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REPORT NO.: MSL-5911
ESC-UAG-86-70

JOB/PROJECT NO.: 6223

DATE: August 5, 1986

TITLE: ANALYSIS OF QUEENY PLANT SOIL SAMPLES FOR PCBs AND OTHER CHROMATOGRAPHABLE COMPOUNDS

AUTHORS: B.M. Hughes, D. McKenzie, and C. Trang

ABSTRACT: Seven soil samples from the Queeny Plant were analyzed for PCBs ad other chromatographable compounds using capillary GC/EC and GC/MS analysis methods. No sample contained greater than 10 ug/g PCBs. Sulfur (S8) was the only other chromatographable compound detected at concentrations greater than approximately 100 ug/g.

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APPROVAL DATE: August 7, 1986

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MONS 024164

REPT NO.: MSL-5911
ESC-UAG-86-70
AUTHORS: B.M. Hughes, D. McKenzie and C. Trang
TITLE: ANALYSIS OF QUEENY PLANT SOIL SAMPLES FOR PCBs AND OTHER CHROMATOGRAPHABLE COMPOUNDS
COPY NO.:

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ANALYSIS REPORT

DATE: July 3, 1986

LOG NUMBER: U-86-05-05-02 PROJECT NO: 760.28-6223

TO: Ken Perica (1760)/Queeny Plant

FROM:

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

Phone No: (314) 694-1464

Title: Analysis of Queeny Plant Soil Samples for PCBs and Other Chromatographable Compounds

ABSTRACT

Seven soil samples from the Queeny Plant were analyzed for PCBs and other chromatographable compounds using capillary GC/EC and GC/MS analysis methods. No sample contained greater than 10 ug/g PCBs. Sulfur (S8) was the only other chromatographable compound detected at concentrations greater than approximately 100 ug/g.

INTRODUCTION

Soil core samples were taken by the Queeny Plant to document the levels of organic contamination which may be present. This study is a part of a building dismantling and land re-use project. These samples were extracted using Standard Extraction Method PCEX24. Details of this method and flow sheet PCFS24 are included in a separate section of this report. The resulting extracts were then analyzed for PCBs using Standard Analysis Method ECAN02 and for other chromatographable compounds using MSAN02. Copies of these methods and flow sheets ECFS02 and MSFS02 are also included in a later section of this report.

SUMMARY

Tables 1 and 2 summarize the capillary GC/EC results for PCBs in soils. Note that no sample contained greater than 10 ug/g PCBs. Tables 3 through 5 summarize the capillary GC/MS results for total chromatographable components in these soil extracts. Only sulfur (S8) was detected in any of these samples. Figures 1 - 3 show total ion chromatograms obtained from the GC/MS analysis of Method Blank, Soil Blank, and the seven soil extracts. The major four peaks in these chromatograms are due to phenol-d5/2-chlorophenol-d4, 1,2-CL2-benzene, anthracene-d10 and pyrene-d10 which were added to the soils and extracts before GC/MS analysis. Recoveries of the surrogate compounds are reported in Tables 3 through 5.

(continued)

QC RESULTS

Quality control data are shown in Tables 1 - 5 and in Figures 1 and 2. These tables and figures show surrogate compound recoveries, duplicate analysis results and method blank and soil blank analysis results.

DETAILS

Standard Sampling Method No(s): Samples supplied by Ken Perica (1760).

Standard Extraction Method No(s): PCEX24 (See PCFS24 for flow sheet)

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

Sample Identification(s): #1, 2.5 - 4
#3, 2.5 - 4
#5, 2.5 - 4
#8, 2 - 3
#1, 7.5 - 9
#4, 2.5 - 4
#7, 2 - 3

Final Report File No(s): A22318

Table File No(s): B22318, C22318, D22318, E22318, F22318

Also included in this report are:

(1) Extraction and Analysis Methods used for this project. This section shows extraction methods (PCEX24/PCFS24), capillary GC/EC methods (ECAN02/ECFS02), and capillary GC/MS methods (MSAN02/MSFS02) used for this project.

(2) Calculations or raw data documentation of surrogate recoveries.

(3) Extraction details for each sample extracted.

(4) Chain-of-custody information.

(5) Analytical Request Information generated at login.

(6) Important Quality Assurance/Quality Control, Analyte Extraction, and Instrumental Analysis Terminology used in this report.

ANALYST(S): B. Mason Hughes, David McKenzie, and Chi Trang

ANALYST APPROVAL: B. Mason Hughes

Table 1. Results of capillary GC/EC analyses of Soil samples using Standard Analysis Method ECAN02

ESC Project No: 760.18 - 6223 Date: May 28, 1986

Sample I.D.	Surrogate Recoveries(%Recovery)		PCB Concentration (ug/g)				
	CL5-benzene	2,4,6-CL3-biphenyl	A1248	A1254	A1260	A1262	Total
Method Blank	111	114	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
Soil Blank	108	110	"	"	"	"	"
#1 2.5-4	-	108	"	"	"	"	"
" (DUP.)	-	108	"	"	"	"	"
#1 7.5-9	-	116	"	"	"	"	"
#3 2.5-4	82	118	2.0	"	"	"	2.0
#4 2.5-4	108	113	10.0	"	"	"	10.0
#5 2.5-4	107	110	5.0	"	"	"	5.0
#7 2-3	109	112	ND(a)	"	"	"	ND(a)
#8 2-3	102	110	6.0	"	"	"	6.00

Footnotes:

a. ND= None Detected (<1.0 ug/g)

TABLE FILE NO: B22318:MH

MONS 024168

TABLE 2. Summary of Aroclor 1254 Spiking Concentrations and Recoveries in Quality Control Experiments for Soil Samples using Standard Analysis Method ECAN02

ESC Project No: 760.18 - 6223 Date: May 23, 1986

Sample I.D.	Surrogate Recoveries (%Recovery)		Aroclor 1254	
	CL5-benzene	2,4,6-CL3-Biphenyl	Amount Added (ug/g)	Recovery (%)
Soil Blank + L. SP	101	104	1.00	107
Soil Blank + H. SP	107	110	10.00	107
#7 2-3 + L. SP	112	115	1.00	121
#7 2-3 + H. SP	111	112	10.00	121

TABLE FILE NO: C22318:MH

Table 3. Results of Wide-Scan Capillary GC/MS Analysis of Soil Samples using Standard Analysis Method MSAN02.

