CATALOG DOCUMENTATION
EMAP-ESTUARIES PROGRAM LEVEL DATABASE
LOUISIANIAN PROVINCE 1991-1994
FISH/INVERTEBRATE TISSUE CHEMISTRY

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1. DATA SET IDENTIFICATION

1.1 Title

EMAP-Estuaries Province Level Database
Louisianian Province
Tissue Chemistry Data

1.2 Catalog Author

Virginia Engle, U.S. Environmental Protection Agency - NHEERL/GED
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1.3 Catalog Revision Date

March 18, 1999

1.4 Data File Name

TISUCHEM

1.5 Task Group

ESTUARIES

1.6 Data set identification code

00055, 00095, 00135, 00175

1.7 Version number for a data set

003, 003, 004, 002

1.8 Requested acknowledgment

If you plan to publish these data in any way, EPA requires a standard statement for work is 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

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2.2 Sample Collection Investigator

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2.4 Data Analysis Investigator

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BRD - GBPO

2.5 Additional Investigators

N/A

3. DATA SET ABSTRACT

3.1 Abstract of the Data Set

The tissue chemistry data set presents the concentrations of a suite of organic and inorganic analytes extracted from the tissue of a target species (a pre-determined list of fish and/or invertebrate species of ecological and/or environmental importance) collected at a station. There is one (1) record for each analyte measured in a sample. A code for each compound is given under ANALYTE. These include inorganics, PCBs, and pesticides. Individual and summed analyte concentrations are presented. The concentration for each analyte is reported in mass units on wet weight basis. Units are reported under a separate attribute, CHMUNITS, as ug/g, ng/g or %. Quality Assurance/Quality Control issues are coded. Depending on the QA code, only a detection limit may be reported. Each taxon is identified by a unique code that can be cross-referenced to the taxon phylogeny. A "type" code indicates a general category of organism (e.g., fish or shrimp) from which the tissue was sampled.

3.2 Keywords for the Data Set

Contaminants, DDT, metals, inorganic analytes, organic analytes, PCB, pesticides, QA Code, fish tissue, tissue chemistry,

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 the randomly located Base Sampling Sites were included in this data set.

4.2 Data Set Objective

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

4.3 Data Set Background Information

Human health concerns about the levels of contaminants in fish and invertebrates have increased over the past decade. To address these concerns on a regional scale, the Louisianian Province collected fish and invertebrates in 1991-1994 for chemical analyses. Edible tissue from selected species were analyzed for PCBs, selected pesticides, and metals to determine if a significant health risk existed.

4.4 Summary of Data Set Parameters

Muscle tissue from fish and invertebrates caught in trawls performed at sampling stations was analyzed for PCBs, selected pesticides, and metals. The organic and inorganic compound concentrations measured generally included: 13 major and trace elements, the pesticide, DDT, and its metabolites, 12 pesticides other than DDT, 21 individual Poly-Chlorinated Biphenyl (PCB) congeners, and the butyltins (MBT, DBT, TBT). This suite of analytes is similar to that measured in the National Oceanic and Atmospheric Administration's (NOAA) National Status and Trends (NS&T) program. Values in this data file include individual inorganic and organic compound concentrations and concentrations summed for several major groups: total PCBs, total DDTs, total chlorinated pesticides. Concentrations of all tissue chemistry analytes are reported on a wet weight basis.

4.5 Year-Specific Information about Data

In 1991, only one fish trawl was designed into the station sampling schema. Occasionally, however, circumstances prevented the completion of one successful trawl resulting in no fish collection data or tissue chemistry samples for that particular station. Beginning in 1992, the Louisianian Province allowed for up to three fish trawls per stations and averaged the indicators between the first of two successfully completed trawls. This increased the chances that nekton specific data would be more accurately represented and tissue chemistry samples would be available for each site. Occasionally, however, a field crew would conduct more than three (3) trawls in order to obtain enough tissue samples for chemistry analysis. Any trawl conducted after the first three (3) attempts was not used for any of the summary calculations. The actual number of trawls taken for each stations is reflected in the Fish Abundance data file.

Tissue samples were obtained from target species only. In 1991, the list of target species included: 4 species of catfish, penaeid shrimp, Atlantic croaker, spot, pinfish, menhaden, and sand seatrout In 1992 -1994, this list of target species was reduced to include only the catfish, shrimp, and croaker. Also, butyltins were only measured in tissue sampled in 1992- 1994.

5. METHODS

5.1 Data Acquisition

5.1.1 Sampling Objective

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

5.1.2 Sample Collection Methods Summary

A balloon trawl (funnel-shaped net) was deployed from the sampling vessel using a hydraulic powered boom and winch system and dragged over the bottom in the general vicinity of

the sampling station to capture bottom and near-bottom fishes and crustaceans. The duration of a trawl was 10 +- 2 minutes and the rate of speed over bottom was 2-3 knots. Following a successful trawl, the net was hauled aboard and the catch was released into a plastic trough or fish sorting table.

Crews were instructed to select the first five individuals collected of specific target species (see Appendix B Table 1) for chemical analysis. Individuals should have been within a 20-40 cm range. If fewer than five individuals were collected in the standard trawl, a third trawls was performed to collect more organisms solely for acquiring enough sample tissue for chemistry analysis. The crew chief determined the duration of a third trawl if conducted at a site. The community structure (See Fish Abundance, Species and Community) does not include any organisms collected during a third trawl. Occasionally, even a third trawl did not result in enough tissue sample for a full complement of analyses.

