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REPORT NO.: MDA-649

JOB/PROJECT NO.: 317

DATE: 8 August 1985

PERIOD COVERED:

TITLE: ANALYSIS OF SAMPLES FOR INCIDENTAL PCB's

AUTHORS: B. M. Hughes

ABSTRACT: Various samples from the Krummrich Plant were analyzed for possible concentration of PCB's. The analyses performed and results of these analyses are shown in this report.

TECHNICAL APPROVAL: P L Sherman 8/8/85
P. L. Sherman
Sr. Research Group Leader

APPROVED BY: R. F. Ivory
R. F. Ivory
Environmental Sciences Center
Service Project Manager

APPROVAL DATE: 8-8-85

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REPORT NO.: MDA-649 AUTHORS: B. M. Hughes
COPY NO.: TITLE: Analysis of Samples for Incidental PCB's.

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ABSTRACT ONLY

H. A. Woltermann - U4E

Analysis:

B. M. Hughes
D. McKenzie
B. Williams

MONS 023639

ANALYSIS REPORT

DATE: August 7, 1985

LOG NUMBER: 1-85-03-29-01 PROJECT NO: 760.18-317

TO: Krumprich/Ivory

FROM:

Environmental Sciences Center
Monsanto Company/Dayton Laboratory
1515 Nicholas Road
Dayton, Ohio 45418

Phone No: (513) 268-3411

Title: Analysis of Incidental PCBs in Various Samples

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

Standard Extraction Method No(s): PCEX15, PCEX21

Standard Analysis Method No(s): MSAN03

Final Report File No(s): A31718

Table File No(s): B31718, C31718, D31718, E31718, F31718, G31718

Eleven samples were received for Incidental PCB analysis. Eight still rubble samples, one Therminol 60 sample, one NODCB waste solids sample, and one ODCB waste solids and water sample were received. The Therminol 60 sample contained no detectable PCBs with a limit of detection of 5 ug/g. The NODCB waste solid sample contained no detectable PCBs with a limit of detection of 1 ug/g. A summary of the NODCB results is given in Table 1. Tables 2 through 5 summarize results of PCB analyses of the still rubble samples. Table 6 summarizes the results of PCB analysis in the ODCB waste solids and water sample.

Capillary GC/MS chromatograms from sample analysis and QA/QC experiments are shown in later sections of this report. The Analytical Request Information shows extract identifications used by ESC/UAG for all experiments conducted. Sample dilution information for the Therminol 60 analysis is also shown in the Analytical Request information.

Values for PCB congeners measured in duplicate analyses in Tables 2, 4, and 6 do not represent normal sample replicability. The large variation in these values is due to the vary inhomogeneous nature of the samples. The still rubble samples contained a wide variety of rocks and powder. The ODCB waste water and solid sample contained water and solids. No truly representative sample aliquots could be obtained from these samples without completely homogenizing the samples, which was inpractical.

Included in this report are:

1) An explanation of the tables generated.
2) Capillary GC/MS chromatograms obtained from sample analysis.
3) Capillary GC/MS chromatograms obtained from the analysis of various QA/QC samples.
4) Descriptions of Standard Extraction and Analysis Methods used.
5) Analytical Request Information Generated for this project.
6) A copy of the ESC/UAG Good Laboratory Practices Manual.

ANALYST(S): B. Mason Hughes, David McKenzie and Barbara Williams

ANALYST APPROVAL:

Table 1. Results of Incidental PCB Analyses.

Project No: 760.18-317 Date: August 7, 1985

Congener Class	Analytical Method	LOQ(a) (ug/g)	Sample Identification: NODCB Waste Solids	NODCB Waste Solids (dup)	Method Blank
			Concentration (ug/g)		
CL1-BP	MSAN03	1(K)	NQ(b)	NQ	NQ
CL2-BP	"	"	"	"	"
CL3-BP	"	"	"	"	"
CL4-BP	"	"	"	"	"
CL5-BP	"	"	"	"	"
CL6-BP	"	"	"	"	"
CL7-BP	"	"	"	"	"
CL8-BP	"	"	"	"	"
CL9-BP	"	"	"	"	"
CL10-BP	"	"	"	"	"
Surrogate Spiking Compound		Amount Added(ug/g)	Recovery (%)		
CL5-Centere		1.0	215	134	155
2,4,6-CL3-BP		1.0	71	60	114
Documentation					
ESC Extraction Method			PCEX15	PCEX15	PCEX15
Date of Extraction			4/8/85	4/8/85	4/8/85
Sample Amount Extracted (g)			1.2141	1.3421	0
Extract Final Volume (ml)			1.0	1.0	1.0
Extract Dilution			none	none	none
ESC Extract or Sample Number			PCB-1259	PCB-12e0	PCB-12e3
Date of GC/MS Analysis			5/10/85	5/10/85	5/10/85
MS FAN			>L0049	>L0071	>L0074

