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SAMPLING ANALYSIS REPORT
(Type of Report)

REPORT NO.: MSL-6806
ESC-UAG-87-68

JOB/PROJEC NO.: 760.28-6259

DATE: March 23, 1987

TITLE: ANALYSIS OF ONE DELAWARE RIVER OIL SAMPLE AND EIGHT WATER/SOIL SAMPLES FOR AROCLOR FORMULATIONS AND MAJOR COMPONENTS

AUTHORS: D. E. McKenzie, B. J. Simpson and B. M. Hughes

ABSTRACT: One oil sample and eight water/soil samples from the Delaware River Plant were analyzed for Aroclor formulations. In addition, the oil sample was analyzed for major chromatographable components. The oil sample was found to contain a mixture of Aroclors 1242 and 1248 at a concentration of approximately 9% (by weight). One of the eight water/soil samples contained approximately 50 ug/g Aroclor 1248 and the remaining samples contained from none detected (with an LOD of 0.005 ug/g) to 12 ug/g. Two water/soil samples contained approximately 75% water. The sample which contains greater than 50 ug/g must be treated as "PCB-containing" and the oil which contains approximately 9% Aroclor 1242/1248 must be treated as a PCB.

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REPT. NO.: ESC-UAG-87-68 AUTHORS: D. E. McKenzie, B. J. Simpson and B. M. Hughes
MSL-6806 TITLE: Analysis of One Delaware River Oil Sample and Eight Water/Soil Samples for Aroclor Formulations and Major Components
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ANALYSIS REPORT

DATE: July 6, 1987

LOG NUMBER: U-86-10-29-01
U-86-12-19-03

PROJECT NO: 760.28-6259

TO: Ed Jamro/Delaware River

FROM:

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

Phone No: (314) 694-1464

Title: Analysis of One Delaware River Oil Sample and Eight Water/Soil Samples for Aroclor Formulations and Major Components

ABSTRACT

One oil sample and eight water/soil samples from the Delaware River Plant were analyzed for Aroclor formulations. In addition, the oil sample was analyzed for major chromatographable components. The oil sample was found to contain a mixture of Aroclors 1242 and 1248 at a concentration of approximately 9% (by weight). One of the eight water/soil samples contained approximately 50 ug/g Aroclor 1248 and the remaining samples contained from none detected (with an LOD of 0.005 ug/g) to 12 ug/g. Two water/soil samples contained approximately 75% water. The sample which contains greater than 50 ug/g must be treated as "PCB-containing" and the oil which contains approximately 9% Aroclor 1242/1248 must be treated as a PCB.

INTRODUCTION

One oil and eight water/soil samples were analyzed for Aroclor formulations and major chromatographable components. The oil sample had been previously analyzed in 1983 and the results were reported in the final report for ESC Project Number 483.42718. Similar analysis methods were used for the present reanalysis of the same oil sample. The present study uses PCEX02 and MSAN02 Standard Extraction and Analysis Methods for the determination of the concentrations of Aroclor formulations and major components in the oil. Copies of these methods and flow sheets for these methods are shown in a later section of this report.

Aroclor formulation concentrations for the eight water/soil samples were determined by using Standard Extraction Methods PCEX02 and PCEX04 and Standard Analysis Method ECAN02. These methods and flow sheets are also included in a later section of this report. The amount of water present in two water/soil samples was determined by using Standard Analysis Method GRAN02, which is also described in a later section of this report.

(continued)

SUMMARY

PDA - #1 Oil

Tables 1 - 4 summarize results associated with the oil analysis and Figure 1 shows total ion chromatograms obtained from the duplicate analyses of PDA #1, and from analyses of the System Performance Standard and Method Blank. Table 1 shows the Aroclor analysis results. The Aroclor pattern in this sample appeared to be from a mixture of Aroclor 1242 and Aroclor 1248. Therefore these two formulations were used to determine the corresponding instrument response and PCB formulation concentrations were averaged to calculate an average PCB concentration. The average value of approximately 9% may be in error by as much as 100%. However, the conclusions that this oil contains PCBs on the order of 10% and thus must be treated as a PCB material, is unaffected by this analytical uncertainty.

Table 2 summarizes what concentrations and what major chromatographable components were detected in PDA - #1 Oil. All concentration values are calculated assuming the total ion response of the mass spectrometer for the identified compound is identical to the total ion response for anthracene-d10. This assumption is not strictly correct. Table 4 shows the calculated concentrations based upon this same assumption for the System Performance Standard which was analyzed at the same time as the oil sample. Major deviations of the calculated concentration from the correct concentration were seen for 1-decanol, pentachlorophenol, DFTPP, and chrysene.

Table 3 compares the current results with those reported in 1983. Although the absolute quantities are different in the two determinations, the identifications of the major components are the same. Any deviation between the 1983 results and the present results may be due to the samples not being truely identical.

Water/Soil Samples

The remaining tables summarize results from the analysis of water, soil, and water/soil samples. Table 6 shows quality control results of Aroclor 1260 spiking studies for analysis results with LODs of 1.0 ug/g and Table 9 shows quality control results of Aroclor 1260 spiking studies for analyses with LODs of 0.005 ug/mL. Table 7 summarizes the concentration of water present in 7VFC and 8VFC.

QC RESULTS

Good replicate values were obtained for Aroclor analysis results for the oil sample and for the determination of water in 8VFC. Surrogate recoveries greater than 70% were measured for all water/soil analyses. Aroclor 1260 spiking studies for two samples showed that greater than 80% of the correct values were determined when spiked at the Level of Detection and at ten times the level of detection.

(continued)

DETAILS

Standard Sampling Method No(s): Samples were supplied by Ed Jamro of the Delaware River Plant.

Standard Extraction Method No(s): PCEX02 (See PCFS02 for method flow sheet.)
PCEX04 (See PCFS04 for method flow sheet.)

Standard Analysis Method No(s): MSAN02 (See MSFS02 for method flow sheet.)
ECAN02 (See ECFS02 for method flow sheet.)
GRAN02 (See GRFS02 for method flow sheet.)

Sample Identification(s): See login information in a later section of this report.

Final Report File No(s): A25930

Table File No(s): B25930, TA259, TB259, TC259, B25918, C25918, D25918
E25918, F25918

Also included in this report are:

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

2) Extraction sheets for all samples analyzed.

3) Examples of capillary GC/EC chromatograms obtained and other raw data documentation.

4) Chain-of-custody information.

5) Information recorded at sample login.

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

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

ANALYST APPROVAL: B. Mason Hughes

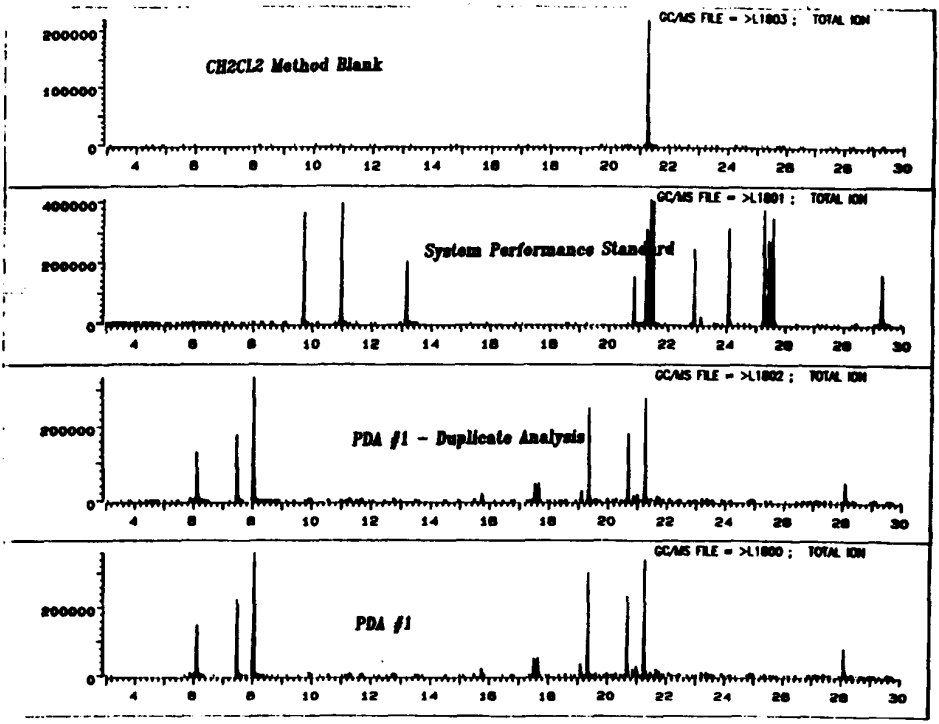


Figure 1. Comparisons of Total Ion Chromatograms Obtained From the Duplicate Analyses of PDA #1, the System Performance Standard, and the Method Blank.

