

CATALOG DOCUMENTATION EMAP-ESTUARIES PROGRAM LEVEL DATABASE 1991 VIRGINIAN PROVINCE FISH TISSUE CHEMISTRY DATA

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# 1. DATA SET IDENTIFICATION

1.1 Title of Catalog document

EMAP-Estuaries Program Level Database 1991 Virginian Province Fish Tissue Chemistry Data

1.2 Authors of the Catalog entry

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1.3 Catalog revision date

15 March 1996

1.4 Data set name

TISUCHEM

1.5 Task Group

Estuaries

1.6 Data set identification code

00035

1.7 Version

001

1.8 Requested Acknowledgment

These data were produced as part of the U.S. EPA's Environmental Monitoring and Assessment Program (EMAP). 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 has 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 Principal Investigator-Sample Processing

Terry Wade Geochemical & Environmental Research Group Texas A&M University

- 3. DATA SET ABSTRACT
  - 3.1 Abstract of the Data Set

The Tissue Chemistry data set presents the concentrations of a suite of inorganic and organic analytes from tissue samples extracted from one or more target species collected at a station. These species were selected because of their ecological and/or environmental importance. Inorganic and organic concentrations are reported by compound. Organic concentrations are also presented as major group (PCB, DDT and pesticides) totals summed from the individual congeners measured (See Data Manipulations). The concentration for each analyte is reported in mass units (wet weight).

Quality Assurance/Quality Control issues are coded. If the QA code indicates that an analyte could not be measured, the detection limit may be reported. A composite flag indicates if the sample was from an individual sample or from an homogenate of several individuals of the same species.

3.2 Keywords for the Data Set

Contaminants, DDT, fish species, fish tissue, inorganic analytes, organic analytes, PAH, PCB, Pesticides, QA Code, target species, tissue chemistry, tissue contaminants, metals

- 4. OBJECTIVES AND INTRODUCTION
  - 4.1 Project Objective

The Environmental Monitoring and Assessment Program (EMAP) was designed to provide a quantitative assessment of the National extent of environmental problems by measuring status and change in selected indicators of ecological condition. EMAP provides a strategy to identify and bound the extent, magnitude, and location of environmental degradation and improvement on a regional scale.

4.2 Data Set Objective

The specific objective of this investigation was to collect information on the levels of contaminants in fish collected in the estuaries of the Virginian Province.

4.3 Data Set Background Information

Human health concerns about the levels of contaminants in fish have increased over the past decade. To address these concerns on a regional scale, EMAP-VP collected fish in 1991 for chemical analyses. Edible tissue from selected species were analyzed for PCBs, selected pesticides and metals to determine if a significant health risk existed. Because of the poor distribution of individual species across the Province, the low levels of contaminants measured, and the expense of the analyses, this indicator was measured only in 1991.

4.4 Summary of Data Set Parameters

Muscle tissue from an individual or several fish caught in trawls performed at EMAP VP sampling stations were analyzed for PCBs, selected pesticides and metals.

- 5. DATA ACQUISITION AND PROCESSING METHODS
  - 5.1 Data Acquisition
    - 5.1.1 Sampling Objective

To collect fish samples suitable for chemical residue analyses of edible tissue. Fish were collected from one or more fish trawls performed at selected EMAP sampling stations.

## 5.1.2 Sample Collection Methods Summary

Standard fish trawls were performed to collect fish for species composition and relative abundance, tissue chemistry and for pathological examination. Tow duration was ten minutes with a towing speed of 2-3 knots against the prevailing current. Speed over the bottom was 1-3 knots.

All fish in the net were sorted by species and enumerated. All species considered to be rare, threatened or endangered were processed immediately and released alive. Up to thirty individuals of a species were measured (fork length) to the nearest millimeter. Crews were instructed to select the first five individuals of a specific size parameter collected of specific target species for chemical analysis (Table 1). If fewer than five individuals were collected in the standard trawl, additional trawls were performed to collect more fish solely for chemistry. Trawling was repeated for up to two hours until five individuals were collected of each target species present in the trawls.