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:
- E - estimated from PCB congener standard analyzed under the same conditions.
- M - measured from native spiking of sample matrix.
- S - estimated from surrogate spiking of sample matrix.
- V - measured in validation experiments of similar sample matrix.

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

Table stored in file: B31718:MH:59

Analyst(s): B. Mason-Hughes, David McKenzie and Barbara Williams

Analyst Approval: B. Mason-Hughes

MONS 023642

Table 2. Results of Incidental PCB Analyses.

Project No: 760.10-317 Date: August 7, 1985

Congener Class	Analytical Method	LOQ(a) (ug/g)	Sample Identification: Still Rubble 0292-5	Still Rubble 0292-5 (dup)	Still Rubble 0292-7
			Concentration (ug/g)		
CL1-BP	HSAN03	1(E)	2.2	10.8	5.3
CL2-BP	"	"	3.9	4.8	NQ(b)
CL3-BP	"	"	NQ	NQ	"
CL4-BP	"	"	"	"	"
CL5-BP	"	"	"	"	"
CL6-BP	"	"	"	"	"
CL7-BP	"	"	"	"	"
CL8-BP	"	"	"	"	"
CL9-BP	"	"	"	"	"
CL10-BP	"	"	"	"	"
Surrogate Spiking Compound	Amount Added(ug/g)		Recovery (%)		
2,4,6-CL3-BP	1.0		87	85	78
Documentation					
ESC Extraction Method			PCEX21	PCEX21	PCEX21
Date of Extraction			4/8/85	4/8/85	4/8/85
Sample Amount Extracted (g)			1.0326	1.2573	1.0064
Extract Final Volume (ml)			1.0	1.0	1.0
Extract Dilution			none	none	none
ESC Extract or Sample Number			PCB-1264	PCB-1265	PCB-1266
Date of CGC/MS Analysis			5/10/85	5/10/85	5/10/85
MS FRN			>L0075	>L0076	>L0078

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:
E - estimated from PCB congener standard analyzed under the same conditions.
M - measured from matrix spiking of sample matrix.
S - estimated from surrogate spiking of sample matrix.
V - measured in validation experiments of similar sample matrix.

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

Table stored in file: C31710:MM:59

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

Analyst Approval: B. Mason Hughes

Table 3. Results of Incidental PCB Analyses.

Project No: 760.18-317 Date: August 7, 1985

	Sample Identification		
	Still Rubble 0292-3	Still Rubble 0292-1	Method Blank (Dayton Soil)
Congener Class / Analytical Method	LOQ(a) (ug/g)	Concentration (ug/g)	
CL1-BP / HSAN33	1(M)	NQ(b)	1.3	NQ(b)
CL2-BP	"	"	NQ	"
CL3-BP	"	"	"	"
CL4-BP	"	"	"	"
CL5-BP	"	"	"	"
CL6-BP	"	"	"	"
CL7-BP	"	"	"	"
CL8-BP	"	"	"	"
CL9-BP	"	"	"	"
CL10-BP	"	"	"	"
Surrogate Spiking Compound	Amount Added(ug/g)	Recovery (%)		
2,4,6-CL3-BP	1.0	75	97	94
Documentation				
ESC Extraction Method		PCEX21	PCEX21	PCEX21
Date of Extraction		4/8/85	4/8/85	4/8/85
Sample Amount Extracted (g)		1.2829	1.04c7	1.0811
Extract Final Volume (ml)		1.0	1.0	1.0
Extract Dilution		none	none	none
ESC Extract or Sample Number		PCB-1267	PCB-1270	PCB-1271
Date of GC/MS Analysis		5/18/85	5/18/85	5/18/85
MS FRN		>L0079	>L0082	>L0083

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:
- I - estimated from PCB congener standard analyzed under the same conditions.
- M - measured from native spiking of sample matrix.
- S - estimated from surrogate spiking of sample matrix.
- V - measured in validation experiments of similar sample matrix.