Table 1. GC/EC Analysis Results for One Delaware River Oil Sample using Standard Extraction and Analysis Methods PCEX02 and ECAN02.

Project Number: 760.28-6259 Date: November 13, 1986

Sample Name	Raw File	ESC Extract Number	LOD(a) %	PCB Formulation(b) Concentration(%) A1242	A1248	Average Conc.(c) (%)
PDA #1	BR107:MH	PCB-1834	2	12.0	5.6	8.8
PDA #1 Duplicate Analysis	BR108:MH	PCB-1835	2	13.5	5.4	9.5

Footnotes:

a. LOD = Limit of Detection in units of weight %.

b. The sample was visually determined to contain a mixture of Aroclor 1242 and Aroclor 1248. Therefore the sample was analyzed for both Aroclor formulations. The values reported are based upon the use of either Aroclor 1242 or Aroclor 1248 for instrument calibration.

c. The average Aroclor concentration based upon the use of Aroclor 1242 and Aroclor 1248 for instrument calibration. This value would be accurate if the formulations were present in equal amounts.

B25930

Table 2. Concentration of Major Components Detected in Delaware River Plant Sample PDA - #1.

Project Number: 760.28-6259 Date: March 20, 1987

File FRN: >L1800 Sample ID: PDA- #1

RT (min)	Relative Conc.(a) (mg/mL)	Probable ID (b)	ID Code (c)	Probable MF (d)	MF Code (c)	Probable MW (e)	MW Code (c)
6.12	52	benzaldehyde	3	C7H6O	3	106	4
7.48	64	chloromethyl benzene	4	C7H7Cl	4	126	4
8.07	147	benzenemethanol	3	C7H8O	3	108	4
9.92	1.5	dichloromethyl benzene	2	C7H6Cl2	2	160	3
9.97	.6	dichloromethyl benzene	2	C7H6Cl2	2	160	2
11.29	1.8	unknown	-	------	-	162	1
11.70	.95	unknown	-	------	-	162	1
12.74	.8	unknown	-	------	-	184	1
15.76	4	dichloro benzenamine	1	C6H5Cl2N	1	198*	2
17.53	11	phenylmethyl methyl benzene	3	C14H14	3	182	3
17.66	13	dimethyl biphenyl	3	C14H14	4	182	4
19.10	8	trichloromethyl benzene	1	C7H5Cl3	1	232*	2
19.36	78	oxybis(methylene)bis benzene	3	C14H14O	3	198	2
20.69	58	unknown	-	------	-	238**	1
21.00	6	phenylmethyl ester benzoic acid	3	C14H12O2	3		
21.27	100	anthracene-d10	4	C14D10	4	188	4
21.75	2	trichloro benzeneamine	1	C6H4Cl3N	1	258*	2
22.09	1.3	unknown	-	------	-	234	2
22.45	.4	unknown monochlorinated compound	-	------	-	258	2
23.21	1.9	unknown	-	------	-	270	1
27.13	.35	unknown phthalate	2	------	-	226	1
27.40	.3	unknown	-	------	-	272	1
28.09	22	butylmethyl ester, benzenedicarboxylic acid	2	C19H2004	2	312	2
30.13	4	unknown phthalate*	2	------	-	---	-

Total 478.9 mg/mL

(continued)

Footnotes:

a) Relative concentration refers to the concentration calculated using the response factor from the anthracene-d10 which was added as an internal standard. All calculations were based on total ion response.
b) Probable ID refers to the component named as the best hit in a mass spectral data base search. Although individual names of compounds are listed, no isomeric identifications were possible. Therefore compound names may be taken to indicate a class of compounds, (i.e. trimethyl benzenes and propyl benzenes - C3 benzenes). An * after the component name indicates a no hit in the library search for that spectrum. However a class may have been assigned on the bases of characteristic fragment ions, (i.e. 149 is characteristic of phthalates).
c) ID code refers to an arbitrary code of confidence ranging from 1 to 4 in which probable ID is rated on the basis the best computer match and mass spectral interpretation experience. A rating of 1 signifies a low confidence of correct compound name, and a rating of 4 indicates a very high confidence that the compound has been correctly identified. Intermediate values, 2 and 3 are values which indicate with increasing certainty that the identity or class of the compound is correct. Molecular formula and molecular weights are also rated in a similar manner on the basis of individual spectral fragmentation information.
d) Probable MF refers to probable molecular formular.
e) Probable MW refers to probable molecular weight. A starred molecular weight indicates that the computer generated spectrum match is wrong, and represents a best guess of the correct molecular weight.

File name: TA259

Table 3. Concentration of Major Components Detected in Delaware River Plant Sample PDA - #1 and Reported in 1983 for Well 51-2.

Project Number: 760.28-6259 Date: March 20, 1987

RT (min)	PDA - #1 Relative Conc.(a) (mg/mL)	Probable ID (b)	Well 51-2 Relative Conc.(a) (mg/mL)
6.12	52	benzaldehyde	25
7.48	64	chloromethyl benzene	26
8.07	147	benzenemethanol	37
9.92	1.5	dichloromethyl benzene	0.58
9.97	.6	dichloromethyl benzene	1.1
11.29	1.8	unknown	1.2
11.70	.95	unknown	-
12.74	.8	unknown	-
15.76	4	dichloro benzenamine	1.5
17.53	11	phenylmethyl methyl benzene	35
17.66	13	dimethyl biphenyl	4
19.10	8	trichloromethyl benzene	2.5
19.36	78	oxybis(methylene)bis benzene	18
20.69	58	unknown	21
21.00	6	phenylmethyl ester benzoic acid	-
21.27	100	anthracene-d10	-
21.75	2	trichloro benzenamine	-
22.09	1.3	unknown	
22.45	.4	unknown monochlorinated compound	-
23.21	1.9	unknown	-
27.13	.35	unknown phthalate	14
27.40	.3	unknown	-
28.09	22	butylmethyl ester, benzenedicarboxylic acid	9.8
30.13	4	unknown phthalate*	3.5
Total	478.9 mg/mL		230 mg/mL

Footnotes:

a) Relative concentration refers to the concentration calculated using the response factor from the anthracene-d10 which was added as an internal standard. All calculations were based on total ion response.

b) Probable ID refers to the component named as the best hit in a mass spectral data base search. Although individual names of compounds are listed, no isomeric identifications were possible. Therefore compound names may be taken to indicate a class of compounds, (i.e. trimethyl benzenes and propyl benzenes = C3 benzenes). An * after the component name indicates a no hit in the library search for that spectrum. However a class may have been assigned on the bases of characteristic fragment ions, (i.e. 149 is characteristic of phthalates).

Table 4. System Performance Component Concentrations Calculated from Anthracene-d10 Total Ion Response.

Project Number: 760.28-6259 Date: March 20, 1987

File FRM: >L1801 Sample ID: System Performance Standard

RT (min)	Relative Conc.(a) (ug/mL)	Identification	Known Conc. (b) (ug/mL)	% Deviation from Known Conc. (%)
9.72	93	2,6-dimethyl phenol	117	19
10.98	106	2,6-dimethyl aniline	114	7
13.16	50	1-decanol	108	54
20.85	38	pentachlorophenol	91	58
21.23	100	anthracene-d10	100	0
21.40	100	1-octadecene	104	4
21.50	95	octadecane	107	11
22.91	55	DFTPP (ultramark 443)	112	51
24.09	81	eicosane	98	17
25.29	90	heneicosane	111	19
25.46	94	pyrene	113	17
25.60	83	methyl stearate	105	21
29.30	55	chrysene	100	45

Footnotes:

a) Relative concentration refers to the concentration calculated using the response factor from the anthracene-d10 which was added as an internal standard. All calculations were based on total ion response.

b) % Deviation from Known Conc. =
100 x [Known Conc. - Relative Conc.] / Known Conc.

File name: TB259

Table 5. Concentrations of Aroclor 1248, and % Recovery of surrogate compounds detected in Delaware River Soil samples using Standard Extraction Method PCEX02 and Standard Analysis Method ECAN02.