After fish/shrimp for chemical analyses were measured, whole organisms were placed in zip-lock bags with one species per bag. The number of specimens per species placed in each bag was contingent on the size of the fish. Samples were then placed immediately on wet ice for transport back to the mobile field laboratory where they were frozen on dry ice prior to shipment to the destination lab.

5.1.3 Beginning Sampling Date

09 July 1991
08 July 1992
06 July 1993
06 July 1994

5.1.4 Ending Sampling Date

10 September 1991
11 September 1992
19 August 1993
15 September 1994

5.1.5 Sampling Platform

Each team was supplied with a 25-foot SeaArk work boat equipped with a 7.5 L gas engine fitted with a Bravo outdrive, an "A" frame boom assembly and hydraulic winch. On-board electronics consist of: a Loran C unit, GPS (beginning in 1993), radar unit, 2 VHF radios, cellular phone, compass, a depth finder, a tool kit, and all required and suggested safety equipment. One completely outfitted spare boat was stored at the Field Operations Center (EPA Lab) as backup.

In 1992, a vessel designed specifically for shallow water conditions was put into service in the Louisianian Province to sample at stations with depths less than 3 feet.

5.1.6 Sampling Equipment

The net used was a 4.9 m (16 ft) -wide, balloon (high profile) trawl with 2.5 cm (1 in) stretched mesh in the bosom, wings, and cod end; no liner was used. The trawl was equipped with 41 X 76 cm (16 X 30 in) wooded doors.

5.1.7 Manufacturer of Sampling Equipment

5.1.8 Key Variables

5.1.9 Sampling Method Calibration

The sampling equipment required no calibration. It only needed to be inspected to insure that the net had not been damaged during previous trawls.

5.1.10 Sample Collection Quality Control

If the trawl was successful and fish were caught, the specimens designated for chemistry or pathology analysis were contained appropriately for shipping to various labs. Each species of fish for a particular station were tracked using a barcode system. As the field crew prepared the specimens for shipping, the fish would be grouped by species and type of lab analyses needed then tagged with a waterproof barcode label bearing a unique identification number. A duplicate barcode was place on the appropriate data sheet. Each barcode label was scanned into a data file using laser barcode readers. This method of tagging provided the EMAP-E team an efficient, accurate and viable accounting of fish shipped to laboratories for further analysis. The laboratories were also supplied with barcode readers so fish received by lab personnel could be documented. The lab receiving files were electronically forwarded to EMAP-E for shipping and receiving reconciliation.

Additionally, periodic field visits were conducted by the QA Officer, Province Manager or other designee(s) throughout the sampling season to ensure proper identification, enumeration, measurement and packaging techniques were being used.

5.1.11 Sample Collection Method Reference

Macauley, J.M. 1991. Environmental Monitoring and Assessment Program-Near Coastal Louisianian Province: 1991 Monitoring Demonstration. Field Operations Manual. EPA/600/X-91/XXX. U.S. Environmental Protection Agency, Office of Research and Development, Environmental Research Laboratory, Gulf Breeze, FL 32561.

Macauley, J.M. 1992. Environmental Monitoring and AssessmentProgram: Louisianian Province:1992 Sampling: Field Operations Manual. EPA/ERL-GB No. SR-119. U.S. Environmental Protection Agency, Office of Research and Development, Environmental Research Laboratory, Gulf Breeze, FL 32561.

Macauley, J.M. 1993. Environmental Monitoring and Assessment Program: Louisianian Province:1993 Sampling: Field Operations Manual. EPA/ERL-GB No. SR-XXX. U.S. Environmental Protection Agency, Office of Research and Development, Environmental Research Laboratory, Gulf Breeze, FL 32561.

Macauley, J.M. 1994. Environmental Monitoring and Assessment Program: Louisianian Province:1993 Sampling: Field Operations Manual. EPA/ERL-GB No. SR-XXX. U.S. Environmental Protection Agency, Office of Research and Development, Environmental Research Laboratory, Gulf Breeze, FL 32561.

5.1.12 Sample Collection Method Deviations

None

5.2 Data Preparation and Sample Processing

5.2.1. Data Preparation Objective

To measure the levels of selected contaminants in fish and invertebrate composite samples collected at EMAP stations.

5.2.2 Data Processing Methods Summary

The analyses of contaminants in 1991-1993 tissue samples were all conducted by the same laboratory; the 1994 sample were conducted by a different laboratory. The two laboratories used similar methods to prepare and analyze the samples; any significant differences will be discussed.

In the laboratory, the sample of composited fish/shrimp was removed from the freezer and allowed to thaw. The sample was rinsed with distilled water and, when available, five individuals were selected for tissue analysis. A composite of five individuals was considered the ideal sample size; however, at times, less than five individuals were available and at other times, when the fish/shrimp were small, the composited sample size was increased to more than five individuals in order to provide an adequate volume of tissue for the analyses.

The scales were removed from those species with scales. Fish were filleted using either a ceramic or titanium bladed knife. A fillet included the skin (except for catfish) and edible muscle tissue from just posterior of the gills to the tail area and laterally, from the mid-dorsal line and continuing down to the belly flap. Any bones were carefully removed from the filleted tissue. for shrimp samples, only the tail muscle was taken for analysis and the shells were also removed. The sample preparations were meant to emulate the manner in which most people are believed to prepare the respective species for human consumption.