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

Table stored in file: D31718:MH:59

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

Analyst Approval: B. Mason Hughes

Table [illegible] Results of Incidental PCB Analyses:

Project No. 760.18-317 Date: August 7, 1985

			Sample Identification		
			Still Bubble 0292-2	Still Bubble 0292-6	Still Bubble 0292-6 (dup)
Congener Class	Analytical Method	LOQ(a) (ug/g)	Concentration (ug/g)		
CL1-BP	MSAN03	1(M)	0.90	1.0	16.9
CL2-BP	"	"	NQ(b)	2.4	15.8
CL3-BP	"	"	"	0.39	7.9
CL4-BP	"	"	2.8	2.0	5.2
CL5-BP	"	"	3.0	NQ	1.9
CL6-BP	"	"	4.1	"	3.3
CL7-BP	"	"	5.6	"	NQ
CL8-BP	"	"	1.3	"	"
CL9-BP	"	"	NQ	"	"
CL10-BP	"	"	"	"	"
Surrogate Spiking Compound	Amount Added(ug/g)		Recovery (%)		
2,4,6-CL3-BP	1.0	106	92		114
Documentation					
ESC Extraction Method			PCEX21	PCEX21	PCEX21
Date of Extraction			4/8/85	4/8/85	4/8/85
Sample Amount Extracted (g)			2.2127	1.5521	1.7737
Extract Final Volume (ml)			1.0	1.0	1.0
Extract Dilution			none	none	none
ESC Extract or Sample Number			PCB-1272	PCB-1273	PCB-1274
Date of CGC/MS Analysis			5/10/85	5/10/85	5/10/85
MS FRM			>L0085	>L0086	>L0087

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:
E - estimated from PCB congener standard analyzed under the same conditions.
M - measured from native spiking of sample matrix.
S - estimated from surrogate spiking of sample matrix.
V - measured in validation experiments of similar sample matrix.

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

Table stored in file: E31718:MM:59

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

Analyst Approval: B. Mason Hughes

Table B. Results of Incidental PCB Analyses.

Project No: 760.10-317 Date: August 7, 1985

Congener Class	Analytical Method	LOQ(a) (ug/g)	Sample Identification: Still Rubble 0292-8	Still Rubble 0292-4	Method Blank
			Concentration (ug/g)		
CL1-BP	MSAN03	1(M)	0.46	NQ(b)	NQ
CL2-BP	"	"	NQ	0.35	"
CL3-BP	"	"	"	0.19	"
CL4-BP	"	"	1.1	2.6	"
CL5-BP	"	"	1.4	2.9	"
CL6-BP	"	"	4.6	1.3	"
CL7-BP	"	"	4.4	NQ	"
CL8-BP	"	"	0.64	"	"
CL9-BP	"	"	NQ	"	"
CL10-BP	"	"	"	"	"
Surrogate Spiking Compound		Amount Added(ug/g)		Recovery (%)	
2,4,6-CL3-BP		1.0	75	111	89
Documentation					
ESC Extraction Method			PCEX21	PCEX21	PCEX21
Date of Extraction			4/8/85	4/8/85	4/8/85
Sample Amount Extracted (g)			2.1952	3.1313	0
Extract Final Volume (ml)			1.0	1.0	1.0
Extract Dilution			none	none	none
ESC Extract or Sample Number			PCB-1277	PCB-1278	PCB-1279
Date of GGC/MS Analysis			5/10/85	5/10/85	5/10/85
MS FRN			>L0090	>L0092	>L0093

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:
E - estimated from PCB congener standard analyzed under the same conditions.
M - measured from native spiking of sample matrix.
S - estimated from surrogate spiking of sample matrix.
V - measured in validation experiments of similar sample matrix.

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

Table stored in file: F31710:MH:59

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

Analyst Approval: B. Mason Hughes

Table 6: Results of Incidental PCB Analyses.