Time Stamp - <860804.1451>

Project No: 760.28-6223

Date: July 3, 1986

		Sample Identification		
		#1 2.5 - 4	#1 2.5 - 4 (duplicate)	#1 7.5 - 9
Analyte	LOQ(a) (ug/g)	Concentration (ug/g) (b)		
Sulfur (S8)	100(N)	25	99	151
Total Chromatographable Compounds (ug/g)		25	99	151
Surrogate Spiking Compound	**Amount Added(ug/g)**	**Recovery (%)**		
Phenol-d5	500	99	94	93
2-CL-phenol-d4	500	101	96	93
1,2-CL2-benzene	500	106	100	94
Pyrene-d10	500	108	84	100
Documentation				
ESC Extraction Method		PCEX24	PCEX24	PCEX24
Date of Extraction		7/3/86	7/3/86	7/3/86
Sample Amount Extracted (g)		1.062	1.184	1.243
Extract Final Volume (ml)		10.0	10.0	10.0
Extract Dilution		none	none	none
ESC Extract or Sample Number		WS-1553	WS-1554	WS-1555
Date of CGC/MS Analysis		5/12/86	5/12/86	5/12/86
MS FRN		>L1215	>L1217	>L1218

Footnotes:

(a) LOQ - Limit of Quantitation in ug/g. Signal-to-noise ratio is greater than 10 when this amount is analyzed using the above referenced Analytical and Extraction Methods. The following letters indicate how this value was determined:
N - measured from native spiking of sample matrix.

(b) Concentrations calculated assuming the total ion response of organic compounds to be equal to the total ion response of anthracene-d10 internal standard.

Table stored in file: D22318

Analyst(s): B. Mason Hughes and David McKenzie

Analyst Approval: B. Mason Hughes

MONS 024170

Table 4. Results of Wide-Scan Capillary GC/MS Analysis of Soil Samples using Standard Analysis Method MSAN02.

Time Stamp - <860804.1451>

Project No: 760.28-6223 Date: July 3, 1986

		Sample Identification		
		#3 2.5 - 4	#4 2.5 - 4	#5 7.5 - 9
Analyte	LOQ(a) (ug/g)	Concentration (ug/g) (c)		
Sulfur (S8)	100(N)	NQ(b)	NQ	NQ
Total Chromatographable Compounds (ug/g)		NQ	NQ	NQ
Surrogate Spiking Compound	**Amount Added(ug/g)**	**Recovery (%)**		
Phenol-d5	500	103	84	97
2-CL-phenol-d4	500	109	84	98
1,2-CL2-benzene	500	112	89	100
Pyrene-d10	500	86	105	110
Documentation				
ESC Extraction Method		PCEX24	PCEX24	PCEX24
Date of Extraction		7/3/86	7/3/86	7/3/86
Sample Amount Extracted (g)		1.654	1.396	1.171
Extract Final Volume (ml)		10.0	10.0	10.0
Extract Dilution		none	none	none
ESC Extract or Sample Number		WS-1556	WS-1557	WS-1558
Date of CGC/MS Analysis		5/12/86	5/12/86	5/12/86
MS FRN		>L1219	>L1220	>L1221

Footnotes:

(a) LOQ - Limit of Quantitation in ug/g. Signal-to-noise ratio is greater than 10 when this amount is analyzed using the above referenced Analytical and Extraction Methods. The following letters indicate how this value was determined:
N - measured from native spiking of sample matrix.

(b) NQ - Not Quantitatable. The congener concentration is below the LOQ value.

(c) Concentrations calculated assuming the total ion response of organic compounds to be equal to the total ion response of anthracene-d10 internal standard.

Table stored in file: E22318

Analyst(s): B. Mason Hughes and David McKenzie

Analyst Approval: B. Mason Hughes

MONS 024171

Table 5. Results of Wide-Scan Capillary GC/MS Analysis of Soil Samples using Standard Analysis Method MSAN02.

Time Stamp - <860804.1452>

Project No: 760.28-6223 Date: July 3, 1986

		Sample Identification	
		#7 2 - 3	#8 2 - 3
Analyte	LOQ(a) (ug/g)	Concentration (ug/g) (c)	
Sulfur (S8)	100(N)	NQ(b)	NQ
Total Chromatographable Compounds (ug/g)		NQ	NQ

Surrogate Spiking Compound	Amount Added(ug/g)	Recovery (%)	
Phenol-d5	500	95	105
2-CL-phenol-d4	500	94	105
1,2-CL2-benzene	500	100	109
Pyrene-d10	500	93	98

Documentation		
ESC Extraction Method	PCEX24	PCEX24
Date of Extraction	7/3/86	7/3/86
Sample Amount Extracted (g)	1.575	1.024
Extract Final Volume (ml)	10.0	10.0
Extract Dilution	none	none
ESC Extract or Sample Number	WS-1559	WS-1562
Date of CGC/MS Analysis	5/12/86	5/12/86
MS FRN	>L1222	>L1225

Footnotes:

(a) LOQ - Limit of Quantitation in ug/g. Signal-to-noise ratio is greater than 10 when this amount is analyzed using the above referenced Analytical and Extraction Methods. The following letters indicate how this value was determined:
N - measured from native spiking of sample matrix.

(b) NQ - Not Quantitatable. The congener concentration is below the LOQ value.