ESC Project No: 760.28 - 6259 Date: January 6, 1987

Sample I.D.	EXTRACT #	LOD(a) (ug/g)	CONC. (ug/g)	SURROGATE RECOVERY (%) CL5-benzene	SURROGATE RECOVERY (%) 2,4,6-CL3-biphenyl
1P/SO	PCB-1871	1.0	ND(b)	92	93
1P/SO-DUP.	PCB-1872	1.0	ND	88	93
1P/SO+LS	PCB-1873	1.0	ND	107	86
1P/SO+HS	PCB-1874	1.0	ND	98	83
2SFO	PCB-1879	1.0	ND	84	94
3SHT	PCB-1880	1.0	12	76	90
4PS/PS	PCB-1876	1.0	ND	82	87
5PS/SS	PCB-1875	1.0	ND	89	92
6SF/SS	PCB-1877	1.0	ND	99	97
6SF/SS-DUP.	PCB-1878	1.0	ND	86	98
7VFC	PCB-2020	1.0	35	nd(c)	nd
7VFC-DUP	PCB-2021	1.0	42	nd	nd
8VFC	PCB-2017	1.0	10	95	100
8VFC-DUP	PCB-2018	1.0	18	95	106
METHOD BLANK	PCB-2022	1.0	ND	100	101

Footnotes:

a. LOD = Limit of detection (in units of ug/g).
b. ND = No Aroclor formulation detected above the LOD.
c. nd = Surrogate compounds not detected because of extract dilution.

TABLE FILE NO: 825918:MH

Table 6. Summary of Aroclor 1260 Spiking Concentrations and Recoveries in Delaware River Soil Samples.

ESC Project No: 760.28 - 6259 Date: January 6, 1987

Sample I.D.	Aroclor 1260	
	Amount Added (ug/g)	Recovery (%)
1P/SO + LOW SPIKE	1	96
1P/SO + HIGH SPIKE	10	83

TABLE FILE NO: C25918:MH

Table 7. Concentrations of water in two Delaware River sediment samples determined using Standard Analysis Method GRAM02.

ESC Project No: 760.28 - 6259 Date: January 7, 1987

Sample I.D.	Water Concentration (%)
7VFC	71
8VFC	75
8VFC-Dup.	75
empty vial	1

TABLE FILE NO: D25918:MH

MONS 024554

Table 8. Concentrations of Aroclor 1248, and % Recovery of surrogate compounds detected in Delaware River Water samples using Standard Extretion Method PCEX04 and Standard Analysis Method ECAN02.

ESC Project No: 760.28 - 6259 Date: January 20, 1987

Sample I.D.	EXTRACT #	LOD(a) (ug/mL)	CONC. (ug/mL)	SURROGATE RECOVERY (%) CL3-benzene	SURROGATE RECOVERY (%) 2,4,6-CL3-biphenyl
4PS/PS	PCB-1958	.005	ND(b)	83	85
4PS/PS-DUP.	PCB-1959	.005	ND	76	78
5PS/SS	PCB-1960	.005	ND	90	83
5PS/SS-DUP.	PCB-1961	.005	ND	84	79
5PS/SS+LS	PCB-1962	.005	ND	73	70
5PS/SS+MS	PCB-1963	.005	ND	73	74
6SP/SS	PCB-1964	.005	ND	88	85
6SP/SS-DUP.	PCB-1965	.005	ND	68	67

Footnotes:

a. LOD = Limit of detection (in units of ug/mL).
b. ND = No Aroclor formulation detected above the LOD.

TABLE FILE NO: 825918:MM

Table 9. Summary of Aroclor 1260 Spiking Concentrations and Recoveries in Delaware River Water Samples.

ESC Project No: 760.28 - 6259 Date: January 20, 1987

Sample I.D.	Aroclor 1260	
	Amount Added (ug/mL)	Recovery (%)
5PS/SS+ LOW SPIKE	.005	83
5PS/SS+ HIGH SPIKE	.050	92

TABLE FILE NO: F25918:MH

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

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Non-EC Active Matrices

ESC Standard Extraction Method No:
PCEX02

MATRI(X),(CES):

Soils and other non-EC active solids and solvents

TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
0.5 - 5.0	10

TYPICAL ANALYTES: Aroclors 1232, 1242, 1248, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers

ESC Standard Analysis Method Nos:
ECAN01, ECAN02

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
pentachlorobenzene	50 - 120
2,4,6-trichlorobiphenyl	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson benzene or hexane for sample extraction or dilution.
Ultrasonic bath.
Nitrogen for extract volume reduction.
Disposable glassware.
(See flow sheet for this extraction method in PCFS02.)

LIMITATIONS: Method applies only to matrices which do not contain interfering electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region.

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

DATES OF METHOD CREATION AND UPDATES: 10/31/83, 2/3/84, 10/5/84

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: ---------- MRC EXTRACT # ----------

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

EXTRACTION DATE and TIME: ----------

SAMPLE COLOR AND DESCRIPTION ----------

		FINAL WEIGHT ----------
WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[ ]	INITIAL WEIGHT ----------
		SAMPLE WEIGHT ----------
SURROGATE SPIKE OF SAMPLE	[ ]	----------

ADD 10 mL BENZENE TO SAMPLE.	[ ]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME ---------- mL

EXTRACT NO: ----------

DATE/TIME COMPLETED ----------

ANALYST: ----------

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

ESC Standard Extraction Method (SEM) **ESC Standard Extraction Method No:**
TITLE: PCBs in Polymeric Matrices **PCEX04**

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Nylon yarn, Corex, Formic Acid, Vydyne, Nylon Salt, Adipic Acid, hexamethylene diamine, SEF fiber.	0.5 - 5.0	3

TYPICAL ANALYTES: Incidental PCBs (CL1-biphenyl through CL10-biphenyl) and other non-hydrolyzable compounds.

ESC Standard Analysis Method Nos: ECAN01, ECAN02, MSAN03

INTERFERENCES: Any compounds which produce major ions with m/e values the same as CL1-biphenyl through CL10-biphenyl isomers and which are not removed by the three-phase silica-gel column.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
pentachlorobenzene	50 - 120
2,4,6-trichloro-biphenyl	50 - 120
3,4,3',4'-CL4-biphenyl-d6	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson hexane for sample extraction or dilution. If extracts contain compounds which interfere with the characteristic ions of PCB isomers, a three-phase silica-gel column may be used.
(See flow sheet for this extraction method in PCFS04.)

LIMITATIONS: Method applies only to matrices which do not contain compounds with the same characteristic masses as the CL1-biphenyl through CL10-biphenyl isomers and which can not be removed using the three-phase silica-gel column. Method is applicable only when matrices are soluble in some type of water soluble solvent which is non miscible with hexane.

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

DATES OF METHOD CREATION AND UPDATES: 4/23/84

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: ---------- MRC EXTRACT # ----------

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

EXTRACTION DATE and TIME: ----------

WEIGH OUT SAMPLE INTO A 40 mL VIAL [ ]

SPIKE 100 uL ---------- [ ]

---------- .

---------- .

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

EXTRACT 3 X 3 mL WITH HEXANE [ ] [ ] [ ]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [ ]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [ ]

EXTRACT NO: ----------

DATE/TIME COMPLETED ----------

ANALYST: ----------

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

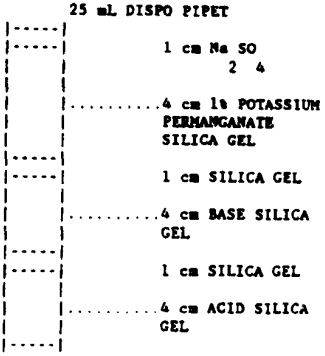
FINAL WEIGHT ----------

INITIAL WEIGHT ----------

SAMPLE WEIGHT ----------

[ ] RINSE COLUMN WITH 2 X 20 mL HEXANE

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET



MONS 024561

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

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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

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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 - <870107.1246>

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 TRAY OF HP-5880. [ ]

RECORD ELECTRON CAPTURE AND GAS CHROMATOGRAPHIC OPERATING PARAMETERS AND 3357-LAS FILE INFORMATION IN INSTRUMENT LOGBOOK. [ ]

CALCULATE RRFs FOR 10 - 30 PCB CONGENERS IN THE APPROPRIATE AROCLOR FORMULATION, USING THE AROCLOR CONCENTRATION AS THE ANALYTE CONCENTRATION FOR EACH PCB CONGENER. [ ]

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

Where:

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

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

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: .....

ESC Standard Analysis Method (SAM)
TITLE: Organic Compounds in Solvents

ESC Standard Analysis Method No: MSAN02

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5985/5987 Capillary GC/MS-Scanning with RTE-VI operating system.

Typical Run Time:	3 - 40 min.
Typical Multiplier Voltage:	1800 - 2400 eV
Typical Threshold:	100 counts
Typical Mass Range:	10 - 500 amu
Typical Scan Rate:	2/second

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Organic Compounds eluting between toluene and n-C34-ane	1	30	30

INTERFERENCES AND LIMITATIONS: Analyte must be chromatographically resolved from other organic compounds.

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

COLUMN TEMPERATURE PROGRAM: 50(4)/8/300 INJECTION TYPE: Splitless

---

CALIBRATION AND STANDARDIZATION:

Quantitative: Use either internal or external standard quantitation technique and calibrate instrument response factors either from the internal standard response or from other standard compounds analyzed under identical conditions.