Fish for chemical analyses were measured, tagged, individually wrapped in aluminum foil and placed, by species, in a zip-lock bag. Samples were then placed immediately on dry ice. For larger fish, the tail was first cut off prior to wrapping the fish in aluminum foil. Care was taken not to penetrate the skin in the mid-section of the fish where the actual sample would be removed from.

| Table 1. Target fis   | sh species and size rar   | iges for chemical  | analyses.   |
|---|---|--|---|
| Common<br>Name  | Scientific<br>Name  | Primary Size<br>Range Size<br>(mm)   | Secondary<br>Range<br>(mm)  |
| Atlantic Croaker<br>Bluefish<br>Channel Catfish<br>Scup<br>Spot<br>Summer Flounder<br>Weakfish<br>White Catfish<br>White Perch<br>Winter Flounder | Micropogonias undulatu<br>Pomatomus saltatrix<br>Ictalurus punctatus<br>Stenotomus chrysops<br>Leiostomus xanthurus<br>Paralichthys dentatus<br>Cynoscion regalis<br>Ameiurus catus<br>Morone americana<br>Pleuronectes americanu | IS 200-300<br>175-225<br>200-300<br>150-200<br>150-250<br>350-450<br>300-400<br>200-300<br>150-250<br>IS 300-400 | 100-200<br>125-175<br>300-400<br>100-150<br>70-150<br>65-350<br>45-300<br>300-400<br>70-150<br>55-300 |
|   |   |  |   |

5.1.3 Beginning sampling Date

22 July 1991

5.1.4 Ending Sampling Date

13 September 1991

5.1.5 Platform

Samples were collected from 8-m (24 ft), twin-engine, Chesapeake style work boats.

# 5.1.6 Sampling Equipment

Fish were collected using a funnel-shaped high rise sampling trawl with a 16-meter footrope with a chain sweep. The trawl net had 5 cm mesh wings and a 2.5 cm cod end.

5.1.7 Manufacturer of Sampling Equipment

Unknown

5.1.8 Key Variables

This data set does not contain values which were measured at the time of sample collection.

5.1.9 Sampling Method Calibration

The sampling gear did not require calibration. It only needed to be inspected to ensure that the net had not been damaged during previous fish trawls.

5.1.10 Sample Collection Quality Control

The first individual collected from each species was shipped to NHEERL-AED for taxonomic verification by an expert. Collection and processing procedures were observed by senior EMAP personnel during field audits to ensure proper procedures were being followed and fish were not exposed to sources of contamination.

5.1.11 Sample Collection Method Reference

Strobel, C.J. and S.C. Schimmel. 1991. Environmental Monitoring and Assessment Program-Near Coastal Component: 1991 Virginian Province Effort Field Operations and Safety Manual. U.S. EPA NHEERL-AED, Narragansett, RI. June 1991.

5.1.12 Sample Collection Method Deviations

The trawls conducted for collection of target species for tissue samples were additional trawls and the data were not included in abundance data.

5.2 Data Preparation and Sample Processing

5.2.1. Sample Processing Objective

To measure the levels of selected contaminants in uncontaminated fish tissue samples collected at EMAP stations.

5.2.2 Sample Processing Methods Summary

Based on the funds available and the distribution of species collected, EMAP-VP provided the analytical laboratory with a list of fish to be composited and analyzed. A total of 84 composites were analyzed as described below. In addition, 40 individual fish were analyzed.

In the laboratory fish were washed in distilled water and the scales removed from those species with scales. Skin was removed ONLY for scaleless species (i.e., catfishes).

Fish were filleted using either a glass or titanium knife. A fillet includes the skin (except for catfish) and edible muscle tissue from the head to tail beginning at the mid-dorsal line and continuing down to the belly flap from the left side of each fish. Bones were carefully removed.

Except for fish without scales, skin-on fillets were analyzed for contaminants because EMAP-E believes that this is how most people prepare and consume fish.

Fish were then composited to create the sample analyzed for the contaminants listed in Table 2. A composite consisted of tissue from three to five individuals of a single species collected at a station. All individuals in a composite were of a similar size, with the smallest individual being no less than 75% the length of the largest.

Fillet composites were homogenized via high-speed blender or homogenizer until no chunks remained. Homogenizer blades were constructed of titanium.

Samples for metals analyses were hot nitric acid-extracted, and analyzed via ICP, graphite furnace atomic absorption spectrometry, or cold vapor atomic absorption spectrometry (Hg).

Samples for PCB and pesticide analyses were soxhlet-extracted using methylene chloride and analyzed via GC-MS or GC-ECD. All results are reported on a wet weight basis.

