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Analysis
(Type of Report)

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ESC-UAG-86-112

JOB/PROJECT NO.: 6253

DATE: October 7, 1986

TITLE: ANALYSIS OF AROCLOR 1248 IN DELAWARE RIVER PLANT WATER SAMPLES

AUTHORS: B.M. Hughes, D. Mckenzie, and B. Simpson

ABSTRACT: A one-liter water sample and four 33-mL water samples from Monsanto's Delaware River Plant were analyzed for Aroclor 1248. Aroclor concentrations ranged between 8 and 3,300 ng/g with levels of detection of 5 and 50 ng/g. A column cleanup method was also developed which aided in measuring the concentration of this formulation in water samples which contained relatively high levels of total organic carbon.

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APPROVAL DATE: 10-8-86

MONS 024410

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TITLE: ANALYSIS OF AROCLOR 1248 IN DELAWARE RIVER PLANT WATER SAMPLES
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ANALYSIS REPORT

DATE: October 3, 1986

LOG NUMBER: U-86-09-23-02 PROJECT NO: 760.28-6253

TO: Ed Jamro/Delaware River Plant (1290)

FROM:

Environmental Sciences Center
Monsanto Company
800 N. Lindbergh Blvd.
St. Louis, Mo. 63167

Phone No: (314) 694-1464

Title: Analysis of Aroclor 1248 in Delaware River Plant Water Samples.

ABSTRACT

A one-liter water sample and four 33-mL water samples from Monsanto's Delaware River Plant were analyzed for Aroclor 1248. Aroclor concentrations ranged between 8 and 3,300 ng/g with levels of detection of 5 and 50 ng/g. A column cleanup method was also developed which aided in measuring the concentration of this formulation in water samples which contained relatively high levels of total organic carbon.

INTRODUCTION

Water samples from the Delaware River Plant were analyzed for PCBs in preparation for full scale tests of a newly installed water purification system. These water samples contained sediment and organic compounds which interfered with normal Aroclor analyses. A rapid extraction and column cleanup method was developed which allowed for quantitation of Aroclor 1248 in these contaminated water samples. A brief description of the extraction method PCEX25 and flow sheet PCFS25 are shown in a later section of this report. The capillary GC/EC analysis method ECAN02 and flow sheet ECFS02 are also shown in a later section.

SUMMARY

Table 1 summarizes the results of these analyses. The first four sample results are for four 40-mL aliquots of the one-liter water sample. Representative aliquots were obtained by shaking the water sample for several seconds and quickly pouring sediment and water into 40-mL vials. The overall precision of these analyses can be estimated from these four results. The remaining four sample results are for four vials which were also submitted for analysis. Notice that the results of the first four aliquots show concentrations of Aroclor 1248 in the same concentration range as Vial #1 - Vial #3 shown in Table 1. In addition, Vial #4 contains Aroclor 1248 at a concentration of between two and three orders of magnitude lower than the first three vials. However, there are still measurable quantities of Aroclor 1248 in Vial #4.

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(continued)

Figures 1 and 2 show capillary GC/EC chromatograms obtained from the analysis of extract P1829 and an Aroclor 1248 standard. Figure 1 shows CL6-benzene (internal standard), and the major Aroclor 1248 peaks. All other peaks are interferences from electron capturing compounds which are also present in these water samples. Figure 2 shows the extract P1829 and Aroclor 1248 on the same display so that the Aroclor 1248 components can easily be seen.

QC RESULTS

All samples were spiked with CL5-benzene and 2,4,6-CL3-biphenyl surrogate compounds. Good recoveries were measured for CL5-benzene in the four water samples from the one-liter bottle. The higher recoveries of 2,4,6-CL3-biphenyl measured for these samples were probably due to interferences. Recoveries could not be determined for these compounds in the four vials due to these interferences. The method blank surrogate recoveries were lower than normal, but these low recoveries do not impact the overall quality or the conclusions of this study. Table 2 summarizes recoveries of Aroclor 1260 which was added to two of the 40-mL aliquots from the one-liter sample.

The levels of detection reported in Table 1 for Aroclor 1248 are estimates based upon PCB congener areas of the major congeners in Aroclor 1248. The level of detection of 5 ng/g reported for Vial #4 should be adequate to obtain concentration data of Aroclor 1248 in the waste treatment plant effluents, if the Vial #4 sample is representative of the samples which will be obtained from the treatment plant. However, it is suggested that some other set of surrogate standards be chosen so that recovery information for these compounds can be obtained for each sample analyzed.

DETAILS

Standard Sampling Method No(s): Samples supplied by Ed Jamro.

Standard Extraction Method No(s): PCEX25 (See flow sheet PCFS25.)

Standard Analysis Method No(s): ECAN02 (See flow sheet ECFS02.)

Sample Identification(s): Monsanto DRP Vial #1, Monsanto DRP Vial #2, Monsanto DRP Vial #3, Monsanto DRP Vial #4, Monsanto DRP Well IW3B

Final Report File No(s): A25318

Table File No(s): B25318, C25318, D25318

(continued)

Also included in this report are:

(1) Brief descriptions of Standard Extraction and Analysis Methods used.

(2) Extraction details for samples associated with this project.

(3) Raw data documentation, included a preliminary report sent on 10/2/86.

(4) Analytical request information recorded at sample login.

(5) Brief descriptions of terminology used in Quality Assurance/Quality Control, Analyte Extraction, and Instrumental Analysis.

ANALYST(S): B. Mason Hughes, David McKenzie, and Brenda Simpson

ANALYST APPROVAL: B. Mason Hughes

Table 1. Concentrations of Aroclor 1248, and % Recovery of surrogate spikes detected in Delaware River water samples using Standard Extration Method PCEX25 and Standard Analysis Method ECAN02.

ESC Project No: 760.28 - 6253 Date: September 29,1986

Sample I.D.	EXTRACT #	LOD(a) (ng/g)	TOTAL AMOUNT(b) (ng/vial)	CONC.(c) (ng/g)	SURROGATE RECOVERY (%) CL5-benzene	SURROGATE RECOVERY (%) 2,4,6-CL3-biphenyl
WELL IW3B	PCB-1826	50	88,900	2,223	90	135
WELL IW3B-DUP	PCB-1827	50	112,000	2,804	109	164
WELL IW3B-LS	PCB-1825	50	107,000	2,685	93	167
WELL IW3B-HS	PCB-1828	50	117,000	2,966	93	154
Average Value		50	106,000	2,660		
. . .		. . .	TOTAL AMOUNT(b) (ng/vial)	CONC.(c) (ng/g)	SURROGATE RECOVERY (%) CL5-benzene	SURROGATE RECOVERY (%) 2,4,6-CL3-biphenyl
VIAL #1	PCB-1829	50	109,000	3,300	I(e)	I
VIAL #2	PCB-1830	50	33,000	1,000	" "	" "
VIAL #3	PCB-1831	50	81,000	2,460	" "	" "
VIAL #4	PCB-1832	5	270	8.2	" "	" "
METHOD BLANK	PCB-1833	5	ND(d)	ND	43	65

Footnotes:

a. LOD = Limit of detection (in units of ng/g).
b. TOTAL AMOUNT is the total amount of Aroclor 1248 in each vial in units of ng/vial.
c. CONC. is the concentration of Aroclor 1248 in each sample in units of ng/g (assuming a 33 mL sample volume for Vial #1 - Vial #4).
d. ND = No Aroclor formulation detected above the LOD.
e. I = Could not be quantified due to matrix interferences.

TABLE FILE NO: D25318:MH

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Table 2. Summary of Aroclor 1260 Spiking Concentrations and Recoveries in Delaware River Water Samples.

ESC Project No: 760.28 - 6253 Date: September 29,1986

Sample I.D.	Total Amount Added (ng/vial)	Aroclor 1260 Amount Added (ng/g)	Aroclor 1260 Recovery (%)
WELL IW3B + LOW SPIKE	20,000	500	158
WELL IW3B + HIGH SPIKE	100,000	2,500	87.5

TABLE FILE NO: C25318:MH

MONS 024416

Figure 1. Identification of peaks.