Qualitative: Mass spectrum of unknown compounds are compared to standard mass spectra in the NBA/EPA Mass Spectral Library which contains over 30,000 compounds. Positive identification is made only after a trained mass spectroscopist has visually compared the unknown compound mass spectrum with those identified as most probable compounds based upon their Match Factor.

CALCULATIONS: Analyte concentration =

[internal standard concentration][(analyte area)/(internal standard area)]

- or -

[standard compound concentration][(analyte area)/(standard compound area)]

where: Areas are the total ion areas of the analyte, the internal standard or standard compound.

note: This calculation is made when unknown compounds are present or when no authentic standards are available.

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

DATE OF METHOD CREATION: 10/31/83 Time Stamp - <870320.1053>

FLOW SHEET MSFS02

FOR ESC ANALYSIS METHOD MSAN02

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

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

MS LOGBOOK PAGE NUMBER: ----------

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

PLACE 150 uL EXTRACT
AND 150 uL INTERNAL [ ]
STANDARD SOLUTION CONTAINING .
100 ug/mL ANTHRACENE-D10 IN .
CH2CL2 IN A 1.6 mL .
AUTOSAMPLER VIAL. .
.

PLACE VIAL IN AUTOSAMPLER [ ] CALCULATE TOTAL ION AREAS OF ANALYTES OF
TRAY OF HP-5985/87. . INTEREST AND ANTHRACENE-10 INTERNAL
. STANDARD.

RECORD MASS SPECTROMETRIC [ ] . . . . . .[ ]
AND GAS CHROMATOGRAPHIC .
OPERATING PARAMETERS IN .
INSTRUMENT LOGBOOK. .
.

[ ] CALCULATE CONCENTRATION
OF ANALYTES OF INTEREST
USING THE FOLLOWING
EQUATION:

$$\text{Analyte Concentration} = \frac{(\text{Analyte Total Ion Area}) \quad (\text{Internal Standard Concentration})}{(\text{Internal Standard Total Ion Area})}$$

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

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

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

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: PDA #1 ---------- MRC EXTRACT # PCB-1834

LOG NO: U-86-10-29-01

EXTRACTION DATE and TIME: DECEMBER 11, 1986

SAMPLE COLOR AND DESCRIPTION: DARK OIL

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.286 g.
SURROGATE SPIKE OF SAMPLE	[ ]	----------
ADD 9 mL BENZENE TO SAMPLE.	[X]	----------
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	SAMPLE COMPLETELY MISCIBLE. SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-1834

EXTRACT NO: ----------

DATE/TIME COMPLETED ----------

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

ESC JOB NO: 760.28-6259 ----------

MONS 024567

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: FDA #1 DUPLICATE — PCB-1835

MRC EXTRACT # U-86-10-29-01

LOG NO:

EXTRACTION DATE and TIME: DECEMBER 11, 1986

SAMPLE COLOR AND DESCRIPTION: DARK OIL

WEIGH OUT SAMPLE INTO A 14 OR 40 mL FIERCE VIAL	[X]	SAMPLE WEIGHT 1.298 g.
SURROGATE SPIKE OF SAMPLE	[ ]	
ADD 9 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	SAMPLE COMPLETELY MISCIBLE. SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-1835

EXTRACT NO:

DATE/TIME COMPLETED

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024568

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: METHOD BLANK ---------- MRC EXTRACT # PCB-1836

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

EXTRACTION DATE and TIME: DECEMBER 11, 1986

SAMPLE COLOR AND DESCRIPTION: CLEAR

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 0.000 g.
SURROGATE SPIKE OF SAMPLE	[ ]	----------

ADD 9 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	SAMPLE COMPLETELY MISCIBLE. SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-1836

EXTRACT NO: ----------

DATE/TIME COMPLETED ----------

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024569

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 1P/SO ---------- MRC EXTRACT # PCB-1871

LOG NO: U-86-12-19-03

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION: SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.220 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10620 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

EXTRACT NO: PCB-1871

DATE/TIME COMPLETED: JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 1P/SO + LOW SPIKE — MRC EXTRACT #: PCB-1873 U-86-12-19-03 — LOG NO:

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION: SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT: 1.295 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	LS- 100 uL. DEM 092386-01(10.82ug/mL) DILUTED 1:10. HS- 100 uL. DEM 092386-01(108.2ug/mL)
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME: 10 mL

EXTRACT NO: PCB-1873

DATE/TIME COMPLETED: JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024571

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 1P/SO + HIGH SPIKE

MRC EXTRACT # PCB-1874 U-86-12-19-03

LOG NO:

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.357 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	LS= 100 uL. DEM 092386-01(10.82ug/mL) DILUTED 1:10. HS= 100 uL. DEM 092386-01(108.2ug/mL)
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

EXTRACT NO: PCB-1874

DATE/TIME COMPLETED JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024572

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

5PS/SS — PCB-1875

SAMPLE ID: ---------- MRC EXTRACT # ----------

U-86-12-19-03

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

DECEMBER 22, 1986

EXTRACTION DATE and TIME: ----------

SOIL SAMPLE

SAMPLE COLOR AND DESCRIPTION ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	1.160 g. SAMPLE WEIGHT ----------
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 ---------- mL

PCB-1875

EXTRACT NO: ----------

JANUARY 20, 1987

DATE/TIME COMPLETED ----------

SIMPSON

ANALYST: ----------

760.28-6259

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

MONS 024573

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 4PS/PS ---------- MRC EXTRACT # PCB-1876

LOG NO: U-86-12-19-03

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.172 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10620 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

EXTRACT NO: PCB-1876

DATE/TIME COMPLETED JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 6SF/SS ---------- PCB-1877

MRC EXTRACT # U-86-12-19-03

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

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.398 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10620 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

EXTRACT NO: PCB-1877

DATE/TIME COMPLETED JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 6SF/SS ---------- MRC EXTRACT # PCB-1878

DUPLICATE ---------- LOG NO: U-86-12-19-03

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.303 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

EXTRACT NO: PCB-1878

DATE/TIME COMPLETED JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

2 SFO — PCB-1879

SAMPLE ID: ---------- MRC EXTRACT # ----------
U-86-12-19-03

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

DECEMBER 22, 1986

EXTRACTION DATE and TIME: ----------

SOIL SAMPLE

SAMPLE COLOR AND DESCRIPTION ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.307 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-1879

EXTRACT NO: ----------

JANUARY 20, 1987

DATE/TIME COMPLETED ----------

SIMPSON

ANALYST: ----------

760.28-6259

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

MONS 024577

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

3 SHT

PCB-1880

SAMPLE ID: ---------- MRC EXTRACT # ----------
U-86-12-19-03

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

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION: SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.117 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-1880

EXTRACT NO: ----------

DATE/TIME COMPLETED: JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024578

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

8 VFC

SAMPLE ID: ---------- PCB-1881

MRC EXTRACT # ---------- U-86-12-19-03

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

EXTRACTION DATE and TIME: DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT 1.360 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-1881

EXTRACT NO: ----------

DATE/TIME COMPLETED JANUARY 20, 1987

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024579

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

7 VFC

SAMPLE ID: ---------- PCB-1882

MRC EXTRACT # ---------- U-86-12-19-03

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

EXTRACTION DATE and TIME: ---------- DECEMBER 22, 1986

SAMPLE COLOR AND DESCRIPTION ---------- SOIL SAMPLE

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	SAMPLE WEIGHT ---------- 1.537 g.
SURROGATE SPIKE OF SAMPLE	[X]	100 uL. DEM 050885-01(10&20 ug/mL)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	SAMPLE SHAKEN ONLY.
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME ---------- 10 mL

PCB-1882

EXTRACT NO: ----------

DATE/TIME COMPLETED ---------- JANUARY 20, 1987

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

ESC JOB NO: ---------- 760.28-6259

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

4PS/PS — PCB-1958

SAMPLE ID: ---------- MRC EXTRACT # ----------

U-86-12-19-03

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

JANUARY 12, 1986

EXTRACTION DATE and TIME: ----------

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

30 mL. PIPPETED—

SAMPLE IN WATER.

30 g.

SAMPLE WEIGHT ----------

SPIKE 150 uL

---------- [X]

DEM 050885-01(1&2ug/mL)

---------- ----------

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

EXTRACT 3 X 3 mL WITH HEXANE {X} {X} [X]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

- 1 cm Na SO (2 4)
- 4 cm 1% POTASSIUM PERMANGANATE SILICA GEL
- 1 cm SILICA GEL
- 4 cm BASE SILICA GEL
- 1 cm SILICA GEL
- 4 cm ACID SILICA GEL
- GLASS WOOL

PCB-1958

EXTRACT NO: ----------

1-20-87

DATE/TIME COMPLETED ----------

SIMPSON

ANALYST: ----------

760.28-6259

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

MONS 024581

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: 4PS/PS DUPLICATE

PCB-1959

MRC EXTRACT # U-86-12-19-03

LOG NO:

EXTRACTION DATE and TIME: JANUARY 12, 1986

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

SAMPLE IN WATER.