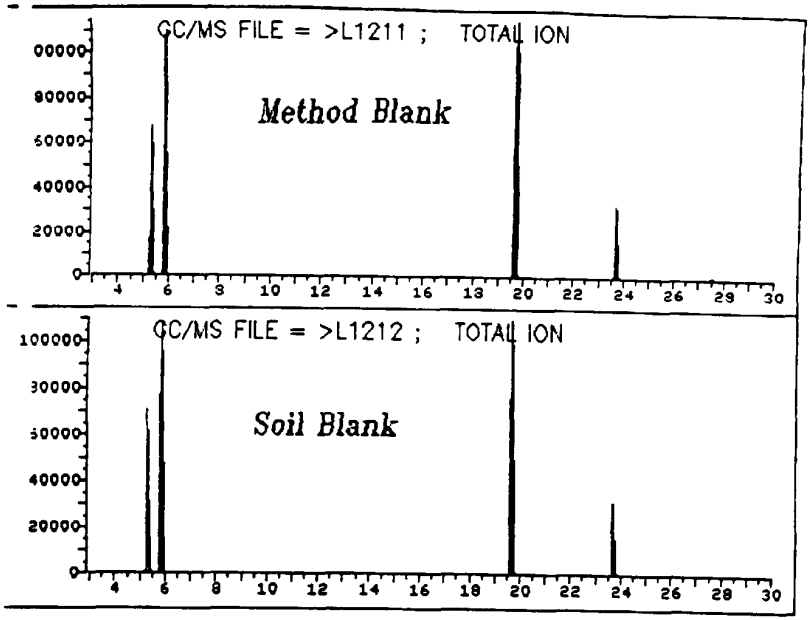
(c) Concentrations calculated assuming the total ion response of organic compounds to be equal to the total ion response of anthracene-d10 internal standard.

Table stored in file: F22318

Analyst(s): B. Mason Hughes and David McKenzie

Analyst Approval: B. Mason Hughes

MONS 024172



igure 1. Total Ion Chromatogram Obtained from the Capillary GC/MS Analysis of Method Blank and Soil Blank Extracts.

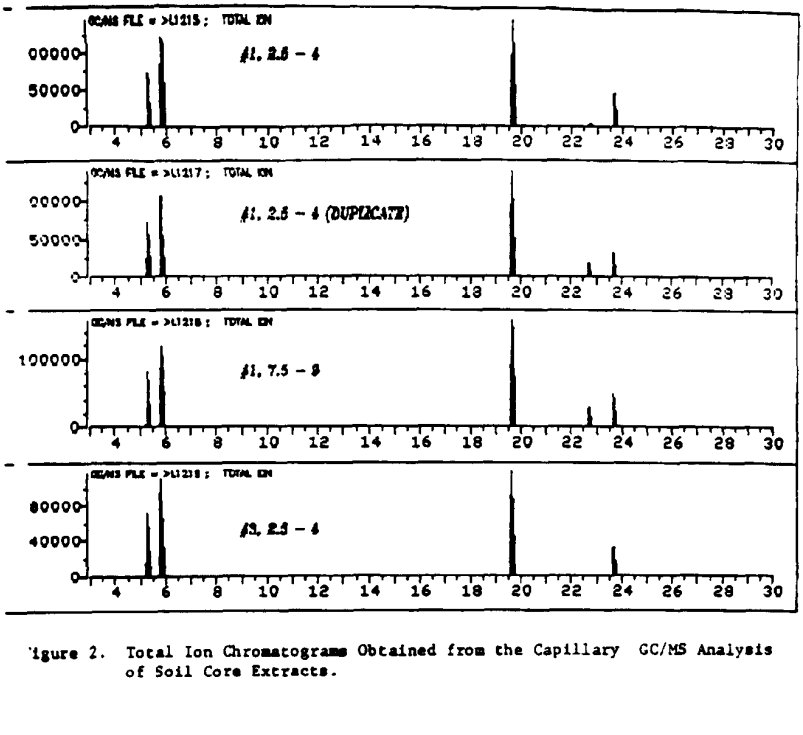
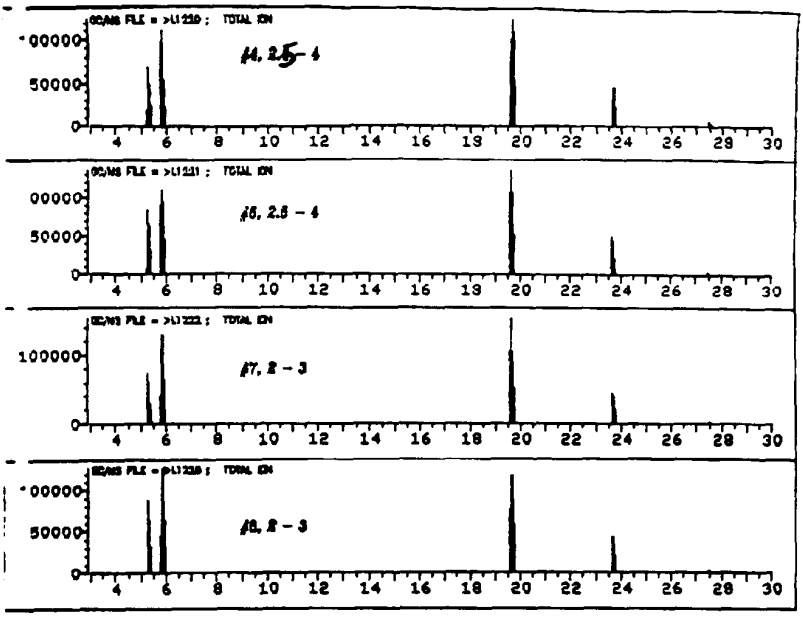


Figure 2. Total Ion Chromatograms Obtained from the Capillary GC/MS Analysis of Soil Core Extracts.



.igure 3. Total Ion Chromatograms Obtained from the Capillary GC/MS Analysis of Soil Core Extracts.

