EPA Method 445.0, section 11.2.2; and you do not need to perform any calculations as required by section 12.1 of Method 445.0

## CALIBRATION

All you need to do is calibrate the instrument with the following procedure. The calibration should remain stable for some time, and unless you change your blank or standard or want to change from reading very high levels to very low levels (or vice versa), you may not have to calibrate every time you read a new batch of samples. (You will, of course, need to recalibrate if you change the lamp or filters.)

Please note, however that the standard must be within the linear range for accurate readings (according to the EPA Method 445.0, using the 13 mm cuvettes, chlorophyll *a* is linear to  $250 \mu\text{g/L}$ )<sup>2</sup>.

### To calibrate:

Have read a blank of 90% acetone and your standard of known concentration of pure chlorophyll *a* in 90% acetone.

1. Turn on the fluorometer and allow it to warm up for 10 minutes.

Because temperature affects fluorescence, do not allow the blank to remain in the instrument any longer than necessary for a stable reading.

2. Prepare a pure chlorophyll *a* standard and a blank of 90% acetone in a 13 mm test tube. Put the standard in the sample chamber and close the lid. Calibrate according to Section VII (Calibration - Raw Fluorescence) or VIII (Calibration - Direct Concentration) in the TD-700 user's manual (whichever you prefer). Remove the standard and insert the blank when the software prompts you to. When the blank reading is stable, press <0>. When finished, remove the blank.

## READING SAMPLES

Refer to your user's manual, Section IX (Reading Samples), for additional details.

For your convenience, the Model TD-700 has a "Discrete Sample Averaging" capability, where the instrument averages a reading over a preset period, allowing you to read samples after they have been in the instrument for the same amount of time. This removes the guesswork from reading the digital display and minimizes error due to temperature changes. Defaults for the Model TD-700 are 7 seconds pre-delay for the signal to stabilize, and an averaging period of 12 seconds. To use Discrete Sample Averaging, after putting in your sample, from the HOME screen, press <\*> and the instrument will countdown a delay period, average the reading, and then display "END" in the left corner of the screen. The averaged reading will be displayed for 5 seconds. If the fluorometer is not connected to a printer or a computer, write down the reading.

Procedure for running samples:

1. Fill a clean cuvette with a sample, wipe the outside of the cuvette dry with a lab wipe, and place in the instrument. Close the lid.
2. Wait about 10 seconds for the reading to stabilize, and log the reading. (Remember: Because of temperature effects, for greatest accuracy, read all samples after they have been in the

fluorometer for approximately the same length of time.) If the display reads “OVER”, dilute the sample by 25% (1 part sample to 3 parts 90% acetone solution), and read it. Multiply the reading by 4 to get the actual concentration.

3. Remove the cuvette and put in the next sample.
4. Repeat steps 1 - 3 until all samples are read.

If you calibrated in the direct concentration calibration procedure, these readings are the actual concentration of extracted chlorophyll *a* in the cuvette. To arrive at the environmental chlorophyll *a*, for each sample you must correct for the volume of water filter and the volume of 90% acetone used in the extraction.

NOTE: It won't hurt the fluorometer to leave it on all day. If you are going to be reading samples off-and-on over the course of a few days, it is better to leave the fluorometer on.

1. The method was developed by Dr. Nicholas A. Welschmeyer of Moss Landing Marine Laboratories, Moss Landing, CA. A paper by Dr. Welschmeyer, Fluorometric Analysis of Chlorophyll *a* in the presence of Chlorophyll *b* and Pheopigments, which details his research, is scheduled to appear in *Limnology and Oceanography*.
2. Method 445. found 250  $\mu\text{g/L}$  to be the upper limit of the linear dynamic range for 13 mm cuvettes using the Turner Designs Model 10 Fluorometer. See section 9.2 of Method 445.0 for procedure for establishing the upper limit of the linear dynamic range for your fluorometer. It will vary somewhat from instrument to instrument.
3. Generally, the standard concentration should be approximately 80% of the maximum concentration you wish to read. This is a rule of thumb and not a rigid requirement. If you are using EPA standards, you can dilute the fluorometric or the spectrophotometric standards of pure chlorophyll *a* with 90% acetone to make the appropriate concentration.