SPIKE 150 uL [X]

DEM 050885-01(162ug/mL)

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

30 mL. PIPPETED-

SAMPLE WEIGHT 30 g.

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

EXTRACT 3 X 3 mL WITH HEXANE [X] [X] [X]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

- 1 cm Na SO 2 4
- 4 cm 1% POTASSIUM PERMANGANATE SILICA GEL
- 1 cm SILICA GEL
- 4 cm BASE SILICA GEL
- 1 cm SILICA GEL
- 4 cm ACID SILICA GEL
- GLASS WOOL

EXTRACT NO: PCB-1959

DATE/TIME COMPLETED 1-20-87

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

MONS 024582

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

5PS/SS — PCB-1960

SAMPLE ID: ---------- MRC EXTRACT # ----------
U-86-12-19-03

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

JANUARY 12, 1986

EXTRACTION DATE and TIME: ----------

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

30 mL. PIPPETED=

30 g.

SAMPLE IN WATER. SAMPLE WEIGHT ----------

SPIKE 150 uL

---------- [X]

DEM 050885-01(1&2ug/mL)

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

EXTRACT 3 X 3 mL [X] [X] [X] WITH HEXANE

APPLY EXTRACT TO [X] COLUMN QUANTITATIVELY

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

PCB-1960

EXTRACT NO: ----------

1-20-87

DATE/TIME COMPLETED ----------

SIMPSON

ANALYST: ----------

760.28-6259

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

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

	1 cm Na_2SO_4
4 cm 1% POTASSIUM PERMANGANATE SILICA GEL	
	1 cm SILICA GEL
4 cm BASE SILICA GEL	
	1 cm SILICA GEL
4 cm ACID SILICA GEL	
	GLASS WOOL

MONS 024583

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: 5PS/SS DUPLICATE

PCB-1961

MRC EXTRACT # U-86-12-19-03

LOG NO:

EXTRACTION DATE and TIME: JANUARY 12, 1986

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

SAMPLE IN WATER.

SPIKE 150 uL [X]
DEM 050885-01(162ug/mL)

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

EXTRACT 3 X 3 mL WITH HEXANE [X] [X] [X]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

PCB-1961
EXTRACT NO:

1-20-87
DATE/TIME COMPLETED

SIMPSON
ANALYST:

760.28-6259
ESC JOB NO:

30 mL. PIPPETED—

30 g.
SAMPLE WEIGHT

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

- 1 cm Na_2SO_4
- 4 cm 10 POTASSIUM PERMANGANATE SILICA GEL
- 1 cm SILICA GEL
- 4 cm BASE SILICA GEL
- 1 cm SILICA GEL
- 4 cm ACID SILICA GEL
- GLASS WOOL

MONS 024584

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: 5PS/SS + LOW SPIKE — PCB-1962

MRC EXTRACT #: U-86-12-19-03

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

EXTRACTION DATE and TIME: JANUARY 12, 1986

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

SAMPLE IN WATER.

SPIKE 150 uL [X]

DEM 050885-01(1&2ug/mL)

DEM 100886-01(1.0ug/mL)

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

EXTRACT 3 X 3 mL WITH HEXANE [X] [X] [X]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

PCB-1962

EXTRACT NO: ----------

DATE/TIME COMPLETED: 1-20-87

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

30 mL. PIPPETED-

SAMPLE WEIGHT: 30 g.

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

- 1 cm Na SO (2 4)
- 4 cm 1% POTASSIUM PERMANGANATE SILICA GEL
- 1 cm SILICA GEL
- 4 cm BASE SILICA GEL
- 1 cm SILICA GEL
- 4 cm ACID SILICA GEL
- GLASS WOOL

MONS 024585

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: 5PS/SS + HIGH SPIKE — MRC EXTRACT #: PCB-1963 — LOG NO: U-86-12-19-03

EXTRACTION DATE and TIME: JANUARY 12, 1986

WEIGH OUT SAMPLE INTO A 40 mL VIAL	[X]	30 mL. PIPPETED-
SAMPLE IN WATER.		SAMPLE WEIGHT 30 g.
SPIKE 150 uL	[X]	
DEM 050885-01(1&2ug/mL)		
DEM 100886-01(10 ug/mL)		
ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEP FIBERS)	[ ]	[X] RINSE COLUMN WITH 2 X 20 mL HEXANE
EXTRACT 3 X 3 mL WITH HEXANE	[X] [X] [X]	
APPLY EXTRACT TO COLUMN QUANTITATIVELY	[X]	
ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE.	[ ]	
NITROGEN BLOW DOWN TO 1 mL	[X]	

EXTRACT NO: PCB-1963

DATE/TIME COMPLETED: 1-20-87

ANALYST: SIMPSON

ESC JOB NO: 760.28-6259

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

- 1 cm Na SO (2 4)
- 4 cm 1% POTASSIUM PERMANGANATE SILICA GEL
- 1 cm SILICA GEL
- 4 cm BASE SILICA GEL
- 1 cm SILICA GEL
- 4 cm ACID SILICA GEL
- GLASS WOOL

MONS 024586

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

6SF/SS — PCB-1964

SAMPLE ID: ---------- MRC EXTRACT # ----------

U-86-12-19-03

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

JANUARY 12, 1986

EXTRACTION DATE and TIME: ----------

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

SAMPLE IN WATER.

SPIKE 150 uL

---------- [X]

DEM 050885-01(1&2ug/mL)

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

EXTRACT 3 X 3 mL WITH HEXANE [X] [X] [X]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

PCB-1964

EXTRACT NO: ----------

1-20-87

DATE/TIME COMPLETED ----------

SIMPSON

ANALYST: ----------

760.28-6259

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

30 mL. PIPPETED-

30 g.

SAMPLE WEIGHT ----------

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

CLEAN-UP COLUMN DESCRIPTION

25 mL DISPO PIPET

1 cm Na SO
2 4

4 cm 1% POTASSIUM PERMANGANATE SILICA GEL

1 cm SILICA GEL

4 cm BASE SILICA GEL

1 cm SILICA GEL

4 cm ACID SILICA GEL

GLASS WOOL

MONS 024587

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

6SF/SS — PCB-1965

SAMPLE ID: DUPLICATE — MRC EXTRACT #: U-86-12-19-03

LOG NO:

EXTRACTION DATE and TIME: JANUARY 12, 1986

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

SAMPLE IN WATER.

SPIKE 150 uL [X]

DEM 050885-01(1&2ug/mL)

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

EXTRACT 3 X 3 mL WITH HEXANE [X] [X] [X]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

PCB-1965

EXTRACT NO:

1-20-87

DATE/TIME COMPLETED

SIMPSON

ANALYST:

760.28-6259

ESC JOB NO:

30 mL. PIPETTED—

SAMPLE WEIGHT 30 g.

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET

- 1 cm Na_2SO_4
- 4 cm 1% POTASSIUM PERMANGANATE SILICA GEL
- 1 cm SILICA GEL
- 4 cm BASE SILICA GEL
- 1 cm SILICA GEL
- 4 cm ACID SILICA GEL
- GLASS WOOL

MONS 024588

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: METHOD BLANK ---------- MRC EXTRACT # PCB-1966 ----------

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

EXTRACTION DATE and TIME: JANUARY 12, 1986 ----------

WEIGH OUT SAMPLE INTO A 40 mL VIAL [X]

SAMPLE IN WATER.