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

ESC Standard Extraction Method (SEM)
TITLE: PCBs and Other Compounds in Soils, Oils, and Solvents

ESC Standard Extraction Method No: PCEX24

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Soils and other non-EC active solids,solvents and oils.	0.5 - 5.0	10

TYPICAL ANALYTES: Aroclors 1232, 1242, 1248, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers, other compounds priority pollutants.

ESC Standard Analysis Method Nos: ECAN01, ECAN02, MSAN01,MSAN02, MSAN03,

---

INTERFERENCES:

GC/EC Analysis

Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers which are not removed by acid or base washes or the three phase silica gel column.

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

GC/MS Analysis

Various; See individual interferences for individual analytes.

Individual deuterated compounds	30 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson Solvents.
Sodium sulfate for drying the sample matrix.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Nitrogen for extract volume reduction.
Disposable glassware.
Acids and Bases such as: H_2SO_4, HCl, KOH, and NaOH.
(See flow sheet for this extraction method in PCFS24.)

LIMITATIONS: Method applies only to matrices which do not contain interferring electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region when electron capture analytical methods are used. This limitation does not apply to mass spectrometric methods.

PROCEDURE: Method requires that the sample be weighed into a vial, spiked with surrogate spiking compounds, and ultrasonically extracted. The solvent choice is left to the analyst's discretion, along with the number of times the sample is extracted. The extract may be washed with acid and/or base (analyst's discretion), and placed through a multiphase cleanup column (analyst's discretion). The extract may be split into different fractions which can be treated in a manner conducive to the analysis of analyte(s) of choice. This method should only be used by personal familiar with the Fundamentals of Extraction.

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

DATES OF METHOD CREATION AND UPDATES: 5/21/86

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

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

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

EXTRACTION DATE: ----------DATE COMPLETED: ----------

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

Weigh sample into a 14 or 40 mL vial [ ]
Sample weight ---------- g

Spike sample with surrogate [ ]
Surrogate spiking Solution ID./Amount

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [ ]
Extraction solvent:
Amount mL/extraction:
Time extracted ---------- minutes
Number of times extracted:

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to ---------- mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right. Record information at right. ( )
Acid wash:
Acid used---------, Concentration---%
number of times washed-----
Base wash:
Base used---------, Concentration---%
Number of times washed-----

[ ] Pre Rinse column with 30 mL hexane

Place ----- mL extract on cleanup column at right (optional) ( )
25 mL column

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

|-| 1 cm sodium sulfate
|-| 1 cm silica gel
| | 4 cm potasium permangante silica gel
|-|
|-| 1 cm silica gel
| | 4 cm base silica gel
|-|
|-| 1 cm silica gel
| | 1 cm acid silica gel
\x/ glass wool plug

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [ ]

FINAL VOLUME ----------

OTHER COMMENTS:

EXTRACT NO: ----------JOB NUMBER:----------ANALYST:----------

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 interfarences. Exact retention times of most likely PCB isomers can be obtained from the analysis of Aroclor 1248, Aroclor 1254 or Aroclor 1260 formulations.

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

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

---

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

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

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

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

DATE OF METHOD CREATION: 10/25/83, 2/3/84 Time Stamp - <860630.1036>

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: ----------

MONS 024180

ESC Standard Analysis Method (SAM) **ESC Standard Analysis Method No:**
TITLE: Organic Compounds in Solvents **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

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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 - <860630.1036>

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. .
.
[ ] GALCULATE 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: ----------

MQNS 024182

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

Recovery Information:

Time Stamp - <860623.0821>

Table 1. Raw data from the GC/MS analysis of .....

Project No: Date:

GC/MS File Namr	Sample ID	Area	Area	Area	Area	Area
>L1210	Spk. Std	100	100	100	100	
		95	92	92	94	
>L1211	WS-1549	103	107	106	97	
	method Blank	98	98	100	92	
>L1212	WS-1550	108	112	111	86	
	Blank Spike	103	103	105	81	
>L1213	WS-1551	111	115	117	117	
~~>L1214~~	Low PCB Spk.	106	106	110	110	
>L1214	WS-1552	103	110	116	108	
	high PCB Spk	98	101	109	102	
>L1215	WS-1553	104	110	112	114	
	#1, 2½-4	99	101	106	103	

BLTB14

Time Stamp - <860623.0821>

Table 1. Raw data from the GC/MS analysis of .....

Project No: ..... Date:

GC/MS File Numr	Sample ID	Area	Area	Area	Area	Area
>L1216	Sim Synth.	109	118	113	111	
		104	108	107	105	
>L1217	WS-1554	99	105	106	89	
	#1, 2½-4 (duplicate)	94	96	100	84	
>L1218	WS-1555	98	101	100	106	
	#1, 7½-9	93	93	94	100	
>L1219	WS-1556	108	119	119	91	
	#3, 2½-4	103	109	112	86	
>L1220	WS-1557	89	92	94	111	
	#4, 2½-4	84	84	89	105	
>L1221	WS-1558	102	107	106	117	
	#5, 2½-4	97	98	100	110	

BLTB14

Time Stamp - <860623.0821>

Table 1. Raw data from the GC/MS analysis of .....

Project No: Date:

GC/MS File Name	Sample ID	IS Area	Area	Area	Area	Area
>L1222	WS-1559	100	103	106	99	
	#7, 2-3	95	94	100	93	
>L1223	WS-1560	77	84	80	117	
	#7, 2-3 - low PCB Spk	73	77	76	110	
>L1224	WS-1561	101	111	116	119	
	#7, 2-3 - high PCB Spk	96	102	109	112	
>L1225	WS-1562	110	114	116	104	
	#8, 2-3	105	105	109	98	
>L1226	Surr Spk	105	108	106	106	
		100	99	100	100	
Surr Spk: Avr		105	109	106	106	

BLTB14

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

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: Method Blank — MRC EXTRACT #: WS-1549
LOG NO: NA.
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: NA.