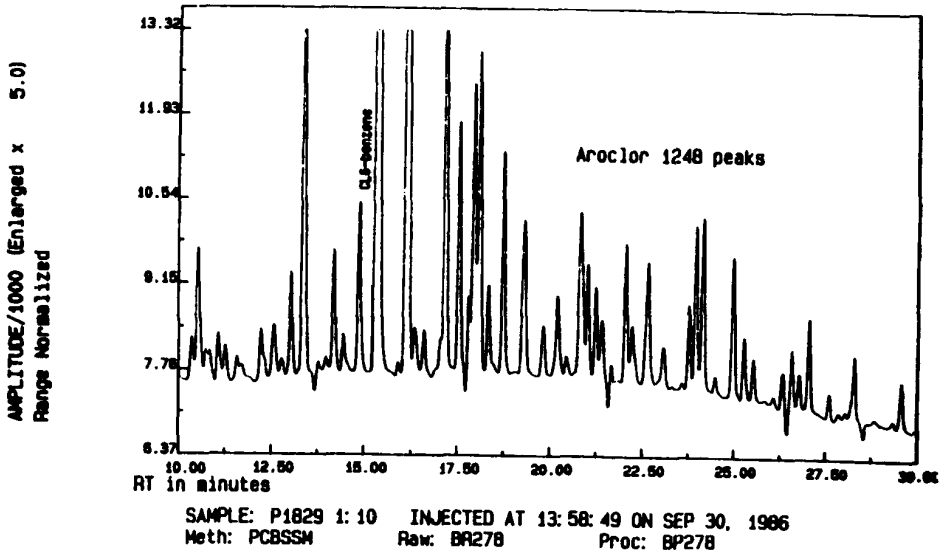
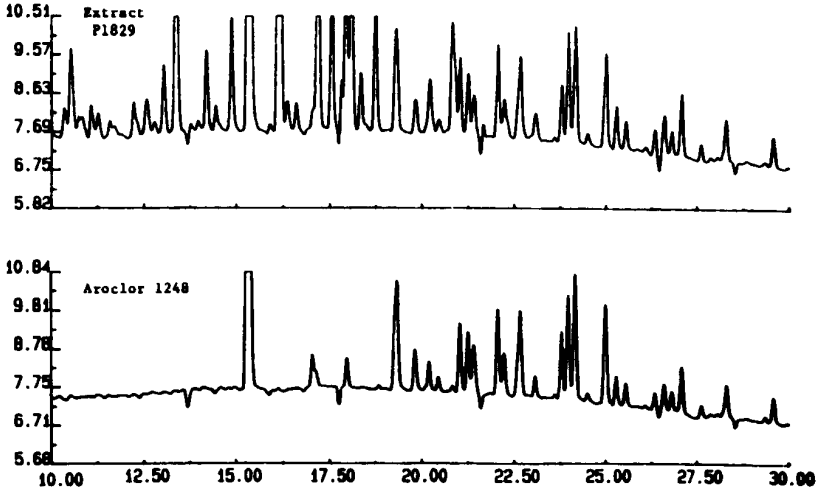


Figure 2. Comparison of Aroclor 1248 with Extract P1829.



Top Raw: BR278 (10.0- 30.0) (Enlarged x 20.0)
Bottom Raw: BR274 (10.0- 30.0) (Enlarged x 10.0)

MONS 024418

DETAILED EXPLANATION OF TABLES

In an effort to streamline reports generated by ESC on PCBs present in a wide variety of materials, using a wide variety of techniques, Standard Extraction Methods (SEMs) and Standard Analysis Methods (SAMs) have been developed which can be easily interfaced with each other. An abbreviated form of these SEMs and SAMs have been stored on the Hewlett-Packard 3357-LAS laboratory automation system used by ESC. In a effort to communicate these details with customers of ESC, this information is supplied with each PCB analysis. However, many projects involve a very small number of samples which are not necessarily the same matrix. Therefore, major information on the technical details of an analysis are presented in tabular form within the same table which reports the data.

Each table includes a general title, ESC Project Number and Date on which the table was generated. The sample identification is the customer identification which was on the sample bottle. The next section of the table gives results for the particular analyte listed. For Aroclor formulations, the formulation is identified either visually or from ESC developed data analysis programs. The concentration of the formulation is calculated as the average value of the Aroclor formulation, based upon the analysis of a number of individual PCB isomers in the formulation.

This table format is both compact and precise. It was developed in an attempt to quickly and accurately generate data which the customer can quickly and accurately evaluate. If there are any suggestions concerning this format, please contact me at METS 544-1466.

DESCRIPTION STORED IN TBEX04:MH

The following pages describe Standard Sampling, Extraction, and/or Analysis Methods used for this project.

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Water Matrices that Require Cleanup

ESC Standard Extraction Method No: PCEX25

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Water matrices which contain electron capturing compounds that interfere with the quantitation of PCBs.	10 - 1000 mL	1 - 10

TYPICAL ANALYTES: A1232, A1242, A1248, A1254, A1260 and A1262 and PCB isomers mono- through deca-chlorobiphenyl.

INTERFERENCES: Any electron capturing species in the retention time region of monochlorobiphenyl isomers through decachlorobiphenyl isomers for ECAN01 and ECAN02. Any species in the appropriate retention time region which give the same relative charac-teristic mass responses as PCB isomers for MSAN03.	TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
	pentachlorobenzene	50 - 120
	2,4,6-trichloro-biphenyl	50 - 120
	3,3',4,4'-tetra-chlorobiphenyl-d6	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson Solvents: hexane, methylene chloride, and others.
Sulfuric acid for ionizing and hydrolizing esters, ethers, bases, and alcohols.
Potasium hydroxide for removal of organic acids, phenols, and other acidic compounds.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Disposable glassware.
(See flow sheet for this extraction method in PCFS25.)

LIMITATIONS: Method applies only to matrices which contain hydrolizable or ionizable electon capturing species in the monochlorobiphenyl through decachlorobiphenyl retention time region using EC detection methods.
PLEASE NOTE!! MS SAMS are valid only for PCB isomers from CL3-BP through CL10-BP due to reactivity of CL1-BP and CL2-BP to 80% sulfuric acid cleanup.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND UPDATES: 10/1/86

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

SAMPLE ID: ..... MRC EXTRACT #.....

..... LOG NO: .....

EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH .....

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION.....
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER) [ ]

SURROGATE SPIKE OF SAMPLE [ ]

EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS [ ]

ADD ---- mL ---- SULFURIC ACID AND SHAKE [ ]

ADD ---- mL ---- KOH AND SHAKE [ ]

REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g) [ ]

ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL [ ]
APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)

ELUTE WITH 40 mL HEXANE [ ]

REDUCE EXTRACT TO FINAL VOLUME. [ ]

RECORD FINAL VOLUME ---------- mL

SAMPLE VOLUME .....

SURROGATE SPIKE
.....
.....
.....

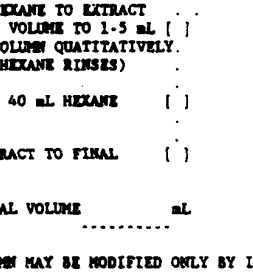
RECORD AMOUNT USED FOR EACH EXTRACTION
mL mL mL
1)------ 2)------ 3)------

RECORD # OF TIMES EXTRACT IS ACID WASHED .....

RECORD # OF TIMES EXTRACT IS BASE WASHED .....

[ ] PRERINSE COLUMN WITH 2 X 15 mL HEXANE

SUGGESTED COLUMN DESCRIPTION
25 mL DISPO PIPET



NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY:
.....

OTHER OBSERVATIONS:

EXTRACT NO: ..... DATE/TIME COMPLETED----------/----------

ANALYST: ..... ESC JOB NO: .....

MONS 024422

ESC Standard Analysis Method (SAM) ESC Standard Analysis Method No:
TITLE: Aroclor Formulations in Solvents ECAN02

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5880 Capillary GC/EC interfaced to an HP-3357-LAS Laboratory Data System

---

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Aroclor formulations 1232, 1242, 1248, 1254, 1260, and 1262	0.1	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl through octachlorobiphenyl isomers are potential interferences. Exact retention times of most likely PCB isomers can be obtained from the analysis of Aroclor 1248, Aroclor 1254 or Aroclor 1260 formulations.

CHROMATOGRAPHIC COLUMN: 30-meter fused silica J&W DB-5 (0.25 uM) with wide bore (0.32mm).

COLUMN TEMPERATURE PROGRAM: 130(1)/4/300 INJECTION TYPE: Splitless

---

CALIBRATION AND STANDARDIZATION: Use internal standard quantitation technique and calibrate instrument response factors from authentic Aroclor formulation standards. Use a minimum of 10 PCB isomers for the determination of formulation identity and quantity.

CALCULATIONS: Formulation concentration =
(isomer RRF)(isomer area/internal standard area)

where: RRF is the relative response factor for the appropriate PCB isomer in a given formulation. Formulation concentration is obtained from the average of at least 10 such calculations.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATE OF METHOD CREATION: 10/25/83, 2/3/84 Time Stamp - <861002.1536>

FLOW SHEET ECFS02

FOR ESC ANALYSIS METHOD ECAN02

EXTRACT ID: ---------- ESC EXTRACT # ----------

---------- LOG NO: ----------

EC LOGBOOK PAGE NUMBER: ----------

EXTRACT COLOR AND DESCRIPTION ----------

PLACE 150 uL EXTRACT
OR AROCLOR STANDARD,
AND 150 uL INTERNAL [ ]
STANDARD SOLUTION CONTAINING .
0.04 ug/mL CL6-BENZENE IN .
BENZENE IN A 1.6 mL .
AUTOSAMPLER VIAL. .
.

PLACE VIAL IN AUTOSAMPLER [ ] CALCULATE RRFs FOR 10 - 30 PCB CONGENERS
TRAY OF HP-5880. . IN THE APPROPRIATE AROCLOR FORMULATION.
. USING THE AROCLOR CONCENTRATION AS THE

RECORD ELECTRON CAPTURE [ ] . . . . . [ ] ANALYTE CONCENTRATION
AND GAS CHROMATOGRAPHIC . FOR EACH PCB CONGENER.
OPERATING PARAMETERS AND .
3357-LAS FILE INFORMATION .
IN INSTRUMENT LOGBOOK. .