Table 2. List of analytes.

METALS (ug/g wet weight) PCB CONGENERS (ng/g wet weight) PCB8 Ag Al PCB18 PCB28 As Cd PCB44 Cr PCB52 Cu PCB66 Fe PCB101 PCB105 Hg Mn PCB110/77 Ni PCB118 Pb **PCB126** Se **PCB128** Sn PCB138 Zn PCB153 PCB170 PCB180 PCB187 PCB195 **PCB206 PCB209** 

CHLORINATED PESTICIDES (ng/g wet weight)

Aldrin Alpha-Chlordane Trans-Nonachlor Dieldrin Endrin Heptachlor epoxide Lindane (gamma-BHC) Mirex 2,4'-DDE 4,4'-DDE 2,4'-DDD 4,4'-DDD 2,4'-DDT 4,4'-DDT

5.2.3 Sample Processing Method Calibration

NA

5.2.4 Sample Processing Quality Control

To prevent contamination, fish were washed in distilled water and filleted using either a glass or titanium knife. Homogenizer blades were constructed of titanium.

5.2.5 Sample Processing Method Reference

Not available.

5.2.6 Sample Processing Method Deviations

NA

6. DATA MANIPULATIONS

Summary values were calculated for groups of total organic concentrations. The values under a summed analyte are the sum of the values of a specific set of compounds.

6.1 Name of new or modified values

TOT PCB, TOT DDT, TOT PEST

#### 6.2 Data Manipulation Description

Total concentrations were summed for specific suites of compounds or congeners. These include totals for the following groups: PCBs (TOT\_PCB), DDTs (TOT\_DDT) and pesticides (TOT\_PEST). The summed concentrations exclude analytes having a QA\_CODE of SC-A. This code indicates that the analyte was not detected in a particular sample. Set groups of compounds were summed to have consistency across Provinces.

# 6.3 Data Manipulation Examples

TOT\_DDT = Sum of concentrations of OPDDE, OPDDD, OPDDT, PPDDE, PPDDD and PPDDT for a station

TOT\_PCB = Sum concentrations of congeners for a station: 8, 18, 28, 52, 44, 66, 101, 118, 153, 105, 138, 187, 128, 180, 170, 195, 206 and 209

TOT\_PEST = Sum concentrations of the following pesticides for a station: Dieldrin, Lindane, Aldrin, Mirex, Heptachlor, Heptachlor epoxide, Alpha-chlordane, Hexachlorobenzene and Trans-Nonachlor

## 7. DATA DESCRIPTION

7.1 Description of Parameters

| 1 STA_NAMEChar88.The Station Identifier2 VST_DATENum8 YYMMDD6.The Date the Sample was Collected3 SAMPTYPEChar10\$10.Organismal Derivation of Sample Mat4 SPECCODEChar9\$8.EMAP Taxon Code5 COMPOSITChar3\$3.Composite Code (Y/N)6 NUM_CMPTNum83.Count (#) of Organisms in Composite7 ANALYTEChar88.Analyte Code8 CONCNum813.6Concentration of Analyte (wet wt.)9 CHMUNITSChar15150uality Assurance Code for Data | Parameter<br># SAS Name  | Data<br>Type Leng  | gth Format   | Parameter<br>Label  |     |
|--|--|--|--|---|-----|
| 11 DETLIMITNum813.6MethodDetectionLimitforAnalyte12 TOT_ANALNum83.Analytes (#)Included in Summed Cor13 ANAL_CATChar1515.GeneralCategory for Group of Analy   | 1 STA_NAME<br>2 VST_DATE<br>3 SAMPTYPE<br>4 SPECCODE<br>5 COMPOSIT<br>6 NUM_CMPT<br>7 ANALYTE<br>8 CONC<br>9 CHMUNITS<br>10 QA_CODE<br>11 DETLIMIT<br>12 TOT_ANAL<br>13 ANAL_CAT | Char 8<br>Num 8<br>Char 10<br>Char 9<br>Char 3<br>Num 8<br>Char 8<br>Num 8<br>Char 12<br>Char 15<br>Num 8<br>Num 8<br>Num 8<br>Char 15 | 8.<br>YYMMDD6.<br>\$10.<br>\$8.<br>\$3.<br>3.<br>8.<br>13.6<br>12.<br>15.<br>13.6<br>3.<br>15. | The Station Identifier<br>The Date the Sample was Collected<br>Organismal Derivation of Sample Mater<br>EMAP Taxon Code<br>Composite Code (Y/N)<br>Count (#) of Organisms in Composite<br>Analyte Code<br>Concentration of Analyte (wet wt.)<br>Conc. Units (ug/g or ng/g)<br>Quality Assurance Code for Data<br>Method Detection Limit for Analyte<br>Analytes (#) Included in Summed Conc.<br>General Category for Group of Analyte | ial |

7.1.6 Precision to which values are reported

All concentrations are rounded to three significant figures.

