

CATALOG DOCUMENTATION  
EMAP-ESTUARIES PROGRAM LEVEL DATABASE  
1993 VIRGINIAN PROVINCE  
SEDIMENT TOXICITY TEST DATA

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1. DATA SET IDENTIFICATION

1.1 Title of Catalog document

EMAP-Estuaries Program Level Database  
1993 Virginian Province  
Sediment Toxicity Test Data

1.2 Authors of the Catalog entry

Charles Strobel, US EPA NHEERL-AED  
Melissa Hughes, OAO Corp.

1.3 Catalog revision date

14 March 1996

1.4 Data set name

Toxicity

## 1.5 Task Group

Estuaries

## 1.6 Data set identification code

00104

## 1.7 Version

001

## 1.8 Requested Acknowledgment

If you plan to publish these data in any way, EPA requires a standard statement for work it has supported:

"Although the data described in this article have been funded wholly or in part by the U. S. Environmental Protection Agency through its EMAP-Estuaries Program, it has not been subjected to Agency review, and therefore does not necessarily reflect the views of the Agency and no official endorsement should be inferred."

## 2. INVESTIGATOR INFORMATION

### 2.1 Principal Investigator

Darryl Keith  
U.S. Environmental Protection Agency  
NHEERL-AED

### 2.2. Investigation Participant-Sample Collection

Charles J. Strobel  
U.S. Environmental Protection Agency  
NHEERL-AED

### 2.3 Sample Processing-Principal Investigator

Dr. K. John Scott  
Science Applications International Corporation, Inc.

## 3. DATA SET ABSTRACT

### 3.1 Abstract of the Data Set

The SEDIMENT TOXICITY TEST data set provides summary data on a sediment toxicity test associated with a station. The test was conducted using an homogenized sample composed of several grabs. A static ten-day sediment toxicity test was conducted using the amphipod *Ampelisca abdita*. The mean test sample survival as per cent of the mean control survival is presented. A flag indicates if test and control mortalities were significantly different.

### 3.2 Keywords for the Data Set

Sediment toxicity test, toxicity test results, amphipod, Ampelisca, marine amphipod

## 4. OBJECTIVES AND INTRODUCTION

### 4.1 Program Objective

The Environmental Monitoring and Assessment Program (EMAP) was designed to periodically estimate the status and trends of the Nation's ecological resources on a regional basis. EMAP provides a strategy to identify and bound the extent, magnitude and location of environmental degradation and improvement on a regional scale based on randomly located station sites. Only Base Sampling Sites (BASE) were included in this data set. Sediment toxicity testing is considered a core indicator in the EMAP program for which there presently exists sufficient data to define the sensitivity and reliability of responses to stress with a high degree of confidence.

### 4.2 Data Set Objective

The toxicity of estuarine sediments to the amphipod *Ampelisca abdita* was estimated in a 10-day, static laboratory exposure.

### 4.3 Data Set Background Information

Sediment toxicity tests are the most direct measure available for estimating the potential for contaminant-induced effects in benthic communities. These tests provide information that is independent of chemical characterizations and ecological surveys. They improve upon direct measures of contaminants because many chemicals are bound tightly to sediment particles or are complexed chemically, making them biologically unavailable. Mortality in these laboratory exposure tests can provide evidence of toxic contamination without requiring interpretation of how complex mixtures might interact to affect biota. However, sediment toxicity cannot be used entirely in replacement of direct measurement of sediment contaminant concentrations, since the latter is an important part of interpreting observed mortality in toxicity tests.

Although amphipod toxicity test methods have gained general acceptance, a number of factors that affect their application over the broad geographic and habitat range were assessed by EMAP-Estuaries. Salinity effects are also a potential concern. Sediment exposure tests for EMAP were all conducted at the same salinity using full strength seawater (30 ppt), regardless of the salinity at the collection site; thus, low salinity sediments were adjusted to full strength salinity for the test. This increase in salinity may have decreased bioavailability of metal contaminants.

#### 4.4 Summary of Data Set Parameters

A summary of replicate sediment toxicity test results were compared to summary test control data. The ten-day test was conducted with the amphipod, *Ampelisca abdita*. A sediment homogenate was used which was derived from several samples collected at a station.

### 5. DATA ACQUISITION AND PROCESSING METHODS

#### 5.1 Data Acquisition

##### 5.1.1 Sampling Objective

Collect one sediment sample per station suitable to conduct a sediment toxicity test with a marine organism. One sediment sample was expected to be collected at each station.