## TD-700 OPTICAL CONFIGURATION GUIDE

Application	Part #*	Recommended Lamp**	Excitation Filter	Emission Filter	PM Tube***
<b>Chlorophyll <i>a</i> - Traditional</b> <b>Acidification Method:</b> Chlorophyll <i>a</i> and pheophytin <i>a</i> extractive methods with acidification where little chlorophyll <i>b</i> is present.	7000-961	10-045 Daylight White Lamp	10-050R 340-500 nm	10-051R >665 nm	Red Sensitive PMT
<b>Chlorophyll <i>b</i> - Welshmeyer</b> <b>Non-Acidification Method:</b> Chlorophyll <i>a</i> extractive methods without acidification.	7000-962	10-045 Daylight White Lamp or 10-089 Blue Lamp	10-113 436 nm	10-115 680nm	Red Sensitive PMT
<b>Chlorophyll <i>a</i> - Freshwater In-Vivo</b> <b>Method:</b> Chlorophyll <i>a</i> with interferences from humic substances of other pheopigments	7000-963 Blue Lamp	10-089 340-500 nm	10-050R 680nm	10-115	Red Sensitive PMT
<b>Fluorescein Optical Kit:</b> Applications include FITC, Thiazole Orange, PicoGreen™, Enzcheck™ Protease, and NanoOrange™	7000-964	7000-930 Quartz Halogen Lamp or 10-045 Daylight White Lamp	10-105 486nm	10-109R-C 510-700nm	Standard PM Tube
<b>Short U.V. Optical Kit:</b> Applications include: Short Wavelength Oils. Note: Quartz sample cuvettes must be purchased with this filter kit.	7000-965	10-046 Clear Quartz Lamp	10-038R 254nm	10-107R-C 300-400nm	Standard PM Tube
<b>Rhodamine Optical Kit</b>	7000-966	10-046 Clear Quartz Lamp or 7000-930 Quartz Halogen Lamp	10-103 550nm	10-052R >570nm (nearest PMT) 10-058R 325-700nm (nearest sample)	Standard PM Tube
<b>Long U.V. Optical Kit:</b> Applications include: Hoechst Dye 33258, Aflatoxins, Histamines, Alkaline Phosphatase, Methylumbelliferyl, and Long Wavelength Oils.	7000-967	10-049 Near U.V. Lamp	10-069R 300-400nm	10-110-R-C 410-600nm	Standard PM Tube

\* All filter kits include the appropriate 1" round filters listed under each application, and o' rings required for filter installation.

\*\* Lamps must be purchased separately.

\*\*\* The Red Sensitive PM Tube recommended for the chlorophyll applications is factory installed and should be ordered with the instrument.

## APPENDIX E

# SAMPLE FIELD DATA FORMS<sup>1</sup>

<sup>1</sup> Field Data Forms from Coastal 2000 Gulf Region



STATION FORM  
Coastal 2000 - Gulf Region

STAGING AREA \_\_\_\_\_

STATION DEPTH (ft.): \_\_\_\_\_

STATION NAME  
\_\_\_\_\_

<b>DAY</b>	DATE: (MM/DD/YY)		ARRIVAL TIME (HH:MM)	
	TIME ZONE: <input type="radio"/> Eastern / <input type="radio"/> Central		DEPART TIME (HH:MM)	
<b>GPS</b>	LAT: (00°00.00)		LON: (00°00.00)	
	Site Within 0.02 NM: <input type="radio"/> Yes or <input type="radio"/> No <span style="float: right;">If NO, explain in 'NOTES'</span>			
<b>CREW</b>	CAPTAIN:		VISITOR 1:	
	CREW 1:		VISITOR 2:	
	CREW 2:		VISITOR 3:	
<b>WEATHER</b>	CONDITIONS AT STATION:			
<b>HABITAT TYPE</b>	<input type="checkbox"/> Tidal River		<input type="checkbox"/> Coral Reef	<input type="checkbox"/> Other
	<input type="checkbox"/> Open Water		<input type="checkbox"/> Marsh	
	<input type="checkbox"/> Bayou/Inlet		<input type="checkbox"/> Oyster Bed	
	<input type="checkbox"/> Inter-Tidal		<input type="checkbox"/> Marina	
	<input type="checkbox"/> Rocky/Shell Bottom		<input type="checkbox"/> Grass Bed	
<b>SAV</b>	Present ? <input type="radio"/> Yes or <input type="radio"/> No			
<b>MARINE DEBRIS</b>	Present?		Type	
	<input type="radio"/> Yes or <input type="radio"/> No		<input type="checkbox"/> GLASS	<input type="checkbox"/> CANS
			<input type="checkbox"/> PLASTIC	<input type="checkbox"/> Other. . .

PLACE COMMENTS ON BACK OF FORM

RECORDED BY: \_\_\_\_\_ COMPUTER ENTRY BY: \_\_\_\_\_



**WATER SAMPLE COLLECTION**  
**Coastal 2000 - Gulf Region**STATION NAME  
\_\_\_\_\_DATE: \_\_\_\_\_ FILTRATION METHOD: ☐ SYRINGE or ☐ VACUUM  
(MMDDYY)**SURFACE (0.5m)**NUTRIENT  
(SN)\*☐ (Filtered Sample - 60 ml)CHLOROPHYLL  
(SCL)☐ (25 mm Filter) Volume Filtered: \_\_\_\_\_ (ml)TSS  
(SSS)☐ (Unfiltered Sample - 1 Liter)**MID-DEPTH**

N/A (&lt;2 m)

☐NUTRIENT  
(MN)☐ (Filtered Sample - 60 ml)CHLOROPHYLL  
(MCL)☐ (25 mm Filter) Volume Filtered: \_\_\_\_\_ (ml)TSS  
(MSS)☐ (Unfiltered Sample - 1 Liter)**BOTTOM (0.5 off  
bottom)**NUTRIENT  
(BN)☐ (Filtered Sample - 60 ml)CHLOROPHYLL  
(BCL)☐ (25 mm Filter) Volume Filtered: \_\_\_\_\_ (ml)TSS  
(BSS)☐ (Unfiltered Sample - 1 Liter)