Project No: 760.1B-317 Date: August 7, 1985

			Sample Identification		
			Solids and Water 02931-2	Solids and Water 02931-2 (dup)	Method Blank
Congener Class	Analytical Method	LOQ(a) (ug/g)	Concentration (ug/g)		
CL1-BP	HSAM03	1(M)	12.7	3.0	MQ
CL2-BP	"	"	MQ(b)	MQ	"
CL3-BP	"	"	"	"	"
CL4-BP	"	"	"	"	"
CL5-BP	"	"	"	"	"
CL6-BP	"	"	"	"	"
CL7-BP	"	"	"	"	"
CL8-BP	"	"	"	"	"
CL9-BP	"	"	"	"	"
CL10-BP	"	"	"	"	"
Surrogate Spiking Compound		Amount Added:ug/g)	Recovery (%)		
2,4,6-CL3-BP		1.0	97	91	87
Documentation					
ESI Extraction Method			PCEX21	PCEX21	PCEX21
Date of Extraction			4/8/85	4/8/85	4/8/85
Sample Amount Extracted (g)			1.390	1.2820	0
Extract Final Volume (ml)			1.0	1.0	1.0
Extract Dilution			none	none	none
ESI Extract or Sample Number			PCB-1280	PCB-1281	PCB-1285
Date of GGC/MS Analysis			5/10/85	5/10/85	5/10/85
ES FBK			>LO094	>LO095	>LO100

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 reference Analytical and Extraction Methods. The following letters indicate how this value was determined:
E - estimated from PCB congener standard analyzed under the same conditions.
M - measured from matrix spiking of sample matrix.
S - estimated from surrogate spiking of sample matrix.
V - measured in validation experiments of similar sample matrix.

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

Table stored in file: 631718:HN:59

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

Analyst Approval: B. Mason Hughes

The Following Page is a Detailed Explanation of Tables Generated for this Project.

DETAILED EXPLANATION OF INCIDENTAL PCB TABLES

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

Each table includes a general title, ESC Project Number and Date on which the table was generated. The sample identification is the customer identification which was on the sample bottle. The next section of the table gives results for the particular PCB congener class listed. The level of quantitation (LOQ) using the Analytical and Extraction Methods shown in the table, is given for each congener class. This LOQ is determined in one of four ways and is described in the table footnotes.

The next section of the table deals with quality control used in each sample analysis. The surrogate spiking compound (if used) which is added to the sample before extraction and analysis, is listed, along with the amount added and % Recovery measured for each sample. The final section before the footnotes deals with how each sample was extracted and data documentation. The ESC Extraction Method, Date of Sample Extraction, Amount of Sample Extracted, Extract Final Volume, Extract Dilution and ESC Extract or Sample Number give critical extraction details of the analysis. The date of GC/MS analysis and the mass spectrometer file reference number (FRN) document how and when the extract or sample was analyzed and where the raw data is stored.

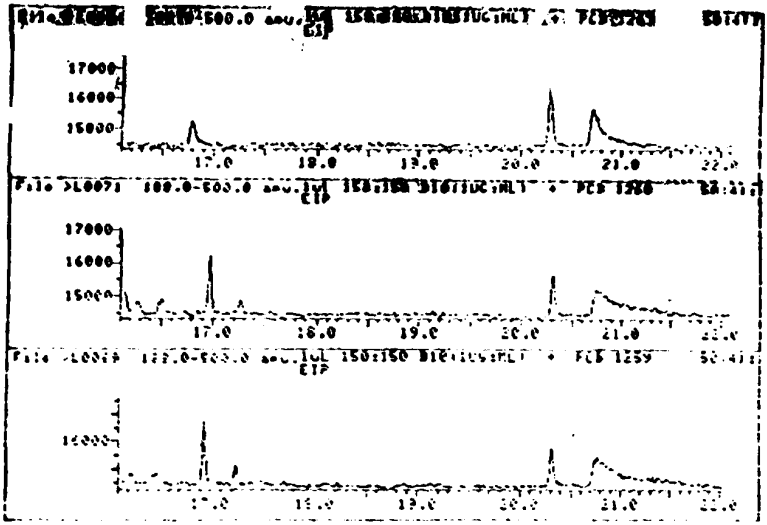
At the bottom of each table, analysts involved in extraction and analysis are listed, along with provisions for signature of the responsible analyst. This table format is both compact and precise. It was developed in an attempt to quickly and accurately generate data which the customer can quickly and accurately evaluate. It should include all required information for certification. If there are any suggestions concerning this format, please contact me at extensions 396, 209, or 409.