Weigh sample into a 14 or 40 mL vial [x]
Sample weight 0.0000 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See Comments
Base wash:
Base used---------, Concentration---%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane
25 mL column
1 cm sodium sulfate
1 cm silica gel
4 cm potasium permangante silica gel
1 cm silica gel
4 cm base silica gel
1 cm silica gel
1 cm acid silica gel
glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1549 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

MONS 024188

FLOW SHEET PCFS24
FOR FSC EXTRACTION METHOD PCEX24

SAMPLE ID: Blank Soil ---------- MRC EXTRACT #: WS-1550
NA.
---------- LOG NO: ----------
EXTRACTION DATE: 5/14/86 ----------DATE COMPLETED: 5/16/86 ----------
SAMPLE COLOR AND DESCRIPTION Brown Soil ----------

Weigh sample into a 14 or 40 mL vial [x]

Sample weight 1.272 g

Spike sample with surrogate [x]

Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. {x}

Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )

Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)

Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See Comments
Base wash:
Base used---------, Concentration---%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )

[ ] Pre Rinse column with 30 mL hexane
25 mL column
| |
|-| 1 cm sodium sulfate
|-| 1 cm silica gel
| | 4 cm potasium permangante silica gel
|-|
|-| 1 cm silica gel
| | 4 cm base silica gel
|-|
|-| 1 cm silica gel
| | 1 cm acid silica gel
\x/ glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume (x)

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

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1550 ----------JOB NUMBER: 760.28-6223 ----------ANALYST: D. McKenzie ----------

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: Blank Soil + low level PCB spike — MRC EXTRACT #: WS-1551
LOG NO: NA.
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Brown Soil

Weigh sample into a 14 or 40 mL vial	[x]	Sample weight 1.218 g
Spike sample with surrogate	[x]	Surrogate spiking Solution ID./Amount 1.0 mL DEM-050285-01 (500 ug/mL) 100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL) 100 uL DEM-010385-03 (100 ug/mL)
Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once.	[x]	Extraction solvent: methylene chloride Amount mL/extraction: 9.0 mL Time extracted 30 minutes Number of times extracted: 1
Adjust volume of extract by nitrogen stream (optional)	( )	Extract volume adjusted to (no adjust) mL
Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right.	(x)	Acid wash: Acid used-H2SO4---, Concentration 75% number of times washed--1-- See Comments Base wash: Base used--KOH----, Concentration-50% Number of times washed--1-- See Comments
Place ~1.0- mL extract on cleanup column at right (optional)	(x)	[x] Pre Rinse column with 30 mL hexane 25 mL column
Elute column with 40 mL hexane or pentane, collect in a centrifuge tube	[x]	
Reduce volume with nitrogen stream to 1.0 mL	[x]	
Record extract final volume	[x]	

FINAL VOLUME 10 mL

	1 cm sodium sulfate
	1 cm silica gel
	4 cm potasium permangante silica gel
	1 cm silica gel
	4 cm base silica gel
	1 cm silica gel
	1 cm acid silica gel
	glass wool plug

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash. Later 1 mL of the original extract was placed through a column as quality control for other samples, and still later the original extract was subjected to 3 acid washes one, overnight, and one base wash with no column for PCB analysis.

EXTRACT NO: WS-1551 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

FLOW SHEET PCFS24
FOR ESG EXTRACTION METHOD PCEX24

SAMPLE ID: Blank Soil + High level PCB spike — MRC EXTRACT #: WS-1552
LOG NO: NA.
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Brown Soil

Weigh sample into a 14 or 40 mL vial	[x]	Sample weight 1.322 g
Spike sample with surrogate	[x]	Surrogate spiking Solution ID./Amount 1.0 mL DEM-050285-01 (500 ug/mL) 100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL) 100 uL DEM-010385-03 (1000 ug/mL)
Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once.	[x]	Extraction solvent: methylene chloride Amount mL/extraction: 9.0 mL Time extracted 30 minutes Number of times extracted: 1
Adjust volume of extract by nitrogen stream (optional)	( )	Extract volume adjusted to (no adjust) mL
Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right. Record information at right.	(x)	Acid wash: Acid used-H2SO4---, Concentration 75% number of times washed--1-- See Comments Base wash: Base used--KOH----, Concentration-50% Number of times washed--1-- See Comments
Place ~1.0- mL extract on cleanup column at right (optional)	(x)	[x] Pre Rinse column with 30 mL hexane 25 mL column
Elute column with 40 mL hexane or pentane, collect in a centrifuge tube	[x]	1 cm sodium sulfate 1 cm silica gel 4 cm potasium permangante silica gel 1 cm silica gel 4 cm base silica gel
Reduce volume with nitrogen stream to 1.0 mL	[x]	1 cm silica gel 1 cm acid silica gel
Record extract final volume	(x)	\x/ glass wool plug

FINAL VOLUME 10 mL

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash. Later 1 mL of the original extract was placed through a column as quality control for other samples, and still later the original extract was subjected to 3 acid washes one, overnight, and one base wash with no column for PCB analysis.

EXTRACT NO: WS-1552 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #1, 2 1/2 - 4 ---------- MRC EXTRACT # WS-1553 U-86-05-05-02
LOG NO: ----------
EXTRACTION DATE: 5/14/86 ----------DATE COMPLETED: 5/16/86 ----------
SAMPLE COLOR AND DESCRIPTION Black and grey clay ----------

Weigh sample into a 14 or 40 mL vial [x]
Sample weight 1.062 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid usad-H2SO4---, Concantration 75%
number of times washed--1-- See Comments
Base wash:
Base used---------, Concentration-50%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane
25 mL column
1 cm sodium sulfate
1 cm silica gel
4 cm potasium permangante silica gel
1 cm silica gel
4 cm basa silica gel
1 cm silica gel
1 cm acid silica gel
\x/ glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

OTHER COMMENTS: Tha extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1553 ----------JOB NUMBER: 760.28-6223 ----------ANALYST: D. McKenzie ----------