[ ] CALCULATE CONCENTRATION
OF AROCLOR FORMULATION
BY AVERAGING THE VALUES
DETERMINED FROM THE 10 -
30 PCB CONGENERS.

Where:

$$\text{RRF - Relative Response Factor} = \frac{\text{(congener area in Aroclor standard)}}{\text{(CL6-benzene area)(Aroclor concentration)}}$$

$$\text{Aroclor Concentration} = \frac{\text{(congener area)}}{\text{(CL6-benzene area) (RRF)}}$$

THESE EQUATIONS ASSUME THAT THE SAME AMOUNT OF INTERNAL STANDARD IS ADDED TO EXTRACTS AND STANDARDS.

The above calculations are performed for 10 - 30 PCB congeners and the values averaged to yield an average Aroclor formulation concentration.

MS FILE NUMBER: ---------- DATE/TIME COMPLETED ----------/-------

ANALYST: ---------- ESC JOB NO: ----------

MONS 024424

The following pages show extraction details for samples associated with this project.

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

WELL IW3B PCB-1826

SAMPLE ID: ---------- MRC EXTRACT #----------
U-86-09-23-02
---------- LOG NO: ----------
9-25-86
EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER) [X]
SAMPLE VOLUME 14 mL.

SURROGATE SPIKE

SURROGATE SPIKE OF SAMPLE [X] 200 ul DEM050885-01 (10 & 20ug/ml)

EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS [X]
RECORD AMOUNT USED FOR EACH EXTRACTION
8 mL 8 mL 8 mL
1)------ 2)------ 3)------

ADD ---- mL ---- SULFURIC ACID AND SHAKE [ ]
RECORD # OF TIMES EXTRACT IS ACID WASHED -----

ADD 10 mL 25 KOH AND SHAKE [X]
RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----

REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g) [X]
[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE

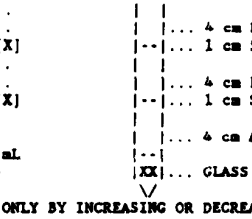
ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL [X]
APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)

ELUTE WITH 40 mL HEXANE [X]

REDUCE EXTRACT TO FINAL VOLUME. [X]

RECORD FINAL VOLUME 10 mL ----------

SUGGESTED COLUMN DESCRIPTION
25 mL DISPO PIPET



NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS:

PCB-1826 10-3-86
EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------
SIMPSON 760.28-6253
ANALYST: ---------- ESC JOB NO: ----------

MONS 024426

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

WELL IW3B-DUP

PCB-1827

SAMPLE ID: ---------- MRC EXTRACT #----------
U-86-09-23-02
---------- LOG NO: ----------
9-25-86
EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER) [X]

SAMPLE VOLUME ---------- 14 ml.

SURROGATE SPIKE OF SAMPLE [X]

SURROGATE SPIKE
200 ul DEM050885-01 (10 & 20ug/ml)

EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS [X]

RECORD AMOUNT USED FOR EACH EXTRACTION
8 mL 8 mL 8 mL
1)------ 2)------ 3)------

ADD ---- mL ---- SULFURIC ACID AND SHAKE [ ]

RECORD # OF TIMES EXTRACT IS ACID WASHED -----

ADD 10 mL 25 KOH AND SHAKE [X]

RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----

REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g) [X]

[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE

ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL [X]
APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)

ELUTE WITH 40 mL HEXANE [X]

REDUCE EXTRACT TO FINAL VOLUME. [X]

RECORD FINAL VOLUME 10 mL ----------

SUGGESTED COLUMN DESCRIPTION
25 mL DISPO PIPET

| | |... 1 cm NA2SO4
|--|... 1 cm SILICA GEL
| |
| |... 4 cm KMnO4 SILICA GEL
|--|... 1 cm SILICA GEL
| |
| |... 4 cm BASE SILICA GEL
|--|... 1 cm SILICA GEL
| |
| |... 4 cm ACID SILICA GEL
|--|
|XX|... GLASS WOOL PLUG

NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS:

PCB-1827
EXTRACT NO: ----------

10-3-86
DATE/TIME COMPLETED----------/----------

SIMPSON
ANALYST: ----------

760.28-6253
ESC JOB NO: ----------

MONS 024427

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

WELL IW3B-LS PCB-1825

SAMPLE ID: ---------- MRC EXTRACT #: U-86-09-23-02

---------- LOG NO: ----------

EXTRACTION DATE and TIME: 9-25-86 ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER)	[X]	SAMPLE VOLUME 14 ml. ----------
		SURROGATE SPIKE ----------
SURROGATE SPIKE OF SAMPLE	[X]	200 ul DEM050885-01 (10 & 20ug/ml) ----------
EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS	[X]	RECORD AMOUNT USED FOR EACH EXTRACTION 8 mL 8 mL 8 mL 1)------ 2)------ 3)------
ADD ---- mL ---- SULFURIC ACID AND SHAKE	[ ]	RECORD # OF TIMES EXTRACT IS ACID WASHED -----
ADD 10 mL 25 KOH AND SHAKE	[X]	RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----
REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g)	[X]	[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE
ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)	[X]	SUGGESTED COLUMN DESCRIPTION 25 mL DISPO PIPET | |--| ... 1 cm NA2SO4 |--| ... 1 cm SILICA GEL | | ... 4 cm KMnO4 SILICA GEL
ELUTE WITH 40 mL HEXANE	[X]	|--| ... 1 cm SILICA GEL | | ... 4 cm BASE SILICA GEL
REDUCE EXTRACT TO FINAL VOLUME.	[X]	|--| ... 1 cm SILICA GEL | | ... 4 cm ACID SILICA GEL
RECORD FINAL VOLUME 10 mL ----------		|--| |XX| ... GLASS WOOL PLUG V

NOTE: COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS: LS-200 ul DEM092386-01(108ug/ml DILUTED 1:10)

PCB-1825 10-3-86
EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------
SIMPSON 760.28-6253
ANALYST: ---------- ESC JOB NO: ----------

MONS 024428

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

WELL IW3B-HS

PCB-1828

SAMPLE ID: ---------- MRC EXTRACT #----------
U-86-09-23-02
---------- LOG NO: ----------
9-25-86
EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER) [X]

SURROGATE SPIKE OF SAMPLE [X]

EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS [X]

ADD ---- mL ---- SULFURIC ACID AND SHAKE [ ]

ADD 10 mL 25 KOH AND SHAKE [X]

REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g) [X]

ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL [X]
APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)

ELUTE WITH 40 mL HEXANE [X]

REDUCE EXTRACT TO FINAL VOLUME. [X]

RECORD FINAL VOLUME 10 mL

14 ml.
SAMPLE VOLUME ----------

SURROGATE SPIKE
---
200 ul DEM050885-01 (10 & 20ug/ml)

RECORD AMOUNT USED FOR EACH EXTRACTION
8 mL 8 mL 8 mL
1)------ 2)------ 3)------

RECORD # OF TIMES EXTRACT IS ACID WASHED -----

RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----

[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE

SUGGESTED COLUMN DESCRIPTION
25 mL DISPO PIPET

| |... 1 cm NA2SO4
|--|... 1 cm SILICA GEL
| |
| |... 4 cm KMnO4 SILICA GEL
|--|... 1 cm SILICA GEL
| |
| |... 4 cm BASE SILICA GEL
|--|... 1 cm SILICA GEL
| |
| |... 4 cm ACID SILICA GEL
|--|
|XX|... GLASS WOOL PLUG
V

NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS: HS-100 ul DEM092386-01(108ug/ml)

PCB-1828 10-3-86
EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------
SIMPSON 760.28-6253
ANALYST: ---------- ESC JOB NO: ----------

MONS 024429

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

VIAL #1 PCB-1829

SAMPLE ID: ---------- MRC EXTRACT #----------
U-86-09-23-02
---------- LOG NO: ----------
9-29-86
EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER)	[X]	1 VIAL SAMPLE VOLUME ----------
		SURROGATE SPIKE
SURROGATE SPIKE OF SAMPLE	[X]	100 ul DEM050885-01 (10 & 20ug/ml)
EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS	[X]	RECORD AMOUNT USED FOR EACH EXTRACTION 8 mL 8 mL 8 mL 1)------ 2)------ 3)------
ADD ---- mL ---- SULFURIC ACID AND SHAKE	[ ]	RECORD # OF TIMES EXTRACT IS ACID WASHED -----
ADD 10 mL 25 KOH AND SHAKE	[X]	RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----
REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g)	[X]	[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE
ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)	[X]	SUGGESTED COLUMN DESCRIPTION 25 mL DISPO PIPET
ELUTE WITH 40 mL HEXANE	[X]	
REDUCE EXTRACT TO FINAL VOLUME.	[X]	
RECORD FINAL VOLUME 10 mL		

	... 1 cm NA2SO4
	... 1 cm SILICA GEL
	... 4 cm KMnO4 SILICA GEL
	... 1 cm SILICA GEL
	... 4 cm BASE SILICA GEL
	... 1 cm SILICA GEL
	... 4 cm ACID SILICA GEL
XX	... GLASS WOOL PLUG

NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS:

PCB-1829 10-3-86
EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------
SIMPSON 760.28-6253
ANALYST: ---------- ESC JOB NO: ----------

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

VIAL #2

SAMPLE ID: ---------- MRC EXTRACT #: PCB-1830 U-86-09-23-02

---------- LOG NO: ----------

EXTRACTION DATE and TIME: 9-29-86 ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER)	[X]	1 VIAL SAMPLE VOLUME ----------
		SURROGATE SPIKE
SURROGATE SPIKE OF SAMPLE	[X]	100 ul DEM050885-01 (10 & 20ug/ml)
EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS	[X]	RECORD AMOUNT USED FOR EACH EXTRACTION 8 mL 8 mL 8 mL 1)------ 2)------ 3)------
ADD ---- mL ---- SULFURIC ACID AND SHAKE	[ ]	RECORD # OF TIMES EXTRACT IS ACID WASHED -----
ADD 10 mL 25 KOH AND SHAKE	[X]	RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----
REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g)	[X]	[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE
ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)	[X]	SUGGESTED COLUMN DESCRIPTION 25 mL DISPO PIPET | |... 1 cm NA2SO4 |--|... 1 cm SILICA GEL | | | |... 4 cm KMnO4 SILICA GEL
ELUTE WITH 40 mL HEXANE	[X]	|--|... 1 cm SILICA GEL | |
REDUCE EXTRACT TO FINAL VOLUME.	[X]	| |... 4 cm BASE SILICA GEL |--|... 1 cm SILICA GEL | |
RECORD FINAL VOLUME 10 mL ----------		| |... 4 cm ACID SILICA GEL |--| |XX|... GLASS WOOL PLUG

NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMnO4

OTHER OBSERVATIONS:

EXTRACT NO: PCB-1830 ---------- DATE/TIME COMPLETED 10-3-86 ----------/----------

ANALYST: SIMPSON ---------- ESC JOB NO: 760.28-6253 ----------

MONS 024431

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

VIAL #3 PCB-1831

SAMPLE ID: ---------- MRC EXTRACT #----------
U-86-09-23-02
---------- LOG NO: ----------
9-29-86
EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER) [X]

1 VIAL
SAMPLE VOLUME ----------

SURROGATE SPIKE

SURROGATE SPIKE OF SAMPLE [X] 100 ul DEM050885-01 (10 & 20ug/ml)

EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS [X]

RECORD AMOUNT USED FOR EACH EXTRACTION
8 mL 8 mL 8 mL
1)------ 2)------ 3)------

ADD ---- mL ---- SULFURIC ACID AND SHAKE [ ]

RECORD # OF TIMES EXTRACT IS ACID WASHED -----

ADD 10 mL 25 KOH AND SHAKE [X]

RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----

REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g) [X]

[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE

ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL [X]

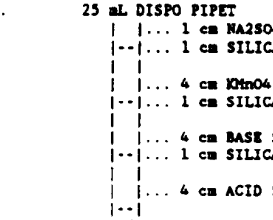
APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)

ELUTE WITH 40 mL HEXANE [X]

REDUCE EXTRACT TO FINAL VOLUME. [X]

RECORD FINAL VOLUME 10 mL

SUGGESTED COLUMN DESCRIPTION
25 mL DISPO PIPET



NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS:

PCB-1831 10-3-86
EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------
SIMPSON 760.28-6253
ANALYST: ---------- ESC JOB NO: ----------

MONS 024432

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

VIAL #4

SAMPLE ID: ---------- MRC EXTRACT #: PCB-1832 U-86-09-23-02

LOG NO: ----------

EXTRACTION DATE and TIME: 9-29-86 /---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER) [X]

SURROGATE SPIKE OF SAMPLE [X]

EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS [X]

ADD ---- mL ---- SULFURIC ACID AND SHAKE [ ]

ADD 10 mL 25 KOH AND SHAKE [X]

REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g) [X]

ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES) [X]

ELUTE WITH 40 mL HEXANE [X]

REDUCE EXTRACT TO FINAL VOLUME. [X]

RECORD FINAL VOLUME 1 mL

1 VIAL
SAMPLE VOLUME ----------

SURROGATE SPIKE
100 ul DEM050885-01 (10 & 20ug/ml)

RECORD AMOUNT USED FOR EACH EXTRACTION
1)------ 8 mL 2)------ 8 mL 3)------ 8 mL

RECORD # OF TIMES EXTRACT IS ACID WASHED -----

RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----

[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE

SUGGESTED COLUMN DESCRIPTION
25 mL DISPO PIPET

| | |... 1 cm NA2SO4
|--|... 1 cm SILICA GEL
| |
| |... 4 cm KMnO4 SILICA GEL
|--|... 1 cm SILICA GEL
| |
| |... 4 cm BASE SILICA GEL
|--|... 1 cm SILICA GEL
| |
| |... 4 cm ACID SILICA GEL
|--|
|XX|... GLASS WOOL PLUG

NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMnO4

OTHER OBSERVATIONS:

EXTRACT NO: PCB-1832 DATE/TIME COMPLETED 10-3-86 /----------

ANALYST: SIMPSON ESC JOB NO: 760.28-6253

MONS 024433

FLOW SHEET PCFS25
FOR ESC EXTRACTION METHOD PCEX25

METHOD BLANK PCB-1833

SAMPLE ID: ---------- MRC EXTRACT #----------
U-86-09-23-02
---------- LOG NO: ----------
9-29-86
EXTRACTION DATE and TIME: ----------/---------- RECORD NATIVE pH ---------

INDICATE IS pH ADJUSTED YES/NO TO WHAT pH-----LIST CONCENTRATION----------
SUGGESTED ADJUSTMENT MEDIA (50% KOH OR 50% H2SO4)

PLACE SAMPLE INTO SEPERATORY FUNNEL (OR SUITABLE EXTRACTION CONTAINER)	[X]	1 VIAL SAMPLE VOLUME ----------
SURROGATE SPIKE OF SAMPLE	[X]	SURROGATE SPIKE ---------- 100 ul DEM050885-01 (10 & 20ug/ml) ----------
EXTRACT SAMPLE WITH METHYLENE CHLORIDE 3 TIMES AND POOL EXTRACTS	[X]	RECORD AMOUNT USED FOR EACH EXTRACTION 8 mL 8 mL 8 mL 1)------ 2)------ 3)------
ADD ---- mL ---- % SULFURIC ACID AND SHAKE	[ ]	RECORD # OF TIMES EXTRACT IS ACID WASHED -----
ADD 10 mL 25 % KOH AND SHAKE	[X]	RECORD # OF TIMES EXTRACT IS BASE WASHED 3 -----
REMOVE WASHES AND DRY EXTRACT WITH NA2SO4 (1g)	[X]	[X] PRERINSE COLUMN WITH 2 X 15 mL HEXANE
ADD 5 mL HEXANE TO EXTRACT AND REDUCE VOLUME TO 1-5 mL APPLY TO COLUMN QUATITATIVELY. (3 X 3 mL HEXANE RINSES)	[X]	SUGGESTED COLUMN DESCRIPTION 25 mL DISPO PIPET | |... 1 cm NA2SO4 |--|... 1 cm SILICA GEL | | | |... 4 cm KMnO4 SILICA GEL
ELUTE WITH 40 mL HEXANE	[X]	|--|... 1 cm SILICA GEL | | | |... 4 cm BASE SILICA GEL
REDUCE EXTRACT TO FINAL VOLUME.	[X]	|--|... 1 cm SILICA GEL | | | |... 4 cm ACID SILICA GEL
RECORD FINAL VOLUME 1 mL		|--| |XX|... GLASS WOOL PLUG V

NOTE! COLUMN MAY BE MODIFIED ONLY BY INCREASING OR DECREASING PHASES LISTED.
LIST COLUMN MODIFICATIONS IF ANY: 8 cm ACID, 2 cm BASE, 2 cm KMNO4

OTHER OBSERVATIONS:

PCB-1833 10-3-86
EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------
SIMPSON 760.28-6253
ANALYST: ---------- ESC JOB NO: ----------

The following pages show calculations or raw data documentation of reported results.

MONSANTO COMPANY/
Environmental Sciences Center

From: BRENDA SIMPSON
Monsanto Company/
Environmental Sciences Center
800 N. Lindbergh Blvd.
St. Louis, Mo. 63167

cc: DAVID MCKENZIE, MASON HUGHES

Date: October 2,1986 Time Stamp - <861002.1006>

Subject: Preliminary Data Report on Delaware River Project.

Reference: none

To: Ed Jamero
Monsanto Deleware River Plant
RT 130
Bridgeport, NJ. 08014

Enclosed are data reported via phone for the analysis of water samples received on 9/23/86. This is just a preliminary report of the samples based on six peaks found in both the samples and Aroclor 1248, using an Internal Standard quantitation method. Due to matrix interferences, baseline interpretation, and possible detector signal saturation the final results may vary from preliminary data. The results of the four vial samples are reported in ng/vial. A final report is to follow.