SPIKE 150 uL ---------- [X]
DEM 050885-01(1&2ug/mL) ----------

---------- ----------

ADD 30 mL B&J WATER. [X]

30 mL. PIPPETED—

SAMPLE WEIGHT 0.0 g. ----------

[X] RINSE COLUMN WITH 2 X 20 mL HEXANE

EXTRACT 3 X 3 mL [X] [X] [X]
WITH HEXANE

APPLY EXTRACT TO COLUMN QUANTITATIVELY [X]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [X]

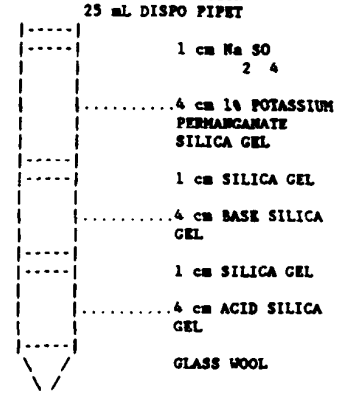
PCB-1966
EXTRACT NO: ----------

1-20-87
DATE/TIME COMPLETED ----------

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

760.28-6259
ESC JOB NO: ----------

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET



MONS 024589

FLOW SHEET GRFS02

FOR ESC EXTRACTION METHOD GRAN02

SAMPLE ID: SVFC ---------- MRC EXTRACT # GR-0001

U-86-12-19-03

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

EXTRACTION DATE and TIME: 12-22-86 / 3:00 PM

SAMPLE COLOR AND DESCRIPTION: BROWN SLUDGE

RECORD WEIGHING CONTAINER WEIGHT. (PIERCE VIAL, OR WEIGHING PAN)	[X]	CONTAINER WEIGHT	25.633 g [A]
WEIGH SAMPLE AND CONTAINER	[X]	SAMPLE AND CONTAINER WEIGHT	27.035 g [B]
		SAMPLE WEIGHT =[B]-[A] =	1.402 g [C]
PLACE WEIGH BOATS IN OVEN AT 120-130 DEGREES CENTIGRADE. FOR A MINIMUM OF 8 HOURS. RECORD OVEN TEMP AND TIME IN OVEN.		OVEN TEMP	120 degrees C.
		TIME IN OVEN	18 Hours
REMOVE SAMPLES FROM OVEN AND ALLOW TO REACH ROOM TEMPERATURE.	[X]		
WEIGH SAMPLE AND CONTAINER AN RECORD FINAL WEIGHT.	[X]	FINAL WEIGHT	25.983 g [D]
PERFORM CALCULATIONS LISTED BELOW.	[X]		

% NON BOUND WATER AND VOLATILE COMPOUNDS = 100 x ([B]-[D])/[C] = 75 %

EXTRACT NO: GR-0001 ---------- DATE/TIME COMPLETED 12-23-86 9:00 AM

ANALYST: SIMPSON ---------- ESC JOB NO: 760.28-6259

MONS 024590

FLOW SHEET GRFS02

FOR ESC EXTRACTION METHOD GRAN02

SAMPLE ID: SVFC (DUPLICATE) ---------- MRC EXTRACT # GR-0002

U-86-12-19-03

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

EXTRACTION DATE and TIME: 12-22-86 / 3:00 PM

SAMPLE COLOR AND DESCRIPTION: BROWN SLUDGE

RECORD WEIGHING CONTAINER WEIGHT. (PIERCE VIAL, OR WEIGHING PAN)	[X]	CONTAINER WEIGHT	25.402 g [A]
WEIGH SAMPLE AND CONTAINER	[X]	SAMPLE AND CONTAINER WEIGHT	26.548 g [B]
		SAMPLE WEIGHT -[B]-[A] =	1.146 g [C]
PLACE WEIGH BOATS IN OVEN AT 120-130 DEGREES CENTIGRADE. FOR A MINIMUM OF 8 HOURS. RECORD OVEN TEMP AND TIME IN OVEN.		OVEN TEMP	120 degrees C.
		TIME IN OVEN	18 Hours
REMOVE SAMPLES FROM OVEN AND ALLOW TO REACH ROOM TEMPERATURE.	[X]		
WEIGH SAMPLE AND CONTAINER AN RECORD FINAL WEIGHT.	[X]	FINAL WEIGHT	25.683 g [D]
PERFORM CALCULATIONS LISTED BELOW.	[X]		

% NON BOUND WATER AND VOLATILE COMPOUNDS = 100 x ([B]-[D])/[C] = 75 %

EXTRACT NO: GR-0002 ---------- DATE/TIME COMPLETED 12-23-86 9:00 AM

ANALYST: SIMPSON ---------- ESC JOB NO: 760.28-6259

MONS 024591

FLOW SHEET GRFS02

FOR ESC EXTRACTION METHOD GRAN02

SAMPLE ID: 7VFC ---------- MRC EXTRACT # GR-0003

LOG NO: U-86-12-19-03

EXTRACTION DATE and TIME: 12-22-86 / 3:00 PM

SAMPLE COLOR AND DESCRIPTION: BROWN SLUDGE

RECORD WEIGHING CONTAINER WEIGHT. (PIERCE VIAL, OR WEIGHING PAN)	[X]	CONTAINER WEIGHT	25.677 g [A]
WEIGH SAMPLE AND CONTAINER	[X]	SAMPLE AND CONTAINER WEIGHT	26.979 g [B]
		SAMPLE WEIGHT -[B]-[A] =	1.302 g [C]
PLACE WEIGH BOATS IN OVEN AT 120-130 DEGREES CENTIGRADE. FOR A MINIMUM OF 8 HOURS. RECORD OVEN TEMP AND TIME IN OVEN.		OVEN TEMP	120 degrees C.
		TIME IN OVEN	18 Hours
REMOVE SAMPLES FROM OVEN AND ALLOW TO REACH ROOM TEMPERATURE.	[X]		
WEIGH SAMPLE AND CONTAINER AN RECORD FINAL WEIGHT.	[X]	FINAL WEIGHT	26.055 g [D]
PERFORM CALCULATIONS LISTED BELOW.	[X]		

% NON BOUND WATER AND VOLATILE COMPOUNDS = 100 x ([B]-[D])/[C] = 71 %

EXTRACT NO: GR-0003 ---------- DATE/TIME COMPLETED 12-23-86 / 9:00 AM

ANALYST: SIMPSON ---------- ESC JOB NO: 760.28-6259

MONS 024592

FLOW SHEET GRFS02

FOR ESC EXTRACTION METHOD GRAN02

SAMPLE ID: BLANK VIAL ---------- MRC EXTRACT # GR-0004

U-86-12-19-03

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

EXTRACTION DATE and TIME: 12-22-86 / 3:00 PM

SAMPLE COLOR AND DESCRIPTION: BROWN SLUDGE

RECORD WEIGHING CONTAINER WEIGHT. (PIERCE VIAL, OR WEIGHING PAN)	[X]	CONTAINER WEIGHT	25.533 g [A]
WEIGH SAMPLE AND CONTAINER	[X]	SAMPLE AND CONTAINER WEIGHT	25.533 g [B]
		SAMPLE WEIGHT =[B]-[A] =	0.0 g [C]
PLACE WEIGH BOATS IN OVEN AT 120-130 DEGREES CENTIGRADE. FOR A MINIMUM OF 8 HOURS. RECORD OVEN TEMP AND TIME IN OVEN.		OVEN TEMP	120 degrees C.
		TIME IN OVEN	18 Hours
REMOVE SAMPLES FROM OVEN AND ALLOW TO REACH ROOM TEMPERATURE.	[X]		
WEIGH SAMPLE AND CONTAINER AN RECORD FINAL WEIGHT.	[X]	FINAL WEIGHT	25.521 g [D]
PERFORM CALCULATIONS LISTED BELOW.	[X]		

% NON BOUND WATER AND VOLATILE COMPOUNDS = 100 x ([B]-[D])/[C] = ? %

EXTRACT NO: GR-0004 DATE/TIME COMPLETED 12-23-86 9:00 AM

ANALYST: SIMPSON ESC JOB NO: 760.28-6259

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 8 VFC DUPLICATE ---------- MRC EXTRACT # PCB-2018 ----------

U-86-12-19-03

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

EXTRACTION DATE and TIME: 2-25-87 / 2:00 PM ----------

SAMPLE COLOR AND DESCRIPTION SANDY BROWN ? ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ---------- INITIAL WEIGHT ---------- SAMPLE WEIGHT 1.248 g ----------
SURROGATE SPIKE OF SAMPLE	[X]	100 uL DEM-050885-01 (DIL 1:10) ----------
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10.0 mL ---------- mL

EXTRACT NO: PCB-2018 ----------

DATE/TIME COMPLETED 2-25-87 / 4:00 PM ----------

ANALYST: MCKENZIE ----------

ESC JOB NO: 760.28-6259 ----------

MONS 024594

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

7 VFC

SAMPLE ID: ---------- MRC EXTRACT # PCB-2020

U-86-12-19-03

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

EXTRACTION DATE and TIME: 2-25-87 / 2:00 PM

SAMPLE COLOR AND DESCRIPTION SANDY BROWN ?

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ---------- INITIAL WEIGHT ---------- SAMPLE WEIGHT 0.934 g
SURROGATE SPIKE OF SAMPLE	[X]	100 uL DEM-050885-01 (DIL 1:10)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10.0 mL ---------- mL

PCB-2020

EXTRACT NO: ----------

DATE/TIME COMPLETED 2-25-87 / 4:00 PM

ANALYST: MCKENZIE

ESC JOB NO: 760.28-6259

MONS 024595

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: 7 VFC DUPLICATE ---------- PCB-2021

MRC EXTRACT # ---------- U-86-12-19-03

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

EXTRACTION DATE and TIME: 2-25-87 / 2:00 PM

SAMPLE COLOR AND DESCRIPTION ---------- SANDY BROWN ?