##### 5.1.2 Sample Collection Methods Summary

The grab sampler was lowered through the water column such that travel through the last 5 meters is no faster than 1 m/sec. This minimized the effects of bow wave disturbance to surficial sediments. The grab penetrated the sediment by gravity releasing a trigger allowing the jaws to close. When the grab was pulled from the sediment using the winch, the jaws closed, encapsulating the sediment sample. The sampler was retrieved and lowered into an on-board cradle.

A successful grab had relatively level, intact sediment over the entire area of the grab and a sediment depth at the center of > 7 centimeters. Unacceptable grabs included those: not containing any sediment, which were partially filled, had shelly substrates or grossly slumped surfaces or were completely filled to the top, where the sediment was oozing out of the hinged top.

To minimize the chance of sampling the exact location twice, after three grabs were taken, the boat was moved five meters downstream by letting out the appropriate length of anchor line.

Large items in the grab such as rocks or pieces of wood were removed from the sediment. The top two centimeters of the sediment at least one cm from the edge of the sample were removed using a stainless steel spoon (all items were washed with Alconox and rinsed with ambient seawater before use). The sediment was placed in a pan or pot and placed in a cooler on ice for refrigerated storage. This procedure was repeated with each sediment grab collected until at least 3,000 cc of sediment had been collected. The sediment composite was then homogenized by stirring with a spoon for 10 minutes. Using a spoon, approximately 3,000 cc of the sediment homogenate was placed in a container for toxicity testing. The toxicity sample bottle was placed on ice.

### 5.1.3 Beginning Sampling Date

27 July 1993

### 5.1.4 Ending Sampling Date

31 August 1993

### 5.1.5 Platform

Sampling was conducted from 8 m (24 ft), twin-engine Chesapeake style work boats.

### 5.1.6 Sampling Equipment

A 1/25 m<sup>2</sup>, stainless steel, Young-modified Van Veen Grab sampler was used to collect sediments. This grab sampled an area of 440 cm<sup>2</sup> and a maximum depth of penetration in the sediment of 10 cm.

### 5.1.7 Manufacturer of Sampling Equipment

Young's Welding, Sandwich, MA

### 5.1.8 Key Variables

This data set does not contain any values which were measured at the time of collection. Analysis of the data after completion of the tests produced summary results.

### 5.1.9 Sampling Method Calibration

The sampling gear did not require any calibration. It required inspection for deformities incurred due to mishandling or impact on rocky substrates.

### 5.1.10 Sample Collection Quality Control

Prior to sampling at each station, the grab sampler was washed with Alconox and thoroughly rinsed with ambient seawater to ensure that no sediment remained from a previous station. The spoon and processing (homogenizing) container used to process the sediment sample in the field were stainless steel coated with Kynar. The sample container was a one-gallon plastic jar.

To minimize airborne contamination, all engines were shut down when ever sediment containers were open.

### 5.1.11 Sample Collection Method Reference

Reifsteck, D.R., Strobel, C.J. and D.J. Keith. 1993. Environmental Monitoring and Assessment Program-Estuarines: 1993 Virginian Province Effort Field Operations and Safety Manual. U.S. EPA NHEERL-AED, Narragansett, RI. June 1993.

#### 5.1.12 Sample Collection Method Deviations

None

### 5.2 Data Preparation and Sample Processing

#### 5.2.1 Sample Processing Objective

Process uncontaminated sediment samples for characterization of sediment toxicity to the amphipod *Ampelisca abdita*.

#### 5.2.2 Sample Processing Methods Summary

Each test replicate consisted of 200 mL of sediment sample (roughly 4 cm in depth) in a quart-size canning jar covered with 600 mL of aerated water. The east coast marine amphipod *Ampelisca abdita* was employed as the test species for all sediment samples collected. Tests were conducted for 10 days under static conditions at a constant temperature of 20 degrees C and dissolved oxygen concentration >60% of saturation. The overlying water in *A. abdita* tests was 30 ppt seawater. Five replicate test chambers, each containing 20 organisms, were tested with sediment from each station along with a control treatment containing known uncontaminated sediment.

Control treatments used the same water, conditions, procedures, and organisms as the other test treatments, except that none of the test material was added to the control sediment or water. The control treatments were used to provide: a) a measure of the acceptability of the test by providing evidence of the health and relative quality of the test organisms, and the suitability of the overlying water, test conditions, and handling procedures, etc.; and b) the basis for interpreting data obtained from the test sediments.