BRENDA SIMPSON
(314) 694-1497

PRELIMINARY DATA, NOT A FINAL REPORT

Table 1. Concentrations of Aroclor 1248, and % Recovery of surrogate spikes detected in Delaware River water samples using Standard Extration Method PCEX25 and Standard Analysis Method ECANO2.

ESC Project No: 760.28 - 6253 Date: September 29,1986

Sample I.D.	EXTRACT #	LOD(a) (ng/g)	CONC.(b) FACTOR	CONC.(c) (ng/g)	% SURROGATE RECOVERY CL5 benzene	% SURROGATE RECOVERY 2,4,6-biphenyl
WELL IW3B	PCB-1826	500	4X	2223	90	135
WELL IW3B-DUP	PCB-1827	500	4X	2804	109	164
WELL IW3B-LS	PCB-1825	500	4X	2685	93	167
WELL IW3B-HS	PCB-1828	500	4X	2966	93	154

Preliminary Sample Data based on Six Peaks shown in Figures 1-6 With Internal Standard Quantitation.

				CONC. (ng/vial)	CL5 benzene	2,4,6-biphenyl
VIAL #1	PCB-1829	500	4X	68,000	I(e)	I
VIAL #2	PCB-1830	500	4X	21,000	" "	" "
VIAL #3	PCB-1831	500	4X	43,000	" "	" "
VIAL #4	PCB-1832	50	40X	240	" "	" "
METHOD BLANK	PCB-1833	50	40X	ND(d)	---	---

Footnotes:

a. LOD = Limit of detection (based on extract concentration).
b. Extract concentration = sample volume/ extract volume (based on an assumed volume of 40ml).
c. ng/g = parts per billion.
d. ND = No Aroclor formulation detected.
e. I = Could not be quantified due to matrix interferences.

TABLE FILE NO: 825318:MM

MONS 024437

Table 2. Summary of Aroclor 1260 Spiking Concentrations and Recoveries in Delaware River Water Samples.

ESC Project No: 760.28 - 6253 Date: September 29,1986

Sample I.D.	Aroclor 1260	
	Amount Added (ug/g)	Recovery (%)
WELL IW3B + LOW SPIKE	20.00	ND
WELL IW3B + HIGH SPIKE	100.00	87.5

TABLE FILE NO: C25318:MH

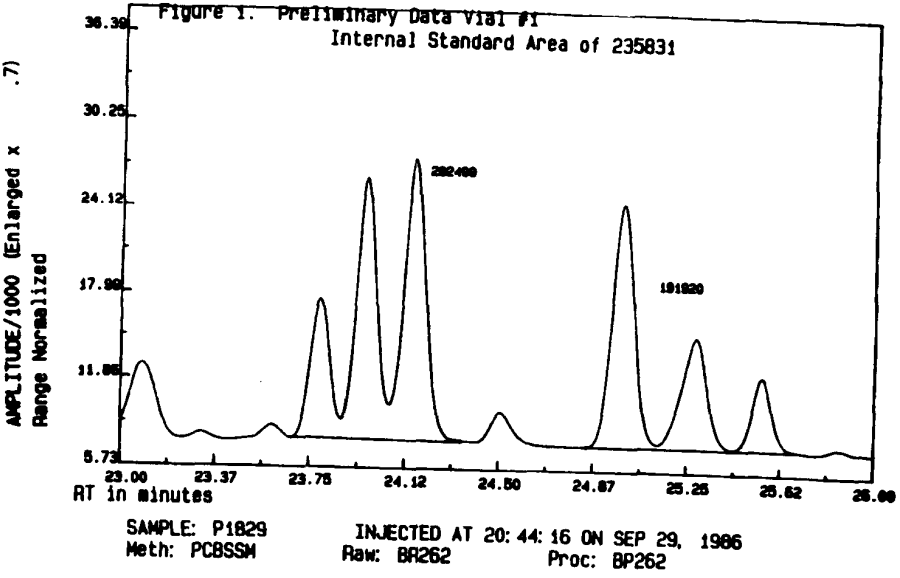


Figure 1. Preliminary Data Vial #1
Internal Standard Area of 235831
AMPLITUDE/1000 (Enlarged x .7)
Range Normalized
36.39
30.25
24.12
17.98
11.86
5.73
282400
191820
23.00
23.37
23.75
24.12
24.50
24.87
25.25
25.62
26.00
RT in minutes
SAMPLE: P1829
Meth: PCBSSM
INJECTED AT 20:44:16 ON SEP 29, 1986
Raw: BR262
Proc: BP262

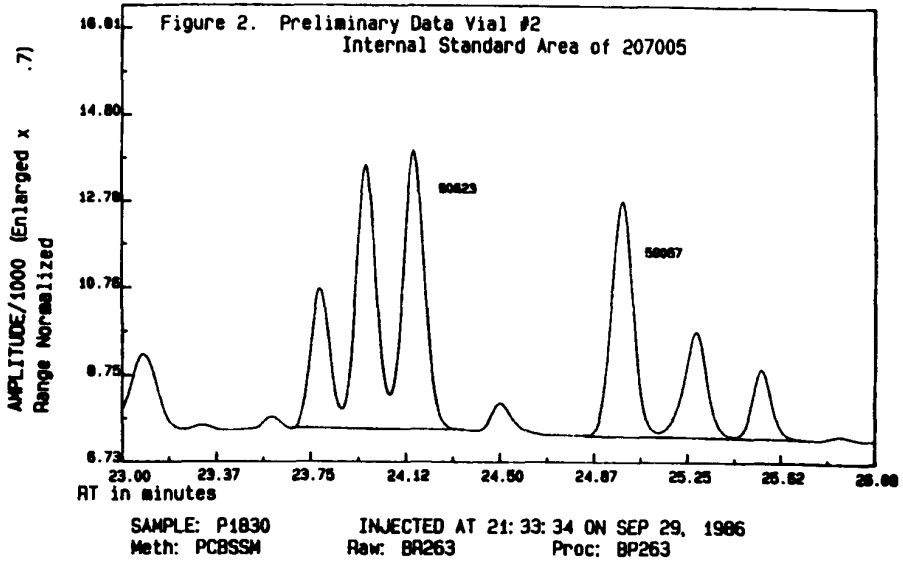
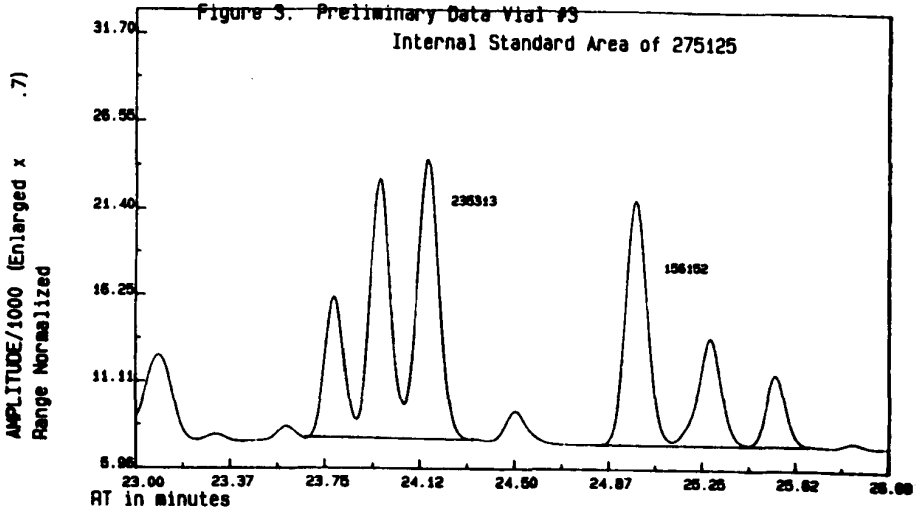


Figure 2. Preliminary Data Vial #2
Internal Standard Area of 207005
AMPLITUDE/1000 (Enlarged x .7)
Range Normalized
16.81
14.80
12.78
10.76
8.75
6.73
90623
58087
23.00
23.37
23.75
24.12
24.50
24.87
25.25
25.62
26.00
RT in minutes
SAMPLE: P1830
Meth: PCBSSM
INJECTED AT 21:33:34 ON SEP 29, 1986
Raw: BR263
Proc: BP263