The sample of fillets from the composited fish was cut into a very small dice/mince using the ceramic or titanium blade and then homogenized as uniformly as possible before being split

into separate aliquots for organic and inorganic analyses. The use of a tissue homogenizer was avoided at this point to prevent possible contamination to the inorganic fraction from the stainless steel blades (a titanium bladed homogenizer was not available to the laboratory).

For 1991-1993 samples, the aliquot for inorganic analysis was microwave digested in HNO3/HCl then analyzed by flame atomic absorption (AA) or graphite furnace AA spectrophotometry (cold vapor AA was used for Hg analysis). The 1994 samples were microwave digested in HNO3 then analyzed by inductively coupled plasma (ICP) spectrophotometry (cold vapor AA was used for Hg analysis).

For 1991-1993 samples, the aliquot for organic analysis was extracted by sonification in acetonitrile followed with back extraction from water with petroleum ether. The pet ether extract was dried over sodium sulfate, evaporated to volume and cleaned up through a Florisil column with ether/hexane as elutants. Final volume was taken to 1 ml and the sample was transferred to an autosampler vial and capped. Analyses of PCBs and chlorinated pesticides was by GC-ECD.

For the 1994 samples, the aliquot for organic analysis was first freeze dried then ground in anhydrous sodium sulfate to dry further before Soxhlet extraction with methylene chloride. The extract was reduced to 5 mil from which an 1-ml aliquot was taken for organophosphorus analysis and the remaining 4 ml were reserved for PCB and chlorinated pesticide analyses. Cleanup of extracts for PCB and chlorinated pesticide analyses was through Florisil column chromatography using both hexane and methylene chloride-hexane mixture to elute; highly polar compounds were eluted using aeetonitrile. All fractions were analyzed using GC-ECD.

All concentrations are reported on a wet weight basis.

5.2.3 Sampling Processing Method Calibration

N/A

5.2.4 Sample Processing Quality Control

N/A

5.2.5 Sample Processing Method Reference

N/A

5.2.6 Sample Processing Method Deviations

None

6. DATA MANIPULATIONS

6.1 Name of New or Modified Values

TOT_ANAL

6.2 Data Manipulation Description

6.2.1 TOT_ANAL

Some of the codes in ANALYTE represent summed concentrations from other analytes. Examples of this include Total PAHs, Total DDTs, etc. In this case, the ANALYTE was not directly reported by the laboratory but is the result of summing the concentrations of analytes in a group. TOT_ANAL represents the number of concentrations that were summed for a given analyte.

6.3 Data Manipulation Examples

6.4 Data Manipulation Computer Code File

6.5 Data Manipulation Computer Code Language

6.6 Data Manipulation Computer Code

7. DATA DESCRIPTION

7.1 Description of Parameters

See Appendix A for list of analytes.

7.1.1 Parameter Name

Parameter Name	Data Type	Max Field Len	Format	Parameter Label
STA_NAME	Char	8	8.	The Station Identifier
VST_DATE	Num	8	YYMMDD6.	The Date the Sample was Collected
SAMPTYPE	Char	10	$10.	Organismal Derivation of Sample Material
SPECCODE	Char	9	$8.	EMAP Taxon Code
COMPOSIT	Char	3	$3.	Composite Code (Y/N)
ANALYTE	Char	8	$8.	Analyte Code
CONC	Num	8	13.6	Concentration of Analyte (wet wt.)
CHMUNITS	Char	12	$12.	Conc. Units (ug/g or ng/g)
QA_CODE	Char	15	$15.	Quality Assurance Code for Data
DETLIMIT	Num	8	13.6	Method Detection Limit for Analyte
TOT_ANAL	Num	8	3.	Analytes (#) Included in Summed Conc.
ANAL_CAT	Char	15	$15.	General Category for Group of Analytes
LABEL	Char	40	$40.	Label for Analyte

7.1.6 Precision to which values are reported

The tissue chemistry concentrations presented are in a format of 6 decimal places. This format is necessary because some concentrations are in ug/g and some concentrations are in ng/g. However, the concentrations are only valid FOR THREE

SIGNIFICANT FIGURES (not necessarily three decimal places), i.e., 345.67 ug/g is 346 ug/g but 0.00235 ng/g remains as 0.00235 ng/g.

7.1.7 Minimum Value in Data Set by Analyte

7.1.8 Maximum Value in Data Set by Analyte

7.2 Data Record Example

7.2.1 Column Names for Example Records

STA_NAME SAMPTYPE SPECCODE COMPOSIT CHMUNITS ANALYTE DETLIMIT QA_CODE TOT_ANAL VST_DATE CONC _LABEL_ ANAL_CAT

7.2.2 Example Data Records

OBS	STA_NAME	SAMPTYPE	SPECCODE	COMPOSIT	CHMUNITS	ANALYTE
1	LA92LR01	FISH	ARIUFELI	Y	ug/g	AG
2	LA92LR01	FISH	ARIUFELI	Y	ug/g	AL
3	LA92LR01	FISH	ARIUFELI	Y	ng/g	ALDRIN
4	LA92LR01	FISH	ARIUFELI	Y	ng/g	ALPHACHL
5	LA92LR01	FISH	ARIUFELI	Y	ug/g	AS