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ---------- INITIAL WEIGHT ---------- SAMPLE WEIGHT 1.072 g
SURROGATE SPIKE OF SAMPLE	[X]	100 uL DEM-050885-01 (DIL 1:10)
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10.0 mL ---------- mL

PCB-2021

EXTRACT NO: ----------

DATE/TIME COMPLETED 2-25-87 / 4:00 PM ----------

ANALYST: MCKENZIE ----------

ESC JOB NO: 760.28-6259 ----------

MONS 024596

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: METHOD BLANK ---------- MRC EXTRACT # PCB-2022 U-86-12-19-03

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

EXTRACTION DATE and TIME: 2-25-87 / 2:00 PM

SAMPLE COLOR AND DESCRIPTION ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ---------- INITIAL WEIGHT ---------- 0.000 g SAMPLE WEIGHT ----------
SURROGATE SPIKE OF SAMPLE	[X]	100 uL DEM-050885-01 (DIL 1:10) ----------
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10.0 mL ---------- mL

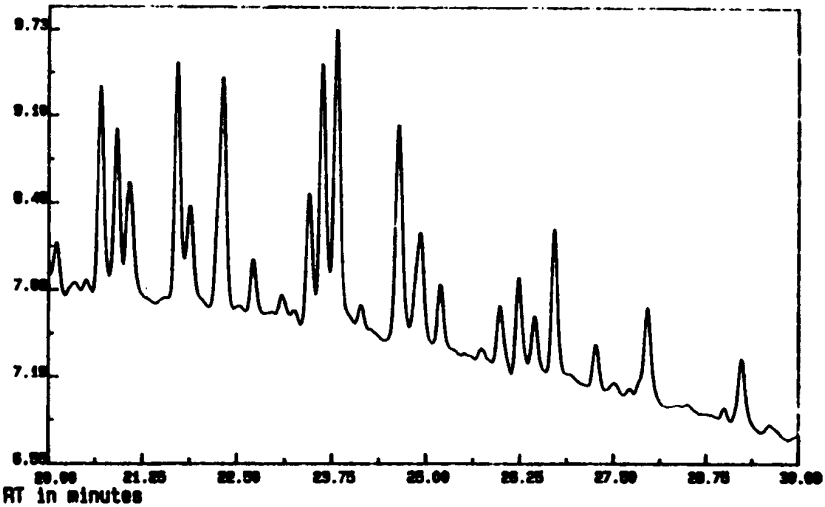
EXTRACT NO: PCB-2022

DATE/TIME COMPLETED 2-25-87 / 4:00 PM

ANALYST: MCKENZIE

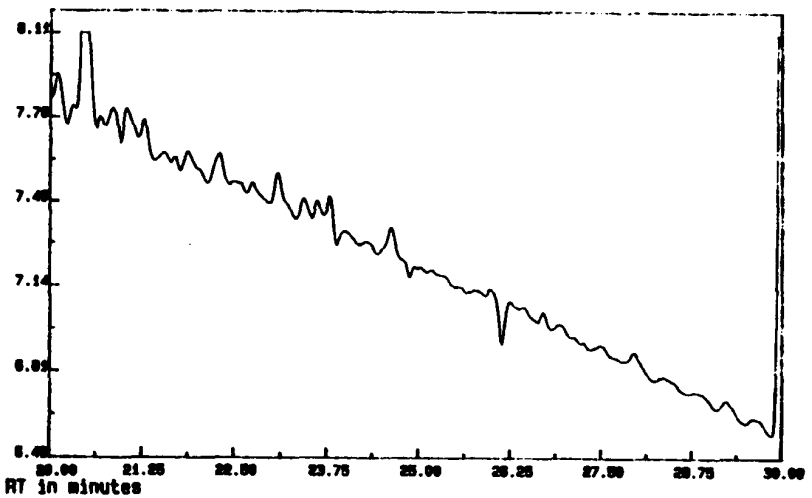
ESC JOB NO: 760.28-6259

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



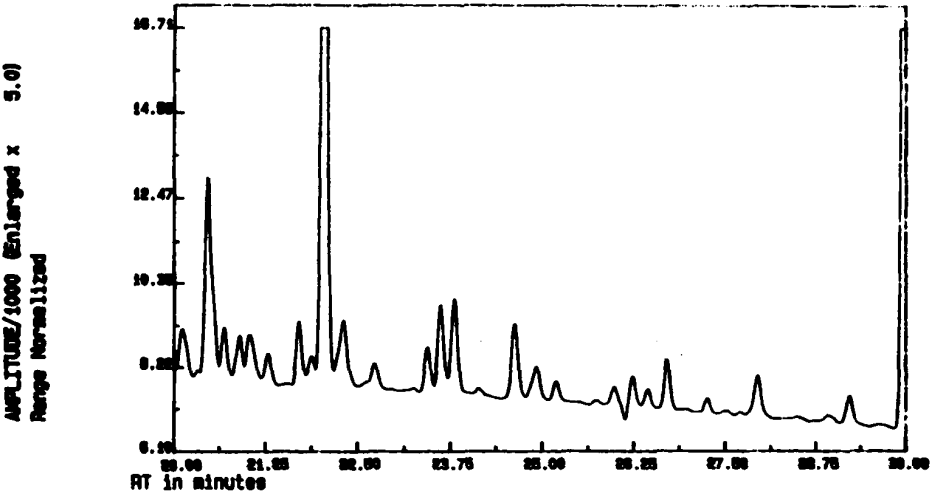
SAMPLE: A1248 (1.0) INJECTED AT 16:48:18 ON DEC 29, 1986
Meth: PCBSSM Raw: BR403 Proc: BP403

MONS 024599



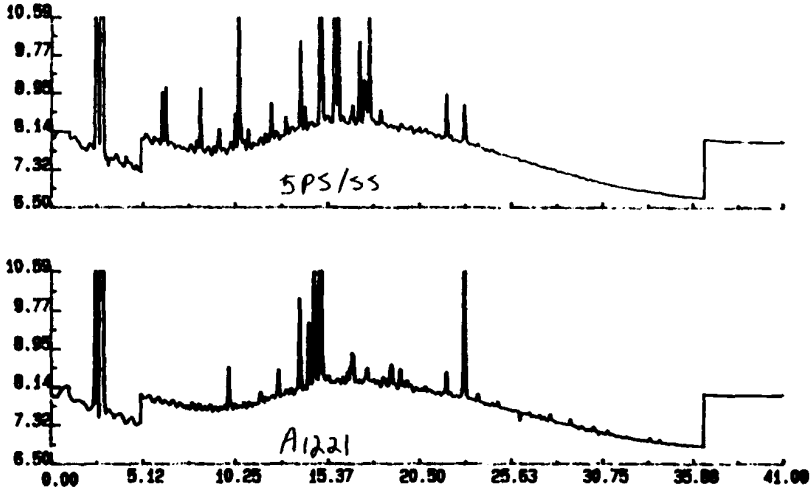
SAMPLE: P1871 INJECTED AT 19:36:10 ON DEC 29, 1986
Meth: PCBSSM Raw: BR406 Proc: BP406

MONS 024600



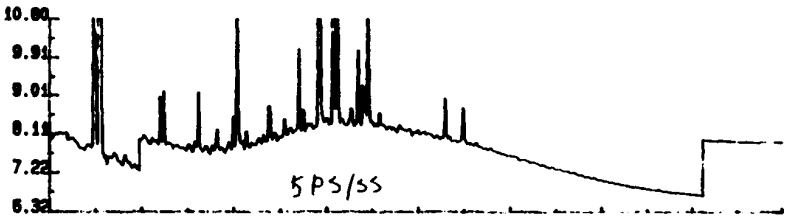
SAMPLE: P1880 INJECTED AT 5:12:42 ON DEC 30, 1986
Meth: PCBSSM Raw: BR417 Proc: BP417

MONS 024601



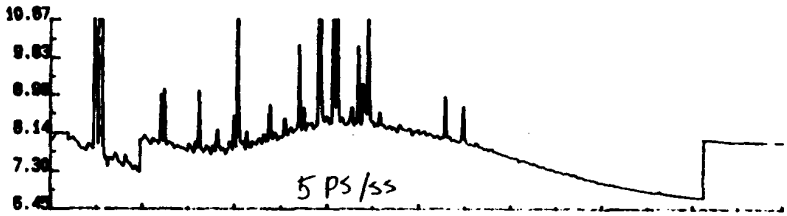
Relative Plots
Top Raw: BR511 (0.0- 41.0) (Enlarged x 30.0)
Bottom Raw: BR502 (0.0- 41.0) (Enlarged x 30.0)