\* Sample label to be placed on container is listed in Italics

RECORDED BY: \_\_\_\_\_ COMPUTER ENTRY BY: \_\_\_\_\_

BENTHIC INFAUNA COLLECTION  
Coastal 2000 - Gulf Region

STATION NAME

DATE: (MMDDYY) \_\_\_\_\_ GRAB TYPE: ☐ Van Veen or ☐ Corer

SAMPLE NO. 1 (BI1)*	TIME (HH:MM):	DEPTH (cm):
	DESCRIPTION:	
	NO. OF JARS USED:	
SAMPLE NO. 2 (BI2)*	TIME (HH:MM):	DEPTH (cm):
	DESCRIPTION:	
	NO. OF JARS USED:	
SAMPLE NO. 3 (BI3)*	TIME (HH:MM):	DEPTH (cm):
	DESCRIPTION:	
	NO. OF JARS USED:	

\*Sample label to be placed on container is listed in Italics

RECORDED BY: \_\_\_\_\_ COMPUTER ENTRY BY: \_\_\_\_\_

**SEDIMENT SAMPLE COLLECTION**

Coastal 2000 - Gulf Region

Station Name  
\_\_\_\_\_

DATE: (MMDDYY) \_\_\_\_\_

**SEDIMENT CHEMISTRY**I. ☐Organics (*SO*)\*

(Glass Jar)

II. ☐Metals (*SM*)

(Nalgene 125 ml)

**TOXICITY (*ST*)**☐

(1 gal - Nalgene)

**GRAIN SIZE (*SG*)**☐

(250 ml - Nalgene)

**SEDIMENT TOC (*TOC*)**☐

(60 ml)

\* Sample label to be placed on container is listed in italics

RECORDED BY: \_\_\_\_\_ COMPUTER ENTRY BY: \_\_\_\_\_

**SEDIMENT SAMPLES ONLY**

**FISH TRAWL #**  
**Coastal 2000 - Gulf Region**

Net Width (ft): \_\_\_\_\_

Station Name  
 \_\_\_\_\_

TRAWL INFO	DATE (mm/dd/yy):
	HELMSMAN:
	LINE OUT (m):
TRAWL START	LAT (00°00.00'):
	LON (00°00.00'):
	HEADING IN DEGREES MAGNETIC:
	START TIME (HH:MM):
TRAWL END	LAT (00°00.00'):
	LON (00°00.00'):
	END TIME (HH:MM):
TRAWL DETAIL	TRAWL TAKEN: <input type="radio"/> Yes or <input type="radio"/> No
	IF NO, EXPLAIN:
	TRAWL SUCCESSFUL: <input type="radio"/> Yes or <input type="radio"/> No
	IF NO, EXPLAIN:
	ANYTHING CAUGHT: <input type="radio"/> Yes or <input type="radio"/> No

RECORDER: \_\_\_\_\_ COMPUTER ENTRY BY: \_\_\_\_\_



**FISH DATA**  
**Coastal 2000 - Gulf Region**

Station Name

DATE: \_\_\_\_\_

COMMON NAME: \_\_\_\_\_

GENUS SPECIES NAME \_\_\_\_\_

Fish Number	Fish Length	Samples		Pathology Type	TRAWL INFO:
		Chemistry	Pathology		NUMBER
1					
2					TYPE
3					○ STANDARD
4					○ NON-STANDARD
5					○ OTHER. . .
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					PATHOLOGY
18					OBSERVATIONS
19					G - GILL ABN
20					U - ULCERS
21					L - LUMPS/BUMPS
22					S - SKELETAL ABN
23					E - EYE ABN
24					
25					
26					
27					
28					
29					TOTAL COUNT:
30					

RECORDER: \_\_\_\_\_ ENTRY BY: \_\_\_\_\_

**APPENDIX F**  
**Field Crew Evaluation Checklist**  
(Gulf Region - Coastal 2000)

# COASTAL 2000 FIELD CREW EVALUATION - GULF REGION

**Date:**

**Crew/Vessel:**

**Location:**

**Evaluator:**

Acceptable

Unacceptable

## **I. Preparation**

Hydrolab Calibration

Supplies/Containers

Sampling Gear

## **II. Water Quality Parameters**

Water Column Profile

Hydrolab

LiCor

Secchi

Water Sampling

Filtration

Nutrient sample

CHL sample

TSS sample

field sheets

## **III. Sediment Grabs**

Benthic grab

Composite grabs

## **IV. Benthic Processing**

sieving

sample transfer

preservation

field sheet

## **Field Crew Evaluation (continued)**

Acceptable Unacceptable

## **V. Composite Sediment**

compositing/mixing

distribution to containers

sample sheet

## **VI. Fish Trawls**

Deployment/retrieval of net

time of trawl

fish IDs

fish measurements

fish composites for chem

sample processing for chem

field sheets

**General comments:**