7.1.7 Minimum Value in data set

| ANALYTE  | Minimum Va | alue |
|----------|------------|------|
| AG       | 0.00231    | l    |
| AL       | 2.66000    | )    |
| ALDRIN   | 0.01890    | )    |
| AS       | 0.03530    | )    |
| CD       | 0.00121    | l    |
| CISCHL   | 0.01730    | )    |
| CR       | 0.01180    | )    |
| CU       | 0.14500    | )    |
| DDT TOT  | 0.32600    | )    |
| DIELDRIN | 0.03620    | )    |
| FE       | 1.49000    | )    |
| HEPTACHL | 0.01920    | )    |
| HEPTAEPO | 0.01910    | )    |
| HEXACHL  | 0.01700    | )    |
| HG       | 0.00286    | 5    |
| LINDANE  | 0.01700    | )    |
| MIREX    | 0.01950    | )    |

| ANALYTE  | Minimum  | Value   |
|--|--|---|
| NI<br>OPDDD<br>OPDDE<br>OPDDT<br>PB<br>PCB101<br>PCB105<br>PCB118  | 0.019<br>0.017<br>0.017<br>0.019<br>0.008<br>0.018<br>0.018<br>0.039   | 930<br>730<br>730<br>950<br>959<br>910<br>970<br>910                                    |
| PCB128<br>PCB138   | 0.018  | 310<br>100  |
| PCB138<br>PCB153<br>PCB170<br>PCB18<br>PCB180<br>PCB187<br>PCB195<br>PCB206<br>PCB209<br>PCB28<br>PCB28<br>PCB44<br>PCB52<br>PCB66 | 0.271<br>0.145<br>0.018<br>0.054<br>0.036<br>0.036<br>0.018<br>0.018<br>0.015<br>0.015<br>0.015<br>0.015<br>0.015<br>0.015<br>0.015<br>0.015<br>0.015<br>0.036<br>0.018<br>0.018<br>0.018<br>0.018<br>0.036<br>0.036<br>0.018<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0.036<br>0. | 100<br>500<br>310<br>440<br>520<br>520<br>310<br>310<br>730<br>910<br>990<br>970<br>920 |
| PCB8<br>PCB TOT  | 0.287  | 300   |
| PEST_TOT   | 0.072  | 240   |
| PPDDD<br>PPDDE   | 0.018  | 500<br>500  |
| PPDDT  | 0.018  | 360   |
| SE   | 0.091  | 190   |
|  | 0.120  | 520   |
| ZN   | 1.930  | 000   |

# 7.1.8 Maximum Value in data set

| ANALYTE  | Maximum Value |
|----------|---------------|
| AG       | 0.03          |
| AL       | 15.60         |
| ALDRIN   | 0.68          |
| AS       | 5.52          |
| CD       | 0.08          |
| CISCHL   | 63.90         |
| CR       | 1.95          |
| CU       | 3.11          |
| DDT_TOT  | 1490.00       |
| DIELDRIN | 52.80         |
| FE       | 20.30         |
| HEPTACHL | 0.23          |
| HEPTAEPO | 5.76          |
| HEXACHL  | 2.01          |
| HG       | 0.26          |
| LINDANE  | 1.53          |
| MIREX    | 1.20          |
| NI       | 0.86          |