Mortality and sublethal effects such as emergence from the sediment were determined during and after exposure to the test sediment. Dead animals were counted and removed daily. At the end of the 10 day exposure, the test sediments were rinsed through a 0.5 mm mesh sieve. The material retained on the sieve was either examined that day or preserved in 5% buffered formalin with Rose Bengal stain for later examination. Any amphipods which were not accounted for when the sieved material was examined were presumed to have died during the test. Survival in control treatments of <85% resulted in the entire test being repeated, discarded or flagged.

#### 5.2.3 Sample Processing Method Calibration

NA

#### 5.2.4 Sample Processing Quality Control

Samples were chilled when collected and shipped on ice. Sediment toxicity samples were stored in the dark at 4 Deg C until used. The samples were tested within 30 days of collection.

Sediment samples were thoroughly homogenized within the storage container and press sieved through a stainless steel screen (1.0 mm mesh) to remove predators and larger particles (e.g., rocks and shells) before addition to test chambers.

#### 5.2.5 Sample Processing Method Reference

U.S. EPA. 1995. Environmental Monitoring and Assessment Program (EMAP): Laboratory Methods Manual-Estuaries, Volume 1: Biological and Physical Analyses. U.S. Environmental Protection Agency, Office of Research and Development, Narragansett, RI. EPA/620/R-95/008.

#### 5.2.6 Sample Processing Method Deviations

None

### 6. DATA MANIPULATIONS

Measurements on a 'per replicate' basis were received from an analytical laboratory. Mean test and control replicate mortality and survival were determined as a basis for relevant data manipulations.

#### 6.1 Name of New or Modified Values

SURVIVAL  
SIG\_CONT

#### 6.2 Data Manipulation Description

##### 6.2.1 SURVIVAL

The values under SURVIVAL represent a comparison of the mean test survival to the mean control survival.

##### 6.2.2 SIG\_CONT

A one-tailed t-test ( $\alpha=0.05$ ) was used to determine if the mean per cent sample mortality was significantly different from the mean per cent control mortality.

#### 6.3 Data Manipulation Examples

##### 6.3.1 SURVIVAL

$((\text{Mean \% Test Survival} / \text{Mean \% Control Survival}) * 100)$

## 7. DATA DESCRIPTION

### 7.1 Description of Parameters

#	Parameter	Data SAS Name	Type	Len	Format	Parameter Label
1	STA_NAME	Char	8	8.		The Station Identifier
2	VST_DATE	Num	8	YYMMDD6.		The Date the Sample was Collected
3	SPECCODE	Char	8	\$8.		EMAP Taxon Code
4	SURVIVAL	Num	8	5.1		% Survival (Samp Mean as % of Control)
5	SIG_CONT	Char	8	\$3.		Sig Diff from Control(Samp x % Mortality)

#### 7.1.6 Precision to which values are reported

Values are reported to one decimal point.

#### 7.1.7 Minimum Value in Data Set

00.0 %

#### 7.1.8 Maximum Value in Data Set

113.0 %

### 7.2 Data Record Example

#### 7.2.1 Column Names for Example Records

STA_NAME	VST_DATE	SPECCODE	SURVIVAL	SIG_CONT
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#### 7.2.2 Example Data Records

VA93-601	930918	AMPEABDI	100.0	N
VA93-602	930813	AMPEABDI	98.0	N

## 8. GEOGRAPHIC AND SPATIAL INFORMATION

### 8.1 Minimum Longitude

-77 Degrees 23 Minutes 37.20 Decimal Seconds

### 8.2 Maximum Longitude

-70 Degrees 01 Minutes 9.00 Decimal Seconds

### 8.3 Minimum Latitude

36 Degrees 56 Minutes 54.00 Decimal Seconds

### 8.4 Maximum Latitude

42 Degrees 11 Minutes 30.00 Decimal Seconds

## 8.5 Name of area or region

Virginian Province

Stations were located in estuaries along the East Coast of the United States from Cape Cod, Massachusetts, to Cape Henry, Virginia, at the mouth of the Chesapeake Bay. The area includes the District of Columbia, Virginia, Maryland, Delaware, Pennsylvania, New Jersey, New York, Connecticut, Rhode Island and Massachusetts.

## 9. QUALITY CONTROL/QUALITY ASSURANCE

### 9.1 Measurement Quality Objectives

The required control chart for toxicity testing using a reference toxicant should show that the LC50 values should fall within 2 standard deviations of the mean.