OBS	DETLIMIT	QA_CODE	TOT_ANAL	VST_DATE	CONC
1	.		.	920725	0.024012
2	2.000000	CH-A	.	920725	.
3	0.100000	CH-A	.	920725	.
4	0.100000	CH-A	.	920725	.
5	.		.	920725	1.320660

OBS	_LABEL_	ANAL_CAT
1	SILVER	METAL
2	ALUMINUM	METAL
3	ALDRIN	PESTICIDE
4	ALPHA-CHLORDANE	PESTICIDE
5	ARSENIC	METAL

7.3 Related Data Sets

7.3.1 Related Data Set Name

7.3.2 Related Data Set Identification Code

8. GEOGRAPHIC AND SPATIAL INFORMATION

8.1 Minimum Longitude

-97 Degrees 27 Minutes 13.20 Decimal Seconds

8.2 Maximum Longitude

-82 Degrees 39 Minutes 28.20 Decimal Seconds

8.3 Maximum Latitude

30 Degrees 48 Minutes 30.00 Decimal Seconds

8.4 Minimum Latitude

26 Degrees 02 Minutes 55.80 Decimal Seconds

8.5 Name of the area or region

Louisianian Province

8.6 Direct Spatial Reference Method

Point

8.7 Horizontal Coordinate System Used

Universal Transverse Mercator

8.8 Resolution of Horizontal Coordinates

0.5

8.9 Units for Horizontal Coordinates

Meters

8.10 Vertical Coordinate System

N/A

8.11 Resolution of Vertical Coordinates

N/A

8.12 Units for Vertical Coordinates

N/A

9. QUALITY CONTROL AND QUALITY ASSURANCE

Because of the complexity and importance of tissue contaminant data, EMAP has expended a tremendous effort in the Quality Assurance of these data as is reflected in the detail provided in this section.

9.1 Measurement Quality Objectives

Measurement Quality Objectives (MQOs) for the Louisianian Province analyses of chemical contaminants in tissue were defined in the Louisianian Province Quality Assurance Project Plans (Heitmuller and Valente, 1992). The QAPP required each laboratory to analyze the following quality control (QC) samples along with every batch or

"set" of field samples collected for analytical chemistry: laboratory reagent blank, calibration check standards, laboratory fortified sample matrix (matrix spike), laboratory duplicate (or matrix spike duplicate), and Laboratory Control Material (LCM). Results of these QC samples had to fall within certain preestablished control limits.

Because of EMAP-Estuaries' performance-based approach to QA/QC for analytical chemistry, Standard or Certified Reference Materials (SRMs or CRMs) were typically used as the LCM. SRMs and CRMs have known or "certified" concentrations for many of the analytes being measured and are representative of the matrices of interest. Therefore, SRMs/CRMs are useful for assessing both the accuracy and precision capabilities of the analytical laboratory. The QAPP required the laboratory's average percent recovery (relative to the certified or accepted concentration in the reference material) to fall within the range of 80 to 120% for each inorganic analyte and 65 to 135% for each organic analyte. The QC goal for precision was that the coefficient of variance (CV) of the percent recoveries for a given LCM analyte, across all batches, remain <= 30%. If the laboratory consistently failed to meet these accuracy or precision goals for the LCM, the values reported for the failed analytes were considered to be suspect and were flagged.

The laboratory established method detection limits (MDLs) for each analyte of interest; the reported MDL level was based on a calculated value that represented the laboratory's low end capability (minimal quantity for measuring the concentration of an analyte with statistical confidence). A true "non-detect" (i.e., no peak observed for the analyte) was reported by the laboratory as ND or 0.000 as was flagged with an "a" code. If the laboratory picked up a signal or peak for an analyte that translated to a concentration less than their declared MDL, but still was indicative of the presence of an analyte, the laboratory reported an estimated concentration for that analyte and flagged the data with a "b" code (see section 6.2 for detailed discussion).

9.2 Quality Assurance/Control Methods

If results for the QC samples did not fall within certain pre-established control limits, the analysis of a batch of samples was not considered acceptable. These and other quality control issues are coded with the following data qualifier codes (QA_CODE) or "flags" used in the Louisianian Province tissue chemistry data set:

CH-A CODE

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 DETLIMIT.

CH-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 "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 CODE

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 CODE

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-F CODE

The CH-F code indicates that the tissue samples were lost or destroyed at the laboratory or that they were unusable because of poor preservation techniques.

CH-I CODE

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-X CODE

In favor of expediency, a laboratory may elect to cease reporting some of the analytes. EMAP protocol only requires that the laboratory analyze a given list of chemicals; when they go beyond this list and report additional chemicals, we include them in our data. The "CH-X" code indicates that an analyte has been excluded from a given set of data.

CH-Z CODE

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-Z code.

Only "unflagged", CH-B or CH-C coded values are considered valid and useful for most assessment purposes.

9.3 Actual Measurement Quality

1991-1993 Analyses for Contaminants in Tissue

During 1991-1993 EMAP Monitoring in the Louisianian Province, the laboratory responsible for the analyses of organic and inorganic chemical contaminants in field-collected tissue samples, routinely met the required QC criteria and, overall, the data are were acceptable for EMAP assessments. Results for the 1991-1993 analyses of SRMs/LCMs (primary QC check for relative accuracy) are summarized in Tables 9.3.1(a-c) for organic analytes and Tables 9.3.2 (a-c) for inorganic analytes.