| ANALYTE  | Maximum Value |  |
|----------|---------------|--|
| OPDDD    | 118.00        |  |
| OPDDE    | 100.00        |  |
| OPDDT    | 10.90         |  |
| PB       | 0.07          |  |
| PCB101   | 133.00        |  |
| PCB105   | 27.30         |  |
| PCB118   | 80.40         |  |
| PCB128   | 24.20         |  |
| PCB138   | 216.00        |  |
| PCB153   | 412.00        |  |
| PCB170   | 70.20         |  |
| PCB18    | 66.00         |  |
| PCB180   | 181.00        |  |
| PCB187   | 107.00        |  |
| PCB195   | 46.00         |  |
| PCB206   | 80.10         |  |
| PCB209   | 82.30         |  |
| PCB28    | 64.60         |  |
| PCB44    | 79.00         |  |
| PCB52    | 175.00        |  |
| PCB66    | 48.60         |  |
| PCB8     | 85.60         |  |
| PCB_TOT  | 1180.00       |  |
| PEST_TOT | 156.00        |  |
| PPDDD    | 532.00        |  |
| PPDDE    | 707.00        |  |
| PPDDT    | 25.80         |  |
| SE       | 1.55          |  |
| SN       | 0.12          |  |
| TNONCHL  | 60.30         |  |
| ZN       | 37.70         |  |

7.2 Data Record Example

7.2.1 Column Names for Example Records

STA\_NAME VST\_DATE SAMPTYPE SPECCODE COMPOSIT NUM\_CMPT ANALYTE CONC CHMUNITS DETLIMIT QA\_CODE TOT\_ANAL ANAL\_CAT

# 7.2.2 Example Data Records

| VA91-261 910803<br>ug/g 0.002830 | FISH<br>CH-A | PARADENT<br>. METAL | Y         | 5 | AG     |          |
|----------------------------------|--------------|---------------------|-----------|---|--------|----------|
| VA91-261 910803<br>ug/g 2.130000 | FISH<br>CH-A | PARADENT<br>. METAL | Y         | 5 | AL     |          |
| VA91-261 910803<br>ng/g 0.089400 | FISH<br>CH-A | PARADENT<br>. PESTI | Y<br>CIDE | 5 | ALDRIN |          |
| VA91-261 910803<br>ug/g .        | FISH         | PARADENT<br>. METAL | Y         | 5 | AS     | 0.662000 |

# 8. GEOGRAPHIC AND SPATIAL INFORMATION

8.1 Minimum Longitude

-77 Degrees 19 Minutes 30.00 Decimal Seconds

8.2 Maximum Longitude

-70 Degrees 01 Minutes 00.00 Decimal Seconds

8.3 Minimum Latitude

36 Degrees 56 Minutes 24.60 Decimal Seconds

8.4 Maximum Latitude

42 Degrees 08 Minutes 00.00 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 and the states of Virginia, Maryland, Delaware, Pennsylvania, New Jersey, New York, Connecticut, Rhode Island and Massachusetts.

- 9.0 QUALITY CONTROL AND QUALITY ASSURANCE
  - 9.1 Measurement Quality Objectives

Measurement Quality Objectives (MQOs) for the 1991 Virginian Province fish chemistry analyses were defined in the 1991 Virginian Province Quality Assurance Project Plan (Valente and Schoenherr, 1991). This plan required each laboratory to analyze the following quality control (QC) samples along with every batch or "set" of samples: laboratory reagent blank, calibration check standards, matrix spike/matrix spike duplicate, and Laboratory Control Material (LCM). Results for these QC samples had to fall within certain pre-established control limits for the analysis of a batch of samples to be considered acceptable.

# 9.2 Quality Assurance/Control Methods

# 9.2.1 Inorganic Analyses

For the 1991 Virginian Province analysis of major and trace elements, the laboratory generally met the pre-established acceptability criteria (control limits) for the QC samples (e.g., calibration check samples, laboratory reagent blanks, matrix spikes, and Laboratory Control Materials). The control limits for inorganic analytes is ± 20% of the CRM certified value. These criteria were generally met (Table 3). The average percent recovery for Pb (DOLT) was slightly high; however, the value for the DORM CRM was within the acceptable range and the confidence intervals around the DOLT certified value were rather large.

A problem was noted by the laboratory in analysis of selected samples for mercury. The laboratory analyzed 84 composite samples and 40 individual fish. The analytical laboratory experienced a mercury-contamination

problem with their freeze-drier, resulting in contamination of all 40 individual-fish samples. As a result, these data had to be deleted from the database. However, EMAP-VP's assessment has focused on the composite samples, and none of these were contaminated.

With the removal of the above-mentioned Hg data from the database, the only flags applied are the "A" and "B" codes.