### 9.2 Quality Assurance/Control Methods

QA/QC procedures for sediment toxicity tests involved sample handling and storage, source and condition of test organisms, condition of facilities and equipment, test conditions, instrument calibration, replication, use of reference toxicants, record keeping, and data evaluation. All organisms used in the tests were disease-free and were positively identified to species. Organisms collected from the field prior to testing were obtained from an area known to be free of toxicants and were held in clean, uncontaminated water and facilities. If greater than five percent of the organisms in holding containers were dead or appeared unhealthy during the 48 hours preceding a test, the entire group was discarded.

The sensitivity of *A. abdita* collected from the field was evaluated with a water-only 96-hour reference toxicant test (sodium dodecyl sulfate (SDS)) performed concurrently with each sediment toxicity test. A control chart was prepared and successive toxicity values were plotted and examined to determine if the results were within prescribed limits. In this technique, a running plot was maintained for the toxicity values from successive tests with a given reference toxicant. For regression analysis results (such as LC50s), the mean and upper and lower control limits (+2 Standard Deviations) were recalculated with each successive point until the statistics stabilized. Values which fell outside the upper and lower control limits and trends of increasing or decreasing sensitivity could be readily identified. At the  $P=0.05$  probability level, one in twenty tests would be expected to fall outside of the control limits by chance alone. If the toxicity value from a given test with the reference toxicant did not fall in the expected range for the test organisms, the sensitivity of the organisms and the overall credibility of the test would be suspect. In this case, the test procedure would have been examined for defects and, if possible, the test would have been repeated with a different batch of test organisms.

A 10-day sediment toxicity test was considered unacceptable if one or more of the following occurred:

1. All test chambers were not identical.
2. Treatments were not randomly assigned to test chambers.
3. Test organisms were not randomly or impartially distributed to test chambers.
4. Required control treatments were not included in the test.
5. All test animals were not from the same population, were not all of the same species or were not a acceptable quality.
6. Amphipods from a wild population were maintained in the laboratory for more than two weeks, unless the effects of prolonged maintenance in the laboratory has been shown to have no significant effect on sensitivity.
7. The test organisms were not acclimated at the test temperature and salinity at least 48 h before they were placed in the test chambers.
8. Temperature, dissolved oxygen, and concentration of test material were not measured or were not within the ranges specified:

Temperature: 20oC+3oC for individual readings, 20oC+1oC time-weighted average temperature at the end of the test, no more than 2oC difference among chambers measured concurrently.

Salinity: 30 ppt.

Dissolved Oxygen: DO concentration was maintained at >90% saturation, should never have dropped below 60% saturation.

9. Aeration to the test chambers was off for an extended time such that dissolved oxygen levels dropped to less than 60 % of saturation.
10. Response criteria were not monitored in a "blind" fashion, i.e., observers had knowledge of the treatment of sediments in the test chambers.
11. Mean percent survival of organisms in control treatments was less than 85% or survival in an individual control test chamber was less than 80%.

### 9.3 Actual Measurement Quality

The laboratory's control chart showed that the LC50 for the standard 48-hour water-only toxicity test using the reference toxicant sodium dodecyl sulfate (SDS) ranged from 5.32 to 8.59 mg/l, with all values falling within two standard deviations of the mean, as required in the QA Plan. Several treatments contained fewer than five replicates, but no infractions were serious enough to warrant discarding data.

All test results were used in EMAP's assessment of the ecological condition of the Province.

#### 9.4 Sources of Error

Factors potentially affecting results from static sediment toxicity tests might include:

- Alteration of field sediments in preparation for laboratory testing. Maintaining the integrity of the sediment environment during its removal, transport, and testing in the laboratory is extremely difficult. The sediment environment is composed of a myriad of microenvironments, redox gradients, and other interacting physicochemical and biological processes. Many of these characteristics influence sediment toxicity and bioavailability to benthic and planktonic organisms, microbial degradation, and chemical sorption. Any disruption of this environment complicates interpretations of treatment effect, causative factors, and in situ comparisons. Testing of sediments at temperatures or salinities other than those at which they were collected might affect contaminant solubility, partitioning coefficients, and other physical and chemical characteristics.
- Interactions between the sediment particles, overlying water, interstitial water, and humic substances, and the sediment to overlying water ratio.
- Interactions among chemicals which may be present in test sediment.
- Photolysis and other processes degrading test chemicals.
- Resuspension of sediment during the toxicity test.
- Natural geochemical properties of test sediment collected from the field which may not be within the tolerance limits of the test organisms.
- Recovery of test organisms from the test system.
- Endemic organisms which may be present in field collected sediments including predators, species which may be the same as or closely related to the test species, or microorganisms (e.g., bacteria, molds) and algae colonizing sediment and test chamber surfaces.