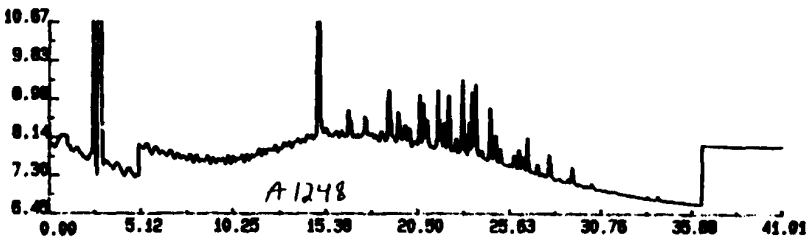
MONS 024602





Relative Plots
Top Raw: BR511 (0.0- 41.0) (Enlarged x 30.0)
Bottom Raw: BR520 (0.0- 41.0) (Enlarged x 30.0)





Relative Plots
Top Raw: BR511 (0.0- 41.0) (Enlarged x 30.0)
Bottom Raw: BR504 (0.0- 41.0) (Enlarged x 30.0)

MONS 024604

Results for [illegible] Jamero Rush Sample (6259)

	Standard		Sample		
Cl_6	157544		186425		
		.845879			
19.00	11945	14788.	17500		1.23
21.37	6954		~~7005~~	7005	0.8
23.80	8030		8078		0.85
26.70	5218		5435		0.88
22.21	10760		11826		0.93
don't use 20.69	6937		4778		0.58
24.63	8745		9862		0.95
27.94	4405		5504		1.06
22.44	3856		4328		.95
19.49	5054		6665		1.11

$0.978\ ug/ml \times \frac{vol_f}{wt_s}$

3SHT

$\frac{9.78\ ug}{1.117\ g} = 8.76\ ug/g$

(approximate at 10.0 ug/g of)

MONS 024605

The following pages show chain-of-custody information for this project.

MONSANTO
Delaware River Plant
Bridgeport, N. J. 08014

CHAIN-OF—CUSTODY RECORD

№ 145

PROJECT LOCATION	NAME OF AREA	PROJECT TELEPHONE NO.	PROJECT NUMBER
PDA #1	PDA #1	(609) 467-3000 X266	

ITEM NUMBER	SAMPLE NUMBER	NUMBER & SIZE OF CONTAINERS	DESCRIPTION		TRANSFER NUMBER & CHECK 1 2 3 4 5 6 7
1	4 PS/PS	1 16oz Glass	50/50 Mixture of Phase Separator OUTLET from PDA #1 & WTP PRIMARY SLUDGE	Instructions ↓ ↓ [illegible] Centrifuge Sample discard solids	
2	5 PS/SS	1 16oz Glass	50/50 Mixture of Phase Separator OUTLET from PDA #1 & WTP Secondary SLUDGE		
3	6 SF/SS	1 16oz Glass	50/50 Mixture OF SAND FILTER OUTLET FROM PDA #1 & WTP Secondary SLUDGE	Run PCB on Remaining Liquid	

Person Responsible for sample	Affiliation	Date	Time
E. JAMBO	Monsanto	[illegible]	

Purpose of analysis (use back of front sheet if necessary)

RUN Total SOLUBLE PCB's

TRANSFER NUMBER	ITEM NUMBER	TRANSFERS RELINQUISHED BY	RECEIVED BY	DATE	TIME
1	1-2	E. JAMBO	[illegible]	[illegible]	[illegible]
2					
3					
4					
5					
6					

MONS 024607

MONSANTO
Delaware River Plant
Bridgeport, N. J. 08014

CHAIN-OF—CUSTODY RECORD

№ 144A

PROJECT LOCATION	NAME OF AREA	PROJECT TELEPHONE NO.	PROJECT NUMBER
PDA #1	PDA #1	(609) 467-3000 X266	

ITEM NUMBER	SAMPLE NUMBER	NUMBER & SIZE OF CONTAINERS	DESCRIPTION		TRANSFER NUMBER & CHECK 1 2 3 4 5 6 7
1	1 P/S O	1 - Qt Glass	Phase Separator outlet 12/17/86		✓
2	2SFO	1 - Qt Glass	Sand Filter outlet 12/17/86.	Run Total PCB's A-1248	✓
3	3SHT HIGH PRIORITY	1 - Qt. Glass	Sludge Hold Tank 12/17/86		✓
4	7VFC	1 - 16oz Glass	VACUUM FILTER CAKE (11/12/86)	Run % Solids	✓
5	8VFC	1 - 16oz Glass	VACUUM FILTER CAKE (10/27/86)		✓

Person Responsible for sample	Affiliation	Date	Time
E. JARRO	Monsanto	12/18	

Purpose of analysis (use back of front sheet if necessary)

Run Total PCB's on Samples 1-5

Run % Solids on Samples 4-5

TRANSFER NUMBER	ITEM NUMBER	TRANSFERS RELINQUISHED BY	ACCEPTED BY	DATE	TIME
1	1-5	E. JARRO	[signature]	[illegible]	[illegible]
2					
3					
4					
5					
6					

MONS 024608

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

Time Stamp - <870325.1306>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: OCTOBER 30, 1986 LOG NO: U-86-10-29-01

REQUESTER: JAMRO/DELAWARE RIVER PROJECT NO: 760.28-6259

LOGIN NOTEBOOK PAGE: 45

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

ANALYSIS REQUESTS SENT TO: FILES, MEES, SHERMAN, HUGHES, McKENZIE

SAMPLE PICKUP BY: SIMPSON

SAMPLE LOCATION: U414

SAMPLE DESCRIPTION FOR 1 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
PDA #1	1 X 3oz.	PCB-1834 PCB-1835-DUP PCB-1834+LS PCB-1834+HS WS-1700 WS-1700-DUP
Method Blank		PCB-1836

Note- DUP refers to duplicate analysis, LS refers to a low level native spike of the extract, and HS refers to a high level native spike of the extract.

RECORDED BY: SIMPSON

REQUIRED ANALYSES: PCBs

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158618

ANALYTICAL TECHNIQUES:

TECHNIQUE (EM)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
x GCGC/MS (30)	3-19-87	MSAN25	157142
x GC/EC (30)	11-10-86	ECAN03	157283

FINAL REPORT NOTEBOOK PAGE: 3,434,455

OTHER NOTEBOOK PAGES:

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS: SAMPLE SPLIT IN HALF FOR DBF ANALYSIS ALSO.

SAMPLE DISPOSITION: HOLD FOR 60 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA259:LI

Time Stamp - <870327.1129>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: DECEMBER 22, 1986 LOG NO: U-86-12-19-03

REQUESTER: ED JAMRO/DELAWARE RIVER PROJECT NO: 760.28-6259

LOGIN NOTEBOOK PAGE: 49

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

ANALYSIS REQUESTS SENT TO: FILES, MEES, SHERMAN, HUGHES, McKENZIE

SAMPLE PICKUP BY: SIMPSON

SAMPLE LOCATION: U420

SAMPLE DESCRIPTION FOR 8 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
1P/SO	1 X 1 qt.	PCB-1871,1872, 1873,1874
2SFO	1 X 1 qt.	PCB-1879
3SHT	1 X 1 qt.	PCB-1880
7VFC	1 X 16 oz.	PCB-2020,2021 GR-0003
8VFC	1 X 16 oz.	PCB-2017,2018 GR-0001,GR-0002
BLANK VIAL		GR-0004
4PS/PS	1 X 16 oz.	PCB-1876,1958, 1959
5PS/SS	1 X 16 oz.	PCB-1875,1960, 1961,1962,1963
6SF/SS	1 X 16 oz.	PCB-1877,1878, 1964,1965
METHOD BLANK		PCB-2022

RECORDED BY: SIMPSON

REQUIRED ANALYSES: PCB

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158621, 158622

ESC STANDARD EXTRACTION METHOD(S): PCEX02,PCEX04,GRAN02

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
x GC/EC (18)	3/19/87	ECAN02	157288

FINAL REPORT NOTEBOOK PAGE: 3,434,455

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: AB259:LI

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

8 VFC

SAMPLE ID: ---------- MRC EXTRACT # PCB-2017

U-86-12-19-03

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

2-25-87 / 2:00 PM

EXTRACTION DATE and TIME: ----------

SANDY BROWN ?

SAMPLE COLOR AND DESCRIPTION ----------

		FINAL WEIGHT ----------
WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	INITIAL WEIGHT ----------
		0.967 g
		SAMPLE WEIGHT ----------
SURROGATE SPIKE OF SAMPLE	[X]	100 uL DEM-050885-01 (DIL 1:10)

ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[X]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
		10.0 mL
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME ---------- mL

PCB-2017

EXTRACT NO: ----------

2-25-87 / 4:00 PM

DATE/TIME COMPLETED ----------

MCKENZIE

ANALYST: ----------

760.28-6259

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

MONS 024614

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 Mees (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 unduly 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 procedures 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.

(continued)

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, at. 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.

(continued)

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.

(continued)

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:

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

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:

(continued)

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

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:

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

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.

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:

(continued)

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:

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

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:

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

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.

(continued)

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.

INANTE:MH