MONS 024192

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #1, 2 1/2 - 4 (duplicate) — MRC EXTRACT #: WS-1554
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Black and grey clay

Weigh sample into a 14 or 40 mL vial [x] — Sample weight 1.184 g

Spike sample with surrogate [x] — Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right. Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1-- See Comments
Base wash:
Base used--KOH----, Concentration-50%
Number of times washed--1-- See Comments

Place ~1.0~ mL extract on cleanup column at right (optional) (x)
[x] Pre Rinse column with 30 mL hexane
25 mL column
1 cm sodium sulfate
1 cm silica gel
4 cm potasium permangance silica gel
1 cm silica gel
4 cm base silica gel
1 cm silica gel
1 cm acid silica gel
glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [x]

Reduce volume with nitrogen stream to ~1.0~ mL [x]

Record extract final volume [x]

FINAL VOLUME 10 mL

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash. Later 1 mL of the original extract was placed through a column as quality control for other samples, and still later the original extract was subjected to 3 acid washes one, overnight, and one base wash with no column for PCB analysis.

EXTRACT NO: WS-1554 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #1, 7 1/2 - 9 — MRC EXTRACT #: WS-1555
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Black and grey mud

Weigh sample into a 14 or 40 mL vial [x]
Sample weight 1.243 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right. Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1-- See Comments
Base wash:
Base used--KOH----, Concentration-50%
Number of times washed--1-- See Comments

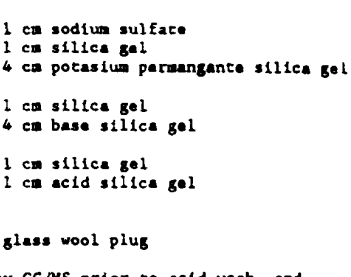
Place ~1.0- mL extract on cleanup column at right (optional) (x)
[x] Pre Rinse column with 30 mL hexane
25 mL column

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [x]

Reduce volume with nitrogen stream to 1.0 mL [x]

Record extract final volume [x]

FINAL VOLUME 10 mL



OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash. Later 1 mL of the original extract was placed through a column as quality control for other samples, and still later the original extract was subjected to 3 acid washes one, overnight, and one base wash with no column for PCB analysis.

EXTRACT NO: WS-1555 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #3, 2 1/2 - 4 — MRC EXTRACT #: WS-1556
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Strong smelling grey and purple mud

Weigh sample into a 14 or 40 mL vial [x]
Sample weight 1.654 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See comments
Base wash:
Base used---------, Concentration---%
Number of times washed-----

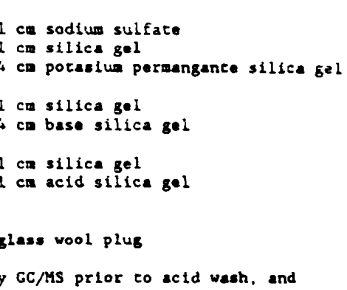
Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane
25 mL column

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL



OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1556 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

MONS 024195

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #4, 2 1/2 - 4 — MRC EXTRACT #: WS-1557
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Crushed bricks and soils

Weigh sample into a 14 or 40 mL vial [x] — Sample weight 1.396 g

Spike sample with surrogate [x] — Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See comments
Base wash:
Base used---------, Concentration---4
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane
25 mL column

Eluta column with 40 mL hexane or pentane, collect in a centrifuge tuba [ ]

Reduce volume with nitrogen stream to mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

	1 cm sodium sulfate
	1 cm silica gel
	4 cm potasium permangante silica gel
	1 cm silica gel
	4 cm base silica gel
	1 cm silica gel
	1 cm acid silica gel
	glass wool plug

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1557 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

MONS 024196

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #5, 2 1/2 - 4 ---------- MRC EXTRACT #: WS-1558
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 ---------- DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Brown and Black clay

Weigh sample into a 14 or 40 mL vial [x]
Sample weight 1.171 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See Comments
Base wash:
Base used---------, Concentration---%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane
25 mL column

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

| |
|-| 1 cm sodium sulfate
|-| 1 cm silica gel
| | 4 cm potasium permangante silica gel
|-|
|-| 1 cm silica gel
| | 4 cm base silica gel
|-|
|-| 1 cm silica gel
| | 1 cm acid silica gel
\x/ glass wool plug

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1558 ---------- JOB NUMBER: 760.28-6223 ---------- ANALYST: D. McKenzie

MONS 024197

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #7, 2- 3 — MRC EXTRACT #: WS-1559
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Brown and grey and yellow clay

Weigh sample into a 14 or 40 mL vial [x] — Sample weight 1.575 g

Spike sample with surrogate [x] — Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See comments
Base wash:
Base used---------, Concentration----%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

25 mL column
| |
|-| 1 cm sodium sulfate
|-| 1 cm silica gel
| | 4 cm potasium permangante silica gel
|-|
|-| 1 cm silica gel
| | 4 cm base silica gel
|-|
|-| 1 cm silica gel
| | 1 cm acid silica gel
\x/ glass wool plug

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1559 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #7, 2- 3 plus low level PCB spike — MRC EXTRACT #: WS-1560 U-86-05-05-02

LOG NO:

EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86

SAMPLE COLOR AND DESCRIPTION: Brown and grey and yellow clay

Weigh sample into a 14 or 40 mL vial [x] — Sample weight 1.025 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug mL
100 uL DEM-010385-03 (100 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See Comments
Base wash:
Base used---------, Concentration---%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexene
25 mL column
1 cm sodium sulfate
1 cm silica gel
4 cm potasium permanganate silica gel
1 cm silica gel
4 cm base silica gel
1 cm silica gel
1 cm acid silica gel
glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1560 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

MONS 024199

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #7, 2- 3 plus high level PCB spike — MRC EXTRACT #: WS-1561 U-86-05-05-02 — LOG NO:

EXTRACTION DATE: 5/14/86 — DATE COMPLETED: 5/16/86

SAMPLE COLOR AND DESCRIPTION: Brown and grey and yellow clay

Weigh sample into a 14 or 40 mL vial [x] — Sample weight 1.019 g

Spike sample with surrogate [x] — Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)
100 uL DEM-010385-03 (1000 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) [ ] — Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. [x]
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See comments
Base wash:
Base used---------, Concentration---%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) [ ]
[ ] Pre Rinse column with 30 mL hexane
25 mL column
| |
|-| 1 cm sodium sulfate
|-| 1 cm silica gel
| | 4 cm potasium permangante silica gel
|-|
|-| 1 cm silica gel
| | 4 cm base silica gel
|-|
|-| 1 cm silica gel
| | 1 cm acid silica gel
\x/ glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1561 — JOB NUMBER: 760.28-6223 — ANALYST: D. McKenzie

FLOW SHEET PCFS24
FOR ESC EXTRACTION METHOD PCEX24

SAMPLE ID: #8, 2 - 3 ---------- MRC EXTRACT # WS-1562
LOG NO: U-86-05-05-02
EXTRACTION DATE: 5/14/86 ---------- DATE COMPLETED: 5/16/86
SAMPLE COLOR AND DESCRIPTION: Black soil with crushed bricks

Weigh sample into a 14 or 40 mL vial [x]
Sample weight 1.024 g

Spike sample with surrogate [x]
Surrogate spiking Solution ID./Amount
1.0 mL DEM-050285-01 (500 ug/mL)
100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)

Extract sample in an ultrasound bath. Record solvent used, amount, time extracted, and number of times extracted. Pool extracts if extracted more than once. [x]
Extraction solvent: methylene chloride
Amount mL/extraction: 9.0 mL
Time extracted 30 minutes
Number of times extracted: 1

Adjust volume of extract by nitrogen stream (optional) ( )
Extract volume adjusted to (no adjust) mL

Remove 2-3 mL extract and perform acid and base washes (optional) Indicate at right Record information at right. (x)
Acid wash:
Acid used-H2SO4---, Concentration 75%
number of times washed--1--See comments
Base wash:
Base used---------, Concentration----%
Number of times washed-----

Place ----- mL extract on cleanup column at right (optional) ( )
[ ] Pre Rinse column with 30 mL hexane
25 mL column
1 cm sodium sulfate
1 cm silica gel
4 cm potasium permangante silica gel
1 cm silica gel
4 cm base silica gel
1 cm silica gel
1 cm acid silica gel
glass wool plug

Elute column with 40 mL hexane or pentane, collect in a centrifuge tube [ ]

Reduce volume with nitrogen stream to ------ mL [ ]

Record extract final volume [x]

FINAL VOLUME 10 mL

OTHER COMMENTS: The extract was analyzed by GC/MS prior to acid wash, and by GC/EC after acid wash.

EXTRACT NO: WS-1562 ---------- JOB NUMBER: 760.28-6223 ---------- ANALYST: D. McKenzie

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

SOIL ANALYSIS STRATEGY

BACKGROUND

The "W" Building Complex has been scheduled for dismantling during June and July of 1986. The building itself was a large, multi-product facility that had been manufacturing various organic chemicals from the 1930's until 1982. In order to evaluate the extent of potential subsurface contamination in and around the building, a series of soil borings were taken. The analysis results of these samples will provide us with data that will dictate the extent of "clean-up" at this location.

Whatever clean-up activities occur at this site, they will be a totally voluntary effort. No outside agency is dictating any remedial action at this location.

CORE SAMPLING SUMMARY

A total of nine borings were taken in and around the "W" Building. Access to all locations was a limiting factor; the attachment shows the exact location of each soil boring.

ANALYSIS NEEDED

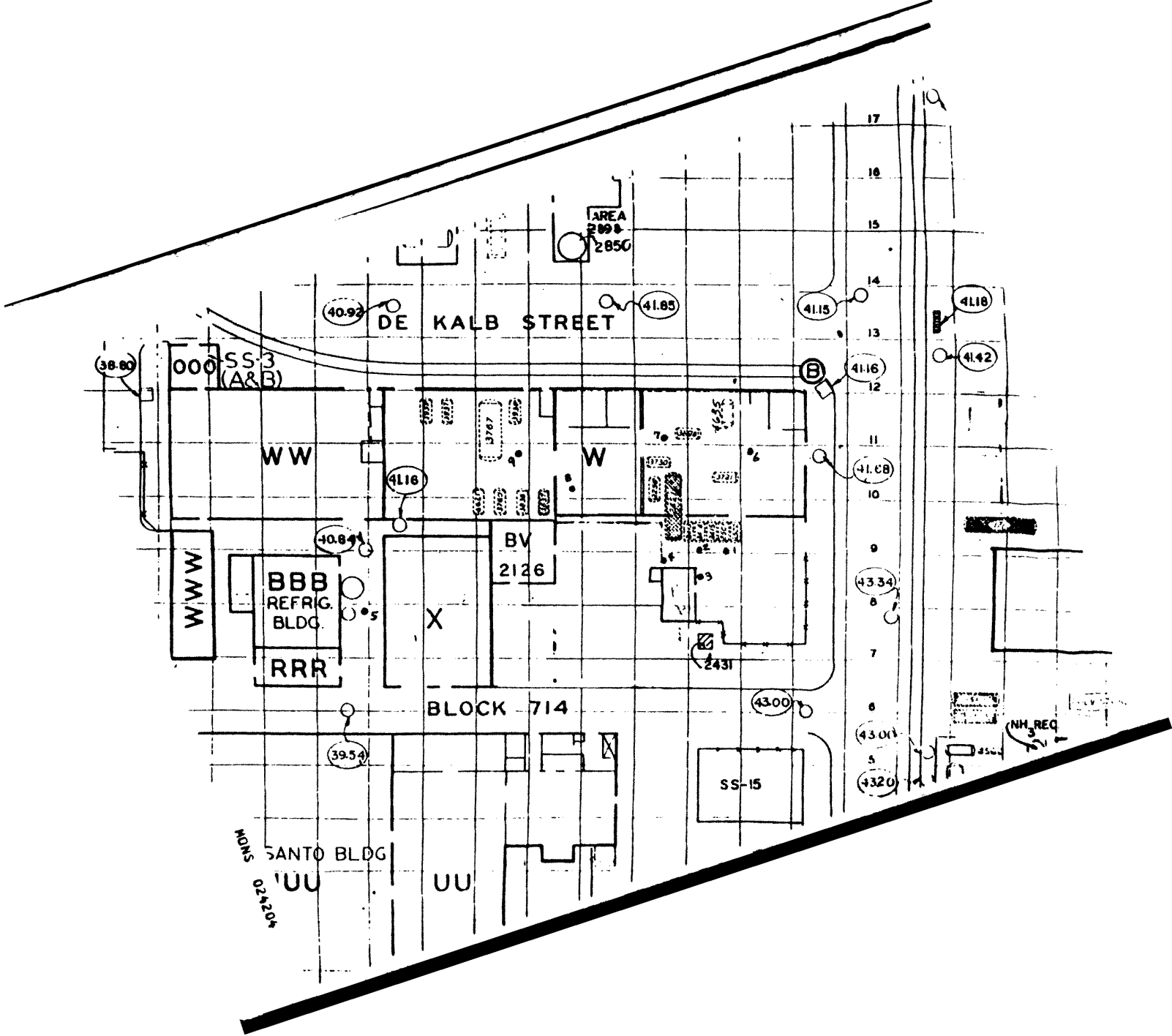
The soil could likely contain a host of organic compounds; however, exact identification and quantification of each is not necessary. Instead a gross evaluation of the samples using TOC analysis followed by GC/MS on selected samples (those greater than 1000 ppm). PCB analysis should also be performed on all samples.