Static tests might not be applicable to materials that are highly volatile or are rapidly biologically or chemically transformed. Furthermore, the overlying water quality may change considerably from the initial overlying water. Because the experimental chambers are aerated, the procedures can usually be applied to materials that have a high oxygen demand. Materials dissolved in interstitial waters might be removed from solution in substantial quantities by adsorption to sediment particles and to the test chamber during the test. The dynamics of contaminant partitioning between solid and dissolved phases at the

initiation of the test should therefore be considered, especially in relation to assumptions of chemical equilibrium.

## 10. DATA ACCESS

### 10.1 Data Access Procedures

Data can be downloaded from the WWW server.

### 10.2 Data Access Restrictions

### 10.3 Data Access Contact Persons

John Paul, Ph.D.  
U.S. EPA NHEERL-AED  
(401) 782-3037 (Tel.)  
(401) 782-3030 (FAX)  
paul.john@epa.gov

Data Librarian EMAP-Estuaries  
U.S. EPA NHEERL-AED  
(401) 782-3184 (Tel.)  
(401) 782-3030 (FAX)  
hughes.melissa@epa.gov

### 10.4 Data Set Format

Data can be downloaded in several formats from the web application and web site.

### 10.5 Information Concerning Anonymous FTP

Not accessible

### 10.6 Information Concerning WWW

Data can be downloaded from the WWW server.

### 10.7 EMAP CD-ROM Containing the Data Set

Data not available on CD-ROM.

## 11. REFERENCES

- Holland, A.F., ed. 1990. Near Coastal Program Plan for 1990: Estuaries. EPA 600/4-90/033. U.S. EPA, NHEERL-AED, Office of Research and Development, Narragansett, RI. November 1990.
- Reifsteck, D.R., Strobel, C.J. and D.J. Keith. 1993. Environmental Monitoring and Assessment Program-Estuaries: 1993 Virginian Province Effort, Field Operations and Safety Manual. U.S. EPA NHEERL-AED, Narragansett, RI. June 1993.

U.S. EPA. 1995. Environmental Monitoring and Assessment Program (EMAP): Laboratory Methods Manual-Estuaries, Volume 1: Biological and Physical Analyses. U.S. Environmental Protection Agency, Office of Research and Development, Narragansett, RI. EPA/620/R-95/008.

Valente, R. and C.J. Strobel. 1993. Environmental Monitoring and Assessment Program-Estuaries 1993 Virginian Province Quality Assurance Project Plan. U.S. EPA NHEERL-AED, Narragansett, RI. May 1993.

12. TABLE OF ACRONYMS

13. PERSONNEL INFORMATION

Virginian Province Manager  
Darryl Keith  
U.S. Environmental Protection Agency  
NHEERL-AED  
27 Tarzwell Drive  
Narragansett, RI 02882-1197  
(401)782-3135 (Tel.)  
(401)782-3030 (FAX)  
keith.darryl@epa.gov

Quality Assurance Officer, Virginian Province  
Charles J. Strobel  
U.S. Environmental Protection Agency  
NHEERL-AED  
27 Tarzwell Drive  
Narragansett, RI 02882-1197  
(401)782-3180 (Tel.)  
(401)782-3030 (FAX)  
strobel.charles@epa.gov

Sample Processing-Principal Investigator  
Dr. K. John Scott  
Science Applications International Corporation, Inc.  
Environmental Testing Center  
165 Dean Knauss Drive  
Narragansett, RI 02882  
(401)782-1900 (Tel.)

John Paul, Ph.D.  
U.S. Environmental Protection Agency  
NHEERL-AED  
Narragansett, RI 02882-1197  
(401) 782-3037 (Tel.)  
(401) 782-3030 (FAX)  
paul.john@epa.gov

Data Librarian, EMAP-Estuaries  
Melissa M. Hughes  
OAO Corporation  
U.S. EPA NHEERL-AED  
Narragansett, RI 02882-1197  
(401) 782-3184 (Tel.)  
(401) 782-3030 (FAX)  
hughes.melissa@epa.gov