B. Mason Hughes
Senior Research Specialist

Environmental Sciences Center
Monsanto Company
Dayton Laboratory
1515 Nicholas Road
Dayton, Ohio 45407

File: INEXPT:MH:SP

The following pages show calculations or raw data documentation of reported results.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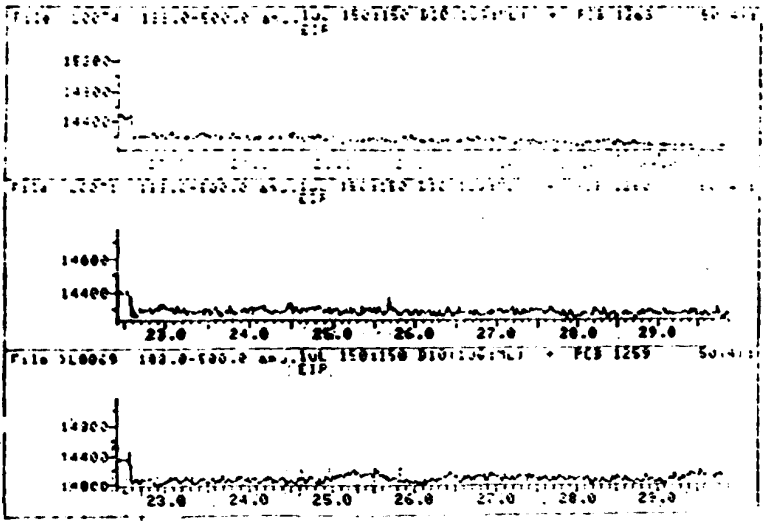
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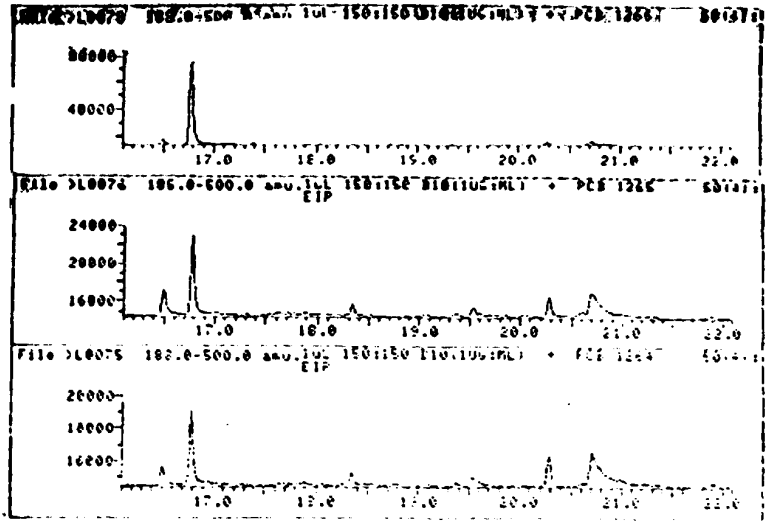
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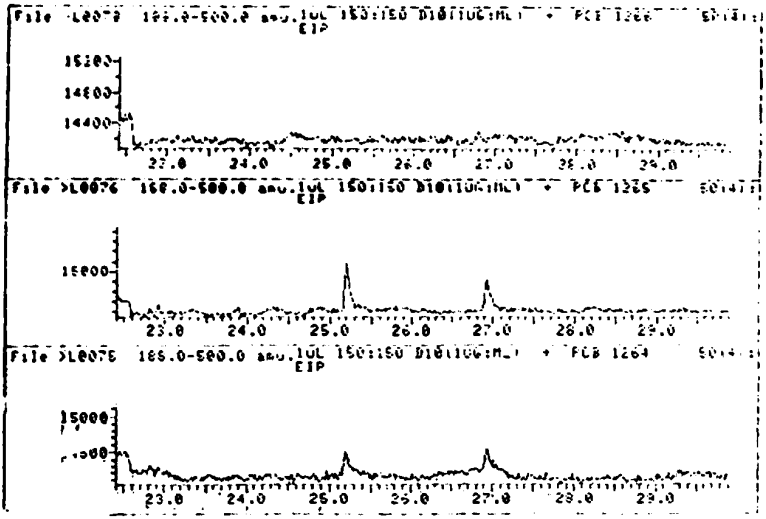
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