One exception for the 1991-93 contaminant analyses relates to the organochlorine (OC) pesticide, toxaphene; these data were judged to be unreliable, as will be explained in the following. Analytical results for toxaphene were reported at elevated concentrations for approximately 20 fish samples collected in 1991; no further occurrences were reported during 1992-93. The GC-ECD chromatogram for toxaphene results in a profusion of peaks routinely referred to as the "thumbprint" of toxaphene. It makes qualitative interpretation "iffy" and quantification, at best, a rough estimation. Confirmation by mass spectrophotometry is normally recommended for toxaphene "hits". However, the laboratory did not have mass spec capabilities readily available, therefore, the toxaphene hits for 1991 are suspect, especially since no further incidents were reported in 1992-93.

1994 Analyses for Contaminants in Tissue

In 1994, the cooperative agreement for analytical chemistry supporting Louisianian Province (LP) Monitoring was awarded to laboratory different from that responsible for the analyses of 1991-93 EMAP-LP tissue samples. Laboratory selection for the initial and subsequent cooperative agreement was based on competitive bids and were awarded for 3-year durations.

Inorganic Analyses - 1994

The laboratory routinely met or exceeded the required QC criteria related to the analyses of inorganic contaminants in tissue samples and all 1994 results for inorganic contaminants were acceptable for EMAP assessments without further qualification. See Table 9.3.2d for a summary of the laboratory's 1994 SRM results for inorganic analytes in the tissue.

Organic Analyses - 1994

The laboratory generally met the QC requirements for most of the analyses of organic contaminants in tissue See Table 9.3.1d for a summary of the laboratory's 1994 SRM results for organic analytes in tissue. However, some analytes, particularly within the organochlorine class, proved to be problematic; as a result, the analytical data for those analytes that consistently failed to meet the QC criteria were dropped from the database. The specific cases are discussed in the following sections.

Organochlorine Pesticides - 1994

For the 1994 tissue analysis, the analytical laboratory consistently failed to meet the quality criteria for both accuracy and precision for the following OC pesticides; they were unacceptable for assessments and dropped from the database:

dicofol	Not acceptable, dropped from database
oxyfluofen	"
endosulfan II	"
2,4-DDT	"
4,4-DDT	"
HCB	"
endrin	"

The remaining OC analytes were have been qualified with the "CH-C" code - deficient in one or more minor QC requirements, however, the overall data quality for these analytes was judged to be reliable for most EMAP regional assessments. Other users should exercise discretion in their particular use of these data.

PCBs - 1994

The PCBs, as a class, met the EMAP-E quality criteria for accuracy and precision (i.e., on the average the percent recovery for SRMs/LCMs was <65% for more than 70% of the analytes and the variability for replicate analyses was within acceptable limits for the majority of the congeners). However, the results of QC samples for the following, individual PCBs did not consistently meet all requirements and the data were qualified with a "CH-C" code and should be considered as estimates, only:

PCB-8	CH-C minor QC deficiencies, generally acceptable
PCB-18	"
PCB-44	"
PCB-105	"
PCB-128	"

The data for these congeners represents a low bias with recovery efficiencies generally <50% for both the SRM/LCM and matrix spike QC samples. One should note, however, because the bias for these data is to the low side, the potential for error in the reported sample concentrations favors conservative underestimates. Based on that assumption, EMAP utilized these data for selected assessments (e.g., reporting total PCBs). Other data users are urged to review their individual situations when utilizing the data for these PCBs.

All analytical results for PCB-77 were declared unreliable and have been dropped from the LP database.

Other than the exceptions discussed above, the PCB data were evaluated as acceptable for EMAP-E assessments without further qualification.

Butyltin Compounds - 1994

The analyses for butyltin compounds in tissue samples collected during the 1994 Monitoring routinely met the established quality standards for accuracy and precision and were deemed acceptable for EMAP assessments. Percent recoveries for LCM analyses (n=5) were 114% for monobutyltin, 91% for dibutyltin, and 92% for tributyltin.

Organophosphorus Compounds (OPs) - 1994

Because OPs degrade rapidly in the environment, their occurrence in tissue samples was not expected at concentrations > 5 ng/g (dry wt), the laboratory detection limits(MDL) for OPs in tissue. To verify that assumption, a subset of tissue samples (approximately 33%) was randomly selected and analyzed for OPs. Had significant hits been encountered, the entire set of 1994 tissue samples would then be analyzed; no concentrations > MDL were measured in the subset of 1994 tissue samples.

9.4 Sources of Error

9.5 Known Problems with the Data

9.6 Confidence Level/Accuracy Judgement

Appendix B (Tables)

9.7 Allowable Minimum Values

9.8 Allowable Maximum Values

9.9 QA Reference Data

10. DATA ACCESS

10.1 Data Access Procedures

A Data Request Package can be requested from a contact under Section 10.3. Data can be downloaded from the WWW site.

10.2 Data Access Restrictions

Data can only be accessed from the WWW site.

10.3 Data Access Contact Persons

Dr. J. Kevin Summers
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macauley.john@epa.gov (E-MAIL)

10.4 Data Set Format

Data can be transmitted in a variety of formats derived from SAS data files when a Data Request Form is submitted.