# 9.2.2 Organic Analyses

Due to a miscommunication within the analytical laboratory, EMAP QA protocols were not followed during the analysis of EMAP-VP 1991 fish tissue samples for organic analytes. However, sufficient data are available for the analysis of the quality of those samples. First, prior to beginning the processing of EMAP samples, the laboratory participated in a performance evaluation. Based on 11 separate analyses of SRM 1974 (Organics in Mussel Tissue), it was determined that the laboratory was sufficiently proficient to begin analyzing EMAP samples. The results of this performance evaluation are listed in Table 4. Second, matrix spiked samples were analyzed with each batch, and these results fall well within EMAP's control limits (Table 5). Third, during the same time period when the laboratory was processing EMAP samples, they were also processing samples for NOAA's NS&T Program. SRM 1974 was used as the laboratory control material for those samples, and was analyzed with each analytical batch. The laboratory has provided EMAP with those results, which fall within EMAP control limits. Fourth, the QA protocols the lab followed for EMAP samples require the analysis of duplicate samples with each batch. Those results were provided to EMAP and showed excellent precision, with a maximum Relative Percent Difference for an analyte in a given set generally being less than 10%.

The only flags applied are the "A" and "B" codes.

# Table 3. Summary results for CRMs DOLT and DORM (Dogfish liver and muscle tissue, respectively) used as a set control for the 1991 Virginian Province fish tissue inorganic analyses.

| nent | Average1   | Stdv2   | C.V.3   | Min.4  | Max.5  |   |
|------|--|---|---|--|--|---|
| DOLT | 101.2  | 2.5   | 2.5   | 98.0   | 104.0  |   |
| DORM | 99.3   | 2.5   | 2.5   | 94.9   | 101.7  |   |
| DOLT | 83.3   | 8.2   | 9.8   | 69.6   | 90.9   |   |
| DORM | 93.0   | 10.4  | 11.2  | 81.4   | 104.7  |   |
| DOLT | 118.8  | 15.2  | 12.8  | 102.5  | 137.5  |   |
| DORM | 106.3  | 8.8   | 8.3   | 97.5   | 117.5  |   |
| DOLT | 91.4   | 9.1   | 10.0  | 81.7   | 107.2  |   |
| DORM | 79.4   | 5.3   | 6.7   | 75.7   | 88.9   |   |
| DOLT | 98.9   | 1.8   | 1.8   | 96.2   | 101.3  |   |
| DORM | 105.7  | 11.9  | 11.2  | 95.4   | 125.0  |   |
| DOLT | NA   | NA  | NA  | NA   | NA   |   |
| DORM | 91.9   | 4.9   | 5.3   | 86.5   | 100.3  |   |
| DOLT | 103.8  | 52.9  | 50.9  | 50.0   | 188.5  |   |
| DORM | 89.0   | 13.5  | 15.2  | 76.7   | 111.7  |   |
| DOLT | 130.4  | 25.7  | 19.7  | 92.7   | 164.7  |   |
| DORM | 89.5   | 35.3  | 39.5  | 55.0   | 142.5  |   |
| DOLT | 102.1  | 2.9   | 2.8   | 99.7   | 107.4  |   |
|      | nent<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT<br>DORM<br>DOLT | Average1   DOLT 101.2   DORM 99.3   DOLT 83.3   DORM 93.0   DOLT 118.8   DORM 106.3   DOLT 91.4   DORM 79.4   DOLT 98.9   DORM 105.7   DOLT NA   DORM 91.9   DOLT 103.8   DORM 89.0   DOLT 130.4   DORM 89.5   DOLT 102.1 | nentAverage1Stdv2DOLT101.22.5DORM99.32.5DOLT83.38.2DORM93.010.4DOLT118.815.2DORM106.38.8DOLT91.49.1DORM79.45.3DOLT98.91.8DORM105.711.9DOLTNANADORM91.94.9DOLT103.852.9DORM89.013.5DOLT130.425.7DORM89.535.3DOLT102.12.9 | MentAverage1Stdv2C.V.3DOLT101.22.52.5DORM99.32.52.5DOLT83.38.29.8DORM93.010.411.2DOLT118.815.212.8DORM106.38.88.3DOLT91.49.110.0DORM79.45.36.7DOLT98.91.81.8DORM105.711.911.2DOLTNANANADORM91.94.95.3DOLT103.852.950.9DORM89.013.515.2DOLT130.425.719.7DORM89.535.339.5DOLT102.12.92.8 | nentAverage1Stdv2C.V.3Min.4DOLT101.22.52.598.0DORM99.32.52.594.9DOLT83.38.29.869.6DORM93.010.411.281.4DOLT118.815.212.8102.5DORM106.38.88.397.5DOLT91.49.110.081.7DORM79.45.36.775.7DOLT98.91.81.896.2DORM105.711.911.295.4DOLTNANANANADORM91.94.95.386.5DOLT103.852.950.950.0DORM89.013.515.276.7DOLT130.425.719.792.7DORM89.535.339.555.0DOLT102.12.92.899.7 | MentAverage1Stdv2C.V.3Min.4Max.5DOLT101.22.52.598.0104.0DORM99.32.52.594.9101.7DOLT83.38.29.869.690.9DORM93.010.411.281.4104.7DOLT118.815.212.8102.5137.5DORM106.38.88.397.5117.5DOLT91.49.110.081.7107.2DORM79.45.36.775.788.9DOLT98.91.81.896.2101.3DORM105.711.911.295.4125.0DOLTNANANANADORM91.94.95.386.5100.3DOLTNANANANADORM91.94.95.386.5100.3DOLTNANANANADORM91.94.95.386.5100.3DOLT103.852.950.950.0188.5DORM89.013.515.276.7111.7DOLT130.425.719.792.7164.7DORM89.535.339.555.0142.5DOLT102.12.92.899.7107.4 |