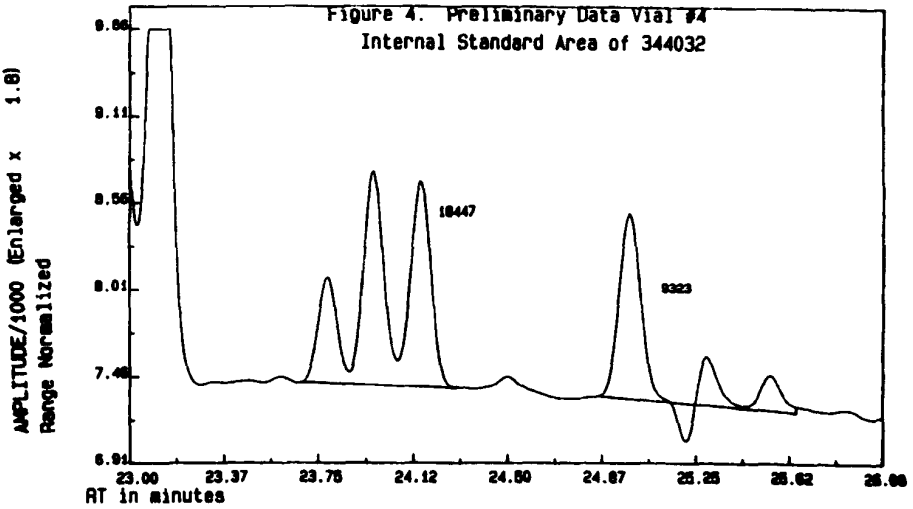
Figure 3. Preliminary Data Vial #3



SAMPLE: P1831 INJECTED AT 22:22:49 ON SEP 29, 1986
Meth: PCBSSM Raw: BR264 Proc: BP264

MONS 024441

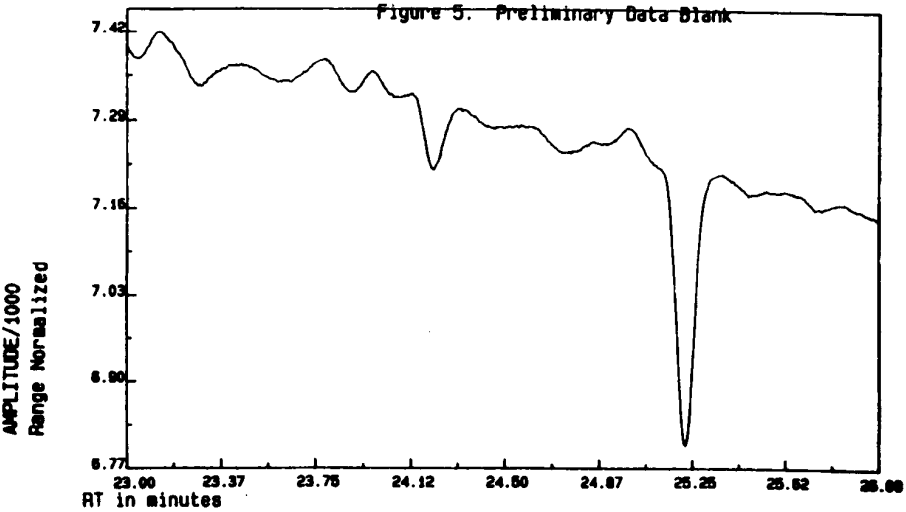
Figure 4. Preliminary Data Vial #4
Internal Standard Area of 344032



SAMPLE: P1832 INJECTED AT 0:01:28 ON SEP 30, 1986
Meth: PCBSSM Raw: BR266 Proc: BP266

MONS 024442

Figure 5. Preliminary Data Blank



SAMPLE: P1833 INJECTED AT 0:50:58 ON SEP 30, 1986
Meth: PCBSSM Raw: BR267 Proc: BP267

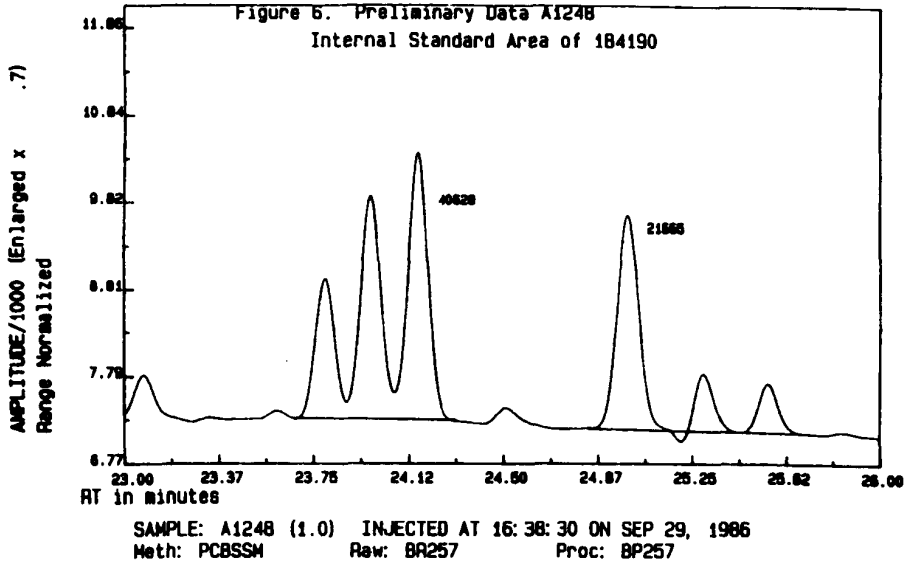


Figure 6. Preliminary Data A1248
Internal Standard Area of 184190
AMPLITUDE/1000 (Enlarged x .7)
Range Normalized
11.06
10.84
9.82
8.81
7.79
6.77
40628
21666
23.00
23.37
23.75
24.12
24.50
24.87
25.25
25.62
26.00
RT in minutes
SAMPLE: A1248 (1.0) INJECTED AT 16:38:30 ON SEP 29, 1986
Meth: PCBSSM Raw: BR257 Proc: BP257

MONS 024444

The following pages describe Analytical Request Information generated for this project.

Time Stamp - <861003.1532>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: September 23,1986 LOG NO: U-86-09-23-02

REQUESTER: Jamero/Delaware River Plant PROJECT NO: 760.28-6253

LOGIN NOTEBOOK PAGE: 40

REPORT RESULTS TO: FILES/MEES, SHERMAN, HUGHES, MCKENZIE, SIMPSON

ANALYSIS REQUESTS SENT TO: FILES, SHERMAN, HUGHES, MCKENZIE

SAMPLE PICKUP BY: SIMPSON

SAMPLE LOCATION: U420

SAMPLE DESCRIPTION FOR 5 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
MONSANTO DRP VIAL #1	1 X 2 oz.	PCB-1829
MONSANTO DRP VIAL #2	1 X 2 oz.	PCB-1830
MONSANTO DRP VIAL #3	1 X 2 oz.	PCB-1831
MONSANTO DRP VIAL #4	1 X 2 oz.	PCB-1832
MONSANTO DRP WELL IW3B	1 X 1 L.	PCB-1826--PCB-1828

RECORDED BY: SIMPSON

REQUIRED ANALYSES: PCB's

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158617

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
X GC/EC (18)	10/2/86	ECAN02	157278A

FINAL REPORT NOTEBOOK PAGE: 3,434,434

OTHER NOTEBOOK PAGES:

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: HOLD FOR 60 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA253:LI

The following sections describe important terminology used in Quality Assurance/Quality Control, Analyte Extraction and Instrumental Analysis. ESC and ESC/UAG Good Laboratory Practices Manuals can be obtained by contacting Bill Maes (U4E) METS 544-1499.

Quality Assurance/Quality Control Terminology

This section describes commonly used terminology and phrases which are used in conjunction with Quality Assurance/Quality Control (QA/QC). This information may be helpful in understanding some details of QA/QC procedures which have been incorporated in the present report. The following two sections also give useful information concerning terminology used in analyte extraction and instrumental analysis of samples and sample extracts.

Quality Assurance/Quality Control - Terminology used to describe activities which are designed to produce analytical results of known quality. In an effort to minimize costs of environmental analyses, specific QA/QC elements have been incorporated into ESC/UAG extraction and analysis steps which should not unduely burden analytical studies with large development and method validation costs. This approach has been taken so that sets of samples can be analyzed for various analytes without extensively validating methods over wide concentration ranges and without using methods which are designed for only one matrix.

One price which one pays for this approach is that analytical precision and accuracy are sacrificed, somewhat. However, in many cases, when clearly defined regulatory limits have been set, confirming that the analyte is present at levels well below or well above the limit does not need the type of accuracy that is required when the regulated analyte concentration is in the vicinity of the regulatory limit. In these latter cases, more extensive QA/QC proceedures are required than are included in the present study.

Precision - Terminology used to evaluate the reproducibility of a certain determination. Parameters which affect analysis precision range from sample acquisition, to sample storage, to sample extraction, and finally to sample extract analysis. Normally, the major factor which affects the precision of the analysis is the sample extraction step. This step normally results in sample analysis precision on the order of + or - 30%.

The precision of a result can be determined in a number of ways. Replicate analyses of the same sample, followed by the calculation of a percent relative standard deviation for that determination is the most popular method of assessing precision. However, this calculation should not be made when there are less than three replicate points from which to calculate the percent relative standard deviation. In addition, replicate extractions and instrumental analyses must be performed. Therefore obtaining percent relative standard deviation data can be very expensive and is not normally obtained unless large studies are being conducted. A second method of assessing precision is with the use of duplicate analyses. The present report includes results of at least one sample which was analyzed in duplicate. NOTE!! It is the responsibility of the analysis requestor to review these data and draw his own conclusions concerning the precision of the determination, and, more importantly, if the precision of the determination meets his needs.