10.5 Information Concerning Anonymous FTP

Not accessible

10.6 Information Concerning Gopher

10.7 Information Concerning World Wide Web

Data can be downloaded from the WWW

10.8 EMAP CD-ROM Containing the Data set

Data not available on CD-ROM.

11. REFERENCES

11.1 EMAP References

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12. GLOSSARY AND TABLE OF ACRONYMS

12.1 Acronym used in the Detailed Documentation

12.2 Definition of Acronym

13. PERSONNEL INFORMATION

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---------- General Category for Analyte=METAL

ANALYTE	_LABEL_
AG	SILVER
AL	ALUMINUM
AS	ARSENIC
CD	CADMIUM
CR	CHROMIUM
CU	COPPER
FE	IRON
HG	MERCURY
NI	NICKEL
PB	LEAD
SE	SELENIUM
SN	TIN
ZN	ZINC

---------- General Category for Analyte=ORGANOPHOS

ANALYTE	_LABEL_
CARBOFEN	CARBOFENOTHION
DIAZINON	DIAZINON
DISULFOT	DISULFOTON
DURSBAN	DURSBAN
ETHION	ETHION
TERBUFOS	TERBUFOS

---------- General Category for Analyte=PCB

ANALYTE	_LABEL_
PCB101	PCB 101
PCB105	PCB 105
PCB118	PCB 118/108/149
PCB126	PCB 126
PCB128	PCB 128
PCB138	PCB 138
PCB153	PCB 153
PCB170	PCB 170
PCB18	PCB 18
PCB180	PCB 180
PCB187	PCB 187/182/159
PCB195	PCB 195
PCB200	PCB 200
PCB206	PCB 206
PCB209	PCB 209
PCB28	PCB 28
PCB29	PCB 29
PCB44	PCB 44
PCB52	PCB 52

PCB66	PCB 66
PCB77	PCB 77
PCB8	PCB 8
PCB87	PCB 87
PCB99	PCB 99
PCBTOT_L	TOTAL PCBS - LA PROVINCE
PCB_TOT	TOTAL PCBS

---------- General Category for Analyte=PESTICIDE

ANALYTE	_LABEL_
ALDRIN	ALDRIN
ALPHACHL	ALPHA-CHLORDANE
DDT	OP-DDT + PP-DDT
DDT_TOT	TOTAL DDT
DIELDRIN	DIELDRIN
ENDOSUL1	ALPHA-ENDOSULFAN
ENDOSULF	ENDOSULFAN
ENDRIN	ENDRIN
HEPTACHL	HEPTACHLOR
HEPTAEPO	HEPTACHLOR-EPOXIDE
HEXACHL	HEXACHLOROBENZENE
LINDANE	LINDANE (GAMMA-BHC)
MIREX	MIREX
OPDDD	O,P'DDD
OPDDE	O,P'DDE
OPDDT	O,P'DDT
PESTOT_L	TOTAL PESTICIDES - LA PROVINCE
PEST_TOT	TOTAL PESTICIDES
PPDDD	P,P'DDD
PPDDE	P,P'DDE
PPDDT	P,P'DDT
TNONCHL	TRANS-NONACHLOR
TOXAPHEN	TOXAPHENE

---------- General Category for Analyte=TBT

ANALYTE	_LABEL_
DBT	DI-BUTYL TIN
MBT	MONO BUTYL TIN
TBT	TRI-BUTYL TIN

TABLE 9-1a. Relative accuracy for the 1991 EMAP-LP analyses of organics in tissue based on the laboratory's analytical results of a laboratory control material (LCM), fish fillets spiked with known concentrations of the analytes of interest. Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's averaged value (n=27)for an analyte against the reported spiked concentration. The accuracy goal for EMAP-LP organic analyses was that the laboratory's values to be within +- 35% agreement to the LCM values. Accuracy criteria only apply for analytes having LCM concentrations >=10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
PCBs:		
PCB 8	89	(72 - 111)
PCB 18	92	(77 - 119)
PCB 28	92	(75 - 109)
PCB 44	95	(76 - 126)
PCB 52	96	(79 - 127)
PCB 66	96	(80 - 115)
PCB 77	94	(74 - 127)
PCB 99	97	(76 - 114)
PCB 101	98	(76 - 123)
PCB 105	95	(83 - 113)
PCB 118	93	(83 - 107)
PCB 126	95	(73 - 114)
PCB 128	96	(82 - 115)
PCB 138	94	(75 - 118)
PCB 153	97	(84 - 112)
PCB 170	97	(82 - 115)
PCB 180	96	(82 - 113)
PCB 187	95	(82 - 109)
PCB 195	92	(78 - 100)
PCB 206	91	(76 - 104)
PCB 209	92	(70 - 115)

TABLE 9-1a. (cont)

Organochlorine pesticides:		
aldrin	102	(86 - 118)
cis-chlordane	101	(90 - 113)
dieldrin	93	(73 - 114)
endosulfan	99	(74 - 127)
endrin	98	(74 - 127
gamma-HCH	105	(73 - 129)
heptachlor	105	(74 - 130)
heptachlor epoxide	101	(78 - 122)
hexachlorobenzene	90	(70 - 113)
mirex	104	(85 - 117)
trans-nonachlor	102	(92 - 118)
2,4'-DDD	99	(79 - 121)
2,4'-DDE	97	(85 - 115)
2,4'-DDT	108	(77 - 128)
4,4'-DDD	101	(76 - 116)
4,4'-DDE	103	(89 - 129)
4,4'-DDT	108	(79 - 125)