| DORM J<br>Zn DOLT J<br>DORM J   | 102.84.101.72.100.73.   | 6 4.4<br>7 2.7<br>9 3.8  | 96.9<br>98.0<br>96.2   | 108.0<br>105.7<br>106.6                       |                            |  |  |  |
|---|---|--|--|---|----------------------------|--|--|--|
| Average percent recovery relative to the CRM certified value.<br>Standard deviation of the percent recovery values.<br>Coefficient of variation of the percent recovery values.<br>Minimum percent recovery for analysis sets<br>Maximum percent recovery for analysis sets |   |  |  |   |                            |  |  |  |
| Table 4. Perfor<br>in tissu<br>analyses<br>differer   | mance evalua<br>ue. Average r<br>of SRM 1974<br>nt days.  | tion result<br>eported val<br>(Organics  | s for analysi<br>ues are based<br>in Mussel Tis                                  | s of organic<br>on 11 separa<br>sue) performe | contaminants<br>te<br>d on |  |  |  |
| Analyte   | Average<br>reported<br>value  | NIST non-<br>certified<br>value1   | Percent<br>difference  |   |                            |  |  |  |
| alpha-chlordane<br>trans-nonachlor<br>Dieldrin<br>2,4'-DDE<br>4,4'-DDE<br>2,4'-DDD<br>4,4'-DDD<br>2,4'-DDT<br>4,4'-DDT  | 21.2<br>17.7<br>11.3<br>M2<br>41.4<br>5.8<br>46.5<br>5.0<br>3.6   | 25<br>17.7<br>11.3<br>5.8<br>46<br>13<br>65<br>5.0<br>3.6                                | -15%<br>0%<br>0%<br>NA<br>-10%<br>-55%<br>-28%<br>0%<br>0%                       |   |                            |  |  |  |
| PCB 18<br>PCB 28<br>PCB 44<br>PCB 52<br>PCB 66<br>PCB 101<br>PCB 105<br>PCB 118<br>PCB 128<br>PCB 128<br>PCB 138<br>PCB 153<br>PCB 180<br>PCB 187   | 20.9<br>85.2<br>72.4<br>113.7<br>98.7<br>127.0<br>46.9<br>115.9<br>17.3<br>122.2<br>153.9<br>13.3<br>27.2 | 20.9<br>65<br>72.4<br>113.7<br>105<br>116<br>48<br>115<br>17<br>121<br>153<br>13.3<br>29 | 0%<br>31%<br>0%<br>0%<br>-6%<br>9%<br>-2%<br>1%<br>2%<br>1%<br>1%<br>0%<br>-6.2% |   |                            |  |  |  |

1 NIST non-certified values are adjusted based on the 95% confidence intervals presented in the certificate of analysis for SRM 1974. Reported values falling within these confidence intervals are listed as having a percent difference of 0%.