PCB ANALYSIS - all samples

TOC ANALYSIS — ≥ 100 ppm - selected GC/MS above 1000 ppm

TOC ANALYSIS — <100 ppm

MONS 024203


DE KALB STREET
AREA
2898
2850
40.92
41.85
41.15
41.18
41.42
41.16
41.68
38.80
SS-3
(A&B)
WW
W
WWW
BBB
REFRIG.
BLDG.
RRR
X
BV
2126
2431
43.34
43.00
43.00
43.20
BLOCK 714
39.54
SS-15
NH3 REQ
SANTO BLDG
UU
UU
MONS
024204

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

Time Stamp - <860804.1501>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 5-5-86 LOG NO: U-86-05-05-02

REQUESTER: QUEENY/KEN PERICA PROJECT NO: 760.28-6223

LOGIN NOTEBOOK PAGE: 21

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

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

SAMPLE PICKUP BY: MCKENZIE

SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 22 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
#8, 2-3	1 x 1 qt	WS-1562
#8, 3-4	1 x 1 qt	
#2, 0-2 1/2	1 x 1 qt	
#1, 0-2 1/2	1 x 1 qt	
#8, 2-3	1 x 1 qt	
#5, 0-2 1/2	1 x 1 qt	
#9, 3-4	1 x 1 qt	
#3, 0-2 1/2	1 x 1 qt	
#2, 2 1/2-4	1 x 1 qt	
#1, 2 1/2-4	1 x 1 qt	WS-1553, WS-1554-DUP
#7, 2-3	1 x 1 qt	WS-1559, WS-1560-LS, WS-1561-HS
#5, 2 1/2-4	1 x 1 qt	WS-1558
#4, 2 1/2-4	1 x 1 qt	WS-1557
#3, 2 1/2-4	1 x 1 qt	WS-1556
#2, 5-6 1/2	1 x 1 qt	
#1, 5-6 1/2	1 x 1 qt	
#6, 1-2	1 x 1 qt	
#5, 5-6 1/2	1 x 1 qt	
#4, 5-6 1/2	1 x 1 qt	
#3, 5-6 1/2	1 x 1 qt	
#2, 7 1/2-9	1 x 1 qt	
#1, 7 1/2-9	1 x 1 qt	WS-1555

NOTE(s): 1. DUP refers to a duplicate analysis, LS refers to a low level native spike, and HS refers to a high level native spike.
2. See flow sheets for notes on additional workup done on samples WS-1551, WS-1552, WS-1554, and WS-1555.

RECORDED BY: McKenzie

REQUIRED ANALYSES: PCBs and Wide Scan on select samples

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158609

ANALYTICAL TECHNIQUES:

	DATE	ESC STANDARD	INSTRUMENT

TECHNIQUE (EN)	COMPLETED	ANALYSIS METHOD	LOGBOOK PAGE
x CCGC/MS (18)	8/4/86	MSAN02	No. 157,127
x GC/EC (18)	8/4/86	ECAN02	No. 157,260

FINAL REPORT NOTEBOOK PAGE: 3,434,411

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: AA223:LI

The following sections describe important terminology used in Quality Assurance/Quality Control, Analyte Extraction and Instrumental Analysis. ESC and ESC/UAG Good Laboratory Practices Manuals can be obtained by contacting Bill 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 unduely burden analytical studies with large development and method validation costs. This approach has been taken so that sets of samples can be analyzed for various analytes without extensively validating methods over wide concentration ranges and without using methods which are designed for only one matrix.

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

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

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

(continued)

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

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

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

(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 conveniance, an LOQ will be determined using other methods. These methods should be clearly described in this report.

Further Information

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

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

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

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

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

QAQCTE:MH

Analyte Extraction Terminology

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

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

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

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

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

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

(continued)

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 ussd 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.

(continued)

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 addsd to the sample extract prior to instrumental analysis. Responses of all analytes are calculated relative to the internal standard using the following relationship:

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

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

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

(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:

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

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

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

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

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

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

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

(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