TABLE 9-1b. Relative accuracy for the 1992 EMAP-LP analyses of organics in tissue based on the laboratory's analytical results of a laboratory control material (LCM), fish fillets spiked with known concentrations of the analytes of interest. Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the reported spiked concentration. The accuracy goal for EMAP-LP organic analyses was that the laboratory's averaged value (n=35) to be within +- 35% agreement to the CRM values. Accuracy criteria only apply for analytes having LCM concentrations >=10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
PCBs:		
PCB 8	86	(63 - 129)
PCB 18	88	(70 - 117)
PCB 28	104	(73 - 134)
PCB 44	95	(75 - 119)
PCB 52	92	(75 - 118)
PCB 66	94	(70 - 110)
PCB 77	97	(68 - 128)
PCB 99	102	(71 - 117)
PCB 101	96	(60 - 130)
PCB 105	97	(63 - 127)
PCB 118	98	(68 - 131)
PCB 126	98	(72 - 118)
PCB 128	104	(73 - 134)
PCB 138	94	(69 - 131)
PCB 153	103	(83 - 127)
PCB 170	96	(76 - 125)
PCB 180	105	(73 - 134)
PCB 187	97	(75 - 117)
PCB 195	95	(67 - 122)
PCB 206	108	(90 - 131)
PCB 209	106	(79 - 130)

TABLE 9-1b. (cont)

Organochlorine pesticides:		
aldrin	96	(67 - 126)
cis-chlordane	108	(68 - 130)
dieldrin	87	(61 - 120)
endrin	92	(67 - 136)
gamma-HCH	85	(65 - 102)
heptachlor	106	(69 - 133)
heptachlor epoxide	94	(69 - 122)
hexachlorobenzene	93	(69 - 117)
mirex	109	(67 - 135)
trans-nonachlor	97	(62 - 125)

2,4'-DDD	94	(14 - 121)
2,4'-DDE	109	(76 - 137)
2,4'-DDT	101	(81 - 124)
4,4'-DDD	94	(75 - 111)
4,4'-DDE	112	(83 - 136)
4,4'-DDT	107	(76 - 121)

TABLE 9-1c. Relative accuracy for the 1993 EMAP-LP analyses of organics in tissue based on the laboratory's analytical results of a laboratory control material (LCM), fish fillets spiked with known concentrations of the analytes of interest. Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the reported spiked concentration. The accuracy goal for EMAP-LP organic analyses was that the laboratory's averaged value (n=21) to be within +- 35% agreement to the CRM values. Accuracy criteria only apply for analytes having LCM concentrations >=10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
PCBs:		
PCB 8	91	(84 - 97)
PCB 18	90	(78 - 97)
PCB 28	94	(90 - 100)
PCB 44	94	(92 - 102)
PCB 52	93	(88 - 99)
PCB 66	95	(93 - 102)
PCB 77	101	(94 - 123)
PCB 99	96	(91 - 109)
PCB 101	95	(87 - 108)
PCB 105	96	(84 - 104)
PCB 118	94	(90 - 99)
PCB 126	96	(93 - 106)
PCB 128	95	(90 - 104)
PCB 138	97	(92 - 100)
PCB 153	95	(92 - 102)
PCB 170	95	(89 - 101)
PCB 180	95	(90 - 104)
PCB 187	94	(85 - 98)
PCB 195	96	(90 - 101)
PCB 206	96	(90 - 101)
PCB 209	95	(92 - 99)

TABLE 9-1c. (cont)

Organochlorine pesticides:		
aldrin	99	(94 - 106)
cis-chlordane	100	(95 - 106)
dieldrin	99	(71 - 114)
endrin	105	(79 - 134)
gamma-HCH	105	(97 - 118)
heptachlor	111	(88 - 122)
heptachlor epoxide	103	(99 - 115)
hexachlorobenzene	104	(92 - 111)
mirex	100	(92 - 103)
trans-nonachlor	99	(95 - 110)
2,4'-DDD	100	(95 - 103)
2,4'-DDE	99	(91 - 109)
2,4'-DDT	105	(83 - 119)
4,4'-DDD	102	(85 - 118)
4,4'-DDE	98	(92 - 112)
4,4'-DDT	101	(95 - 110)

TABLE 9-1d. Relative accuracy for the 1994 EMAP-LP analyses of organics in tissue based on the laboratory's analytical results of a certified reference material (CRM), the National Research Council of Canada (NRC) CRM CARP-1 (a fish homogenate). "True" values were reported by NCR as either "certified" or "non-certified" values (certified values were only available for 9 PCB congeners). Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the reported "true" value. The accuracy goal for EMAP-LP organic analyses was that the laboratory's averaged value (n=xx) to be within +- 35% agreement to the CRM values. Accuracy criteria only apply for analytes having CRM concentrations >= 10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
PCBs:		
PCB 8	-a	-
PCB 18	45	(11 - 78)
PCB 28	128	(110 - 145)
PCB 44	38	(19 - 97)
PCB 52b	87	(79 - 95)
PCB 66	81	(70 - 91)
PCB 101b	84	(75 - 92)
PCB 105b	62	(47 - 77)
PCB 118b	128	(112 -144)
PCB 128	33	(21 - 45)
PCB 138b	77	(64 - 90)
PCB 153b	129	(122 - 136)