2 Matrix interference, no peak was found for 2,4'-DDE

Table 5. Results of laboratory-fortified matrix spikes analyzed with each batch of fish tissue organic samples analyzed (n=10). Values are percent recovery of the spike.

| Average1   | Stdv2  | C.V.3  | Min.4   | Max.5  |  |
|--|--|--|---|--|--|
| 95.2<br>100.2<br>99.3<br>95.2<br>94.4<br>99.1<br>101.6<br>98.7<br>101.9<br>100.5 | 10.9<br>10.9<br>12.1<br>14.0<br>9.6<br>10.5<br>9.3<br>12.7<br>12.5                                   | 11.5<br>10.9<br>12.2<br>14.6<br>10.2<br>10.6<br>9.2<br>12.9<br>12.3<br>15 1                            | 83<br>82<br>80<br>71<br>84<br>86<br>87<br>76<br>79<br>74  | 114<br>112<br>117<br>118<br>112<br>118<br>112<br>118<br>112<br>118<br>120<br>118   |  |
| 99.8   | 7.7  | 7.7  | 87  | 114  |  |
|  | Average1<br>95.2<br>100.2<br>99.3<br>95.2<br>94.4<br>99.1<br>101.6<br>98.7<br>101.9<br>100.5<br>99.8 | Average1Stdv295.210.9100.210.999.312.195.214.094.49.699.110.5101.69.398.712.7101.912.5100.515.299.87.7 | Average1Stdv2C.V.395.210.911.5100.210.910.999.312.112.295.214.014.694.49.610.299.110.510.6101.69.39.298.712.712.9101.912.512.3100.515.215.199.87.77.7 | Average1Stdv2C.V.3Min.495.210.911.583100.210.910.98299.312.112.28095.214.014.67194.49.610.28499.110.510.686101.69.39.28798.712.712.976101.912.512.379100.515.215.17499.87.77.787 | Average1Stdv2C.V.3Min.4Max.595.210.911.583114100.210.910.98211299.312.112.28011795.214.014.67111894.49.610.28411299.110.510.686118101.69.39.28711298.712.712.976118101.912.512.379120100.515.215.17411899.87.77.787114 |

1 Average percent recovery relative to the concentration of the spike.

2 Standard deviation of the percent recovery values.

3 Coefficient of variation of the percent recovery values.

4 Minimum percent recovery for analysis sets

5 Maximum percent recovery for analysis sets

## 9.3 Actual Measurement Quality

The laboratory generally met the pre-established acceptability criteria (control limits) for the QC samples (e.g., calibration check samples, laboratory reagent blanks, matrix spikes and LCMs). The control limits for inorganic analytes is +20% of the CRM certified value. These criteria were generally met. The average percent recovery for Pb (DOLT) was slightly high; however, the value for the DORM CRM was within the acceptable range and the confidence intervals around the DOLT certified value were rather large.

A problem was noted by the laboratory in analysis of selected samples for mercury. The laboratory analyzed 84 composite samples and 40 individual fish. The analytical laboratory experienced a mercury-contamination problem with their freeze-drier, resulting in contamination of all 40 individual-fish samples. As a result, these data had to be deleted from the database. However, EMAP-VP's assessment was focused on the composite samples, and none of these were contaminated.

With the removal of the above-mentioned Hg data from the database, the only flags applied are listed below:

#### 9.2.3 Data Qualifier Codes

Two data qualifier codes or "flags" are used in the 1991 Virginian Province fish chemistry dataset:

### SC-A CODE

The "SC-A" code indicates that an analyte was not detected. When the "SC-A" code is used, the concentration field is left blank and the detection limit for the analyte in that particular sample is reported under the variable "MDL" (method detection limit).

#### SC-B CODE

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 "SC-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.

9.4 Sources of Error

None

- 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

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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

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- Strobel, C.J. and S.C. Schimmel. 1991. Environmental Monitoring and Assessment Program-Near Coastal Component: 1991 Virginian Province Effort Field Operations and Safety Manual. U.S. EPA, NHEERL-AED, Narragansett, RI. June 1991.
- Valente, R. and J. Schoenherr. 1991. Environmental Monitoring and Assessment Program-Near Coastal Virginian Province: Quality Assurance Project Plan. U.S. EPA NHEERL-AED, Narragansett, RI. July 1991.
- 12. TABLE OF ACRONYMS
- 13. PERSONNEL INFORMATION

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