(continued)

Accuracy - Terminology used to determine the "correctness" of the data. The accuracy of most steps in the analysis of samples is typically on the order of 10%. However, when analyte concentrations in the sample extracts approach the level of detection of a determination, accuracy as well as precision can approach 100% or greater. The accuracy of a determination of an analyte which is present at concentrations which are much greater than the instrumental level of detection is most strongly affected by the homogeniety of the sample matrix and the precision of the extraction step, which results in typical accuracies in environmental analyses to be on the order of 30%. Normally, the accuracy of standard solutions which are used to calibrate analytical instrumentation is well below this amount, and therefore is normally not an important factor in producing inaccurate data. From time to time, analyte responses of standard solutions from different sources will be compared to standard solutions used for instrument calibration. This information is normally included in the section entitled "QA/QC Results".

The accuracy of a result can be estimated in a number of ways. The current study uses surrogate standard recovery and native analyte recovery information to aid in assessing the accuracy of a result. This recovery information, normally reported as percent recovery, gives you information concerning the percent of surrogate standards and native analytes which were recovered after adding to your sample matrix and performing the various extractions, clean-ups, and instrumental analyses associated with the determination. The use of surrogates also assumes that the spiked surrogates are bound in the matrix in the same way as the analytes of interest. Surrogate standards are added to every sample while native analytes are added to only 10% of each set of samples. Therefore surrogate standard recovery data could be used to correct the reported results of an analyte concentration, if the user of the data feel such corrections are warranted. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Bias - Terminology used to describe how the accuracy of a determination may be affected. A low bias means that values below the correct value are normally measured. A high bias means that values above the correct value are normally measured. The instrumental analysis step of extract analysis normally does not contribute significantly to analysis bias. Most bias in environmental analysis is introduced in the sample extraction step. A low bias is introduced when the sample is not completely extracted and the extract does not contain 100% of the analyte. A high bias is introduced when the final volume of the extract is less than the correct volume. Surrogate Standards (SS), which are discussed in the next section, can be used to determine the amount of bias which is incorporated in the reported result. NOTE!! No data reported in this report includes any correction for this bias. It is the responsibility of the person requesting the analyses to apply appropriate bias corrections, if he/she feels that such corrections are warranted.

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Level of Detection (LOD) - Terminology used to indicate what analyte concentration will produce a signal-to-noise ratio of 10. For the present report, this value is not experimentally determined. Normally, a Limit of Quantitation is used to determine the threshold concentration above which an analyte can be detected.

Limit of Quantitation (LOQ) - Terminology used to indicate the lowest analyte concentration which can reliably be quantified. The precision of the determination of the analyte concentration at the LOQ is normally on the order of 100%. The LOQ is normally determined experimentally by adding a known amount of analyte to the sample matrix and measuring the percent recovery at the LOQ. In some cases, when there is insufficient sample quantity, or for convenience, an LOQ will be determined using other methods. These methods should be clearly described in this report.

Further Information

The general area of Quality Assurance/Quality Control, along with much of the terminology described above, is very complex. The following references may provide further information which may be useful in understanding the validity of the results reported in this study.

ASTM Standard Practice D4210-83 entitled "Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data".

ASTM Standard Practice D2777-77 entitled "Determination of Precision and Bias of Methods of Committee D-19 on Water".

An article in Analytical Chemistry, authored by 29 well-known environmental scientists. This article was funded by a grant from the Environmental Protection Agency and acknowledges 24 additional scientists in industry and government who were involved in the review of the article. Its citation is: "Principles of Environmental Analysis", L. H. Keith, et. al., Anal. Chem. 1983, 55, 2210 - 2218.

40 CFR Part 136 entitled "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule". This document includes details of EPA's 600-series analysis methods. A current copy of these methods can be found in the October 26, 1984 Federal Register.

QAQCTE:MH

Analyte Extraction Terminology

This section describes commonly used terminology and phrases which are used in conjunction with analyte extraction. This information may be helpful in understanding some details of how analytes in your samples were isolated for instrumental analysis. Instrumental Analysis Terminologies are given in the following section of this report.

Sample - Terminology used to reference the material which you submit for analysis. Samples may be solid, liquid, and in some cases, gases. In general, when a sample is received for analysis, it is logged into the ESC sample login book and proper chain-of-custody documents are maintained in the event this information may be required for litigation purposes.

Analyte - Terminology used to reference specific compounds or formulations of compounds. When an analysis is requested for a certain analyte within a certain sample matrix, normally some type of extraction process is required.

Extraction - This terminology refers to the step which isolates the analyte of interest into an appropriate solvent. Extraction is required for several reasons. In many cases, the concentration of the analyte is below the level of detection of the instrument which will be used for analysis. Therefore, extraction involves removing the analyte of interest from a relatively large volume of sample and placing it into a relatively small volume of solvent. This places the analyte in the solvent at a concentration which can easily be detected by the analytical instrument.

A second reason that extraction is required, is that the sample may not be appropriate for direct instrumental analysis of the analyte of interest. This can be easily understood for analytes in soils. Very few analytical instruments allow soils to be directly inserted into the instrument. Therefore some type of isolation step (i. e. extraction) is required in order to place the analyte in an appropriate matrix for instrumental analysis.

When the analyte of interest is present at sufficient concentration, and the sample matrix can be directly inserted into the analytical instrument, the sample, or the diluted sample, can be directly analyzed. However, this case represents a minority of sample analyses which ESC is asked to perform.

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Matrix - Terminology used to reference the major components of a sample. In most cases, major components are not of direct interest except as they affect the types of extraction steps and other isolation procedures which must be used to isolate the analyte from the sample matrix. For the majority of environmental samples which ESC is asked to analyze, the matrix is normally water or soil. However, many environmental matrices contain relatively high levels of organic compounds which may interfer with the instrumental analysis of the analyte of interest. Therefore, depending upon the analyte of interest, the required level of detection, and the amounts and types of other organic materials which may interfer with the instrumental analysis, extraction methods must be developed which are very specific for a given set of samples. In order to deal with almost a limitless set of matrices, analytes, extraction methods and instrumental methods, an organized approach to sample extraction, followed by instrumental analysis, has been developed at ESC.

Extract - Terminology used to identify the analyte-containing solvent which was obtained from the extraction of a certain matrix.

Extract Clean-up - Terminology used to identify chemical steps which are required in order to reduce the concentration of organic compounds which may be extracted with the analyte of interest. These co-extracted compounds may be at such high levels and/or produce responses so that they interfer with the instrumental analysis of the analyte in the extract.

Extractable Compounds - Terminology which refers to the ability of a compound to be isolated from the sample matrix and placed into the extract solvent. When the sample matrix is primarily soil which contains little organic matter, most organic compounds are extractable compounds. However, when the sample matrix is primarily water, only those compounds which are hydrophobic are extractable compounds.

Standard Extraction Method - Terminology used to reference an organized description of the steps involved in isolating an analyte from a given matrix. These methods are assigned short names which can also be used as computer file names for these methods. The names of specific Standard Extraction Methods which are used in this study are listed in the final report in the section entitled "Details". In addition, copies of these Standard Extraction Methods are shown in a separate section of this report. Standard Extraction Methods also include details of sample clean-up, if required.

Extraction Flow Sheet - Terminology used to reference a graphic description of a Standard Extraction Method. This flow sheet is filled out by the analyst in the laboratory and serves as a detailed record of what occurred in the sample extraction step. Flow sheets of individual sample extractions may be included in a section of this report.

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Surrogate Standards (SS) - One important element in the extraction of an analyte from a matrix, is the determination of what percent of the analyte was actually extracted from the matrix. For methods which involve liquid/liquid extraction of water with an organic solvent, or liquid/solid extraction of soils with an organic solvent, it is imperative that data be obtained which gives the analysis requestor information concerning what percentage of the analyte was isolated in the extract during the extraction step. Surrogate Standards serve this important purpose.

The philosophy on the use of Surrogate Standards is as diverse as the analysts who use them. If cost were no object, analyte recovery information could be obtained by the use of standards addition analysis methods. However, since many samples require extensive labor to isolate an analyte into an extract, standards addition analysis methods are too costly for most studies. (A standards addition analysis requires each sample to be extracted and analyzed at least three times.) Surrogate Standards give the same type of information, with only one analysis being required.

A "Surrogate" Standard is a compound which is chosen to behave exactly as the analyte. It is a surrogate for that analyte and thus information obtained for this surrogate can be assumed to also apply to the analyte of interest. For the analysis of regulated analytes, it is important that when data is generated that shows that the analyte is not present, that we can also show that if the regulated analyte were present in the sample matrix, it would have been detected. This is especially true when complex extraction and instrumental analysis steps are involved in this determination. Therefore, when an extraction is performed, a known amount of a Surrogate Standard is added to the sample before any extraction steps take place. When the extract is then analyzed for the analyte of interest, the amount of the Surrogate Standard is also determined. From this information one can document, for each sample, what recoveries were measured for this Surrogate Compound. From this, one can describe some level of confidence on the data generated for the analyte of interest.

A detailed discussion on how Surrogate Standards are chosen is beyond the scope of this section on Analyte Extraction Terminology. ESC normally uses two types of Surrogate Standards. The first is when the surrogate is exactly the same as the analyte of interest, except that the naturally occurring carbon, hydrogen, or halogen atoms in the analyte have been replaced with non-naturally occurring isotopes. These surrogates can be expected to act exactly as the native analytes. One good example of this type of surrogate is the 2,3,7,8-TCDD surrogate in which all carbon atoms are carbon-13 or all chlorine atoms are chlorine-37.