PCB 170b	116	(102 -130)
PCB 180b	100	(90 - 110)
PCB 187b	105	(94 - 116)
PCB 195	140	(122 - 158)
PCB 206	138	(122 - 153)
PCB 209	168	(146 - 189)

TABLE 9-1d. (cont)

Organochlorine pesticides:		
aldrin	-a	-
cis-chlordane	99	(93 - 100)
dieldrin	115	(110 - 120)
gamma-HCH	-a	-
heptachlor	-a	-
heptachlor epoxide	-a	-
hexachlorobenzene	106	(19 - 192)
mirex	-a	-
trans-nonachlor	82	(73 - 91)
2,4'-DDD	83	(81 - 84)
2,4'-DDE	59	(9 - 109)
2,4'-DDT	-a	-
4,4'-DDD	66	(40 - 92)
4,4'-DDE	95	(86 - 104)
4,4'-DDT	174	(147 - 200)

a Concentration reported as non-detect or <=10 x laboratory's MDL.

b Analyte with NRC certified concentration.

TABLE 9-2a. Relative accuracy for the 1991 EMAP-LP analyses of inorganic contaminants in tissue based on the laboratory's analytical results for a standard reference material (SRM), the National Institute of Standards and Technology (NIST) SRM 1566a (freeze-dried oyster tissue). Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the NIST certified value. The accuracy goal for EMAP-LP inorganic analyses was that the laboratory's averaged value (n=19) be within +-20% agreement to the NIST values. Accuracy criteria only apply for analytes having NIST certified values >= 10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
Aluminum	97	(81 - 112)
Arsenic	104	(79 - 119)
Cadmium	101	(88 - 112)
Chromium	104	(79 - 121)
Copper	101	(86 - 124)
Iron	97	(85 - 117)
Mercury	104	(95 - 117)
Nickel	98	(87 - 126)
Lead	98	(75 - 107)
Selenium	98	(82 - 118)
Silver	96	(74 - 116)
Tin	97	(77 - 115)
Zinc	103	(90 - 121)

TABLE 9-2b. Relative accuracy for the 1992 EMAP-LP analyses of inorganic contaminants in tissue based on the laboratory's analytical results for a standard reference material (SRM), the National Institute of Standards and Technology (NIST) SRM 1566a (freeze-dried oyster tissue). Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the NIST certified value. The accuracy goal for EMAP-LP inorganic analyses was that the laboratory's averaged value (n=35) be within +-20% agreement to the NIST values. Accuracy criteria only apply for analytes having NIST certified values >= 10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
Aluminum	98	(90 - 108)
Arsenic	100	(85 - 115)
Cadmium	100	(77 - 117)
Chromium	97	(79 - 121)
Copper	99	(87 - 110)
Iron	94	(87 - 107)
Mercury	101	(83 - 117)
Nickel	103	(86 - 116)
Lead	99	(85 - 115)
Selenium	102	(84 - 117)
Silver	94	(84 - 111)
Tin	94	(79 - 114)
Zinc	96	(88 - 105)

TABLE 9-2c. Relative accuracy for the 1993 EMAP-LP analyses of inorganic contaminants in tissue based on the laboratory's analytical results for a standard reference material (SRM), the National Institute of Standards and Technology (NIST) SRM 1566a (freeze-dried oyster tissue). Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the NIST certified value. The accuracy goal for EMAP-LP inorganic analyses was that the laboratory's averaged value (n=35) be within +-20% agreement to the NIST values. Accuracy criteria only apply for analytes having NIST certified values >=10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
Aluminum	101	(89 - 128)
Arsenic	94	(85 - 107)
Cadmium	104	(88 - 119)
Chromium	107	(79 - 121)
Copper	110	(93 - 119)
Iron	93	(2.6 - 103)
Mercury	98	(83 - 117)
Nickel	102	(82 - 117)
Lead	101	(75 - 125)
Selenium	89	(77 - 114)
Silver	100	(86 - 116)
Tin	95	(75 - 119)
Zinc	110	(93 - 118)

TABLE 9-2d. Relative accuracy for the 1994 EMAP-LP analyses of inorganic contaminants in tissue based on the laboratory's analytical results for a certified reference material (CRM), the National Research Council of Canada NRC) CRM DORM2 (freeze-dried dogfish liver). Relative accuracy, expressed as percent recovery, was computed by comparing the laboratory's value for an analyte against the NRC certified value. The accuracy goal for EMAP-LP inorganic analyses was that the laboratory's averaged value (n=35) be within +-20% agreement to the CRM values. Accuracy criteria only apply for analytes having CRM certified values >= 10 times the laboratory's MDL.

ANALYTE	RECOVERY (%)	RANGE (% recov)
Aluminum	107	(72 - 146)
Arsenic	105	(96 - 116)
Cadmium	97	(69 - 116)
Chromium	98	(85 - 172)
Copper	97	(85 - 106)
Iron	-	-
Mercury	103	(98 - 107)
Nickel	100	(79 - 201)
Lead	150	(66 - 327)
Selenium	100	(51 - 138)
Silver	97	(76 - 117)
Tin	116	(81 - 210)
Zinc	99	(90 - 106)