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The second type of Surrogate Standard is one which does not have the same chemical structure as the analyte of interest, but is known, or assumed to act chemically identical to the analyte of interest during the extraction and clean-up steps. Examples of this type of surrogates are deuterated compounds which may be added to a matrix in which any chromatographable materials are to be identified. This approach can only be applied to GC/MS analyses, since the surrogates can be differentiated from the native compounds from their different mass spectra. Since it is not known before sample extraction and extract analysis what analytes may be present, surrogates must be chosen that could not possibly be present in the sample. In addition, since only liquid/liquid or liquid/solid extraction would be involved, with no clean-up (since clean-up would remove compounds in which the requester is interested) any hydrophobic deuterated compound could serve as a surrogate. Normally, phenol-d5, 2-CL-phenol-d4, CL2-benzene-d4, pyrene-d10 and chrysene-d12 are used as Surrogate Standards for the analysis of a wide variety of hydrophobic materials in environmental samples.

A sub-set of this second type of Surrogate Standards are the surrogates which are used in the capillary GC/EC analysis of Aroclor formulations. Since mass spectrometric detection is not used, surrogates which contain unique isotopes cannot be distinguished from their native counterparts. Therefore, other compounds are used as Surrogate Standards since they have been observed to act exactly as the polychlorinated biphenyls (PCBs) in the Aroclor formulations. Any halogenated compound can be used which acts exactly as PCBs in the extraction and clean-up step. ESC normally uses CL5-benzene and 2,4,6-CL3-biphenyl for this purpose.

Native Analytes - Terminology which references the analytes of interest which contain naturally-occurring isotopes of carbon, hydrogen, halogens, etc.

Native Spike - Terminology which references the addition of a known amount of native analytes to the sample matrix. Normally, at least one sample will be spiked with the native analyte at the reported level of detection (low native spike) and at 10 times the level of detection (high native spike). Recoveries of the native analytes provide further documentation on the quality of the analyses performed. For sample sets which contain more than 10 samples, the frequency of low and high native spikes is 10% of the number of samples.

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Method Blank - Terminology which refers to a sample which is known to contain none of the analytes of interest. For water analyses, this is normally Burdick and Jackson Brand Water, de-ionized, or distilled water. For soil, it may be soil which has been previously analyzed and known to contain none of the analytes of interest. All extraction steps, using randomly selected glassware, solvents, and all surrogate spiking solutions must be used. This provides documentation that the analytes of interest were not inadvertantly present in extraction glassware and solvents used in the analysis. A minimum of one method blank must be generated with each set of samples. For sample sets containing more than 10 samples, method blanks are generated at a frequency of 10% of the number of samples.

Duplicate Analyses - Terminology used to indicate the duplicate extraction and duplicate extract analysis of a sample. This provides documention on the precision of an analysis. Normally the precision of environmental analyses of homogeneous samples is on the order of 30%. A minimum of one duplicate analysis is required for each sample set. For sample sets containing more than 10 samples, duplicate analyses are generated at a frequency of 10% of the number of samples.

Further Information

The general area of Analyte Extraction, along with much of the terminology described above, is very complex. The following references may provide further information which may be useful in understanding extraction fundamentals and terminology.

40 CFR Part 136 entitled "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule". This document includes details of EPA's 600-series analysis methods. A current copy of these methods can be found in the October 26, 1984 Federal Register.

Statement of Work for the Contract Laboratory Program which is used in conjunction with EPA's Superfund Site studies. A current copy may be obtained by contacting:

Joan F. Fisk, Project Officer
Analytical Support Branch
Hazardous Response Support Division
USEPA
Washington, D. C. 20460

ANEXTE:MH

Instrumental Analysis Terminology

This section describes commonly used terminology and phrases which are used in conjunction with extract analysis. This information may be helpful in understanding some details of how instrumental analyses of sample extracts were performed. The previous section of this report describes terminology associated with analyte extraction.

Sample Extract - Terminology used to describe the solvent containing the analyte of interest which is obtained from some type of sample extraction step. Detailed information on how this extract is prepared can be found in the Standard Extraction Methods and flow sheets associated with the methods. Summaries of these methods which were used for this project can be found in a separate section of this report.

Internal Standard (IS) - Terminology used to reference a compound which is added to the sample extract and standard solutions of the analytes of interest immediately before instrumental analysis. The purpose of the internal standard is to provide data which can be used to correct for variations in injected volumes of extract and standard solutions which may occur during instrumental analysis. The internal standard must not be present in the sample extract. Typical internal standards used in mass spectrometric analyses are benzene-d6, naphthalene-d8, anthracene-d10 and chrysene-d12. These deuterated compounds produce unique mass spectra and are not present in naturally-occurring samples. A commonly used internal standard for GC/EC analysis is hexachlorobenzene.

Internal Standard Quantitation Method - Terminology which refers to a method of data analysis. This method requires that a known amount of an internal standard be added to the sample extract prior to instrumental analysis. Responses of all analytes are calculated relative to the internal standard using the following relationship:

$$RR = \text{Relative Response} = \frac{\text{(Analyte Area)}}{\text{(Internal Standard Area)}}$$

If different volumes of the sample extracts and standard solutions are injected, these differences are reflected by differences in the internal standard area. In addition, if instrument sensitivity changes, these changes are also reflected in changes in the internal standard area. The calculation of the relative response corrects for these types of fluxuations.

In order to determine the analyte concentration-Relative Response relationship, analyte Relative Response Factors are calculated using the following relationship for data obtained from the analysis of standard solutions:

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$$\text{RRF} = \text{Relative Response Factor} = \frac{(\text{Analyte Area})\ (\text{Internal Standard Concentration})}{(\text{Internal Standard Area})\ (\text{Analyte Concentration})}$$

When identical amounts of the internal standard are added to both extracts and standard solutions, the internal standard concentration term in the above equation can be eliminated. When this is done and Relative Response is substituted for the ratio of analyte area to internal standard area, the RRF equation becomes:

RRF = RR/(Analyte Concentration)

Analyte concentrations in the extracts are calculated from the following equation:

Analyte Extract Concentration = RR/RRF

In order to obtain the concentration of the analyte in the sample, the following equation is used:

$$\text{Analyte Sample Concentration} = \frac{(\text{Analyte Extract Concentration})\ (\text{Extract Volume})}{(\text{Amount of Sample Extracted})}$$

The area terms in the above equations may be total ion areas, FID peak areas, EC peak areas, or extracted ion areas. The details of how the internal standard quantitation method is applied to specific methods are described in the various Instrumental Standard Analysis Methods.

The following equation shows what calculations should be made to correct for analytical bias:

$$\underset{\text{(Corrected for Analytical Bias)}}{\text{Analyte Sample Concentration}} = \frac{(\text{Analyte Sample Concentration})}{(\text{Assumed Analyte Recovery})}$$

The Assumed Analyte Recovery can be obtained from an average of surrogate recoveries or native analyte recoveries, measured for the sample in question. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

External Standard Quantitation Method - Terminology which refers to a method of data analysis. This method requires that a known amount of the analyte standard be analyzed under the same conditions as the sample extract. In order to determine the analyte concentration-analyte response relationship, Response Factors are obtained from the following relationship:

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RF = Response Factor = (Analyte Area)/(Analyte Concentration)

Analyte concentrations in the extracts are calculated from the following equation:

Analyte Extract Concentration = (Analyte Area)/RF

In order to obtain the concentration of the analyte in the sample, the following equation is used:

$$\text{Analyte Sample Concentration} = \frac{(\text{Analyte Extract Concentration})\ (\text{Extract Volume})}{(\text{Amount of Sample Extracted})}$$

The area terms in the above equations may be total ion areas, FID peak areas, EC peak areas, or extracted ion areas. The details of how the external standard quantitation method is applied to specific methods are described in the various instrumental Standard Analysis Methods.

The following equation shows what calculations should be made to correct for analytical bias:

$$\text{Analyte Sample Concentration (Corrected for Analytical Bias)} = \frac{(\text{Analyte Sample Concentration})}{(\text{Assumed Analyte Recovery})}$$

The Assumed Analyte Recovery can be obtained from an average of surrogate recoveries or native analyte recoveries, measured for the sample in question. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Standard Analysis Methods - Terminology used to reference an organized description of the steps involved in the instrumental analysis of a sample or a sample extract. These methods are assigned short names which can also be used as computer file names for these methods. The names of specific instrumental Standard Analysis Methods which are used in this study are listed in the final report in the section entitled "Details". In addition, copies of these Standard Analysis Methods are shown in a separate section of this report. Flow sheets of the Standard Analysis Methods are usually also provided in this section.

Analysis Flow Sheet - Terminology used to reference a graphic description of a Standard Analysis Method. This flow sheet gives specific information on how extracts and standards are analyzed and what types of data reduction methods are used.

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Further Information

The general area of Extract Analysis, along with much of the terminology described above, is very complex. References provided in the previous section entitled "Analyte Extraction Terminology" may provide further information which may be useful in understanding instrumental analysis fundamentals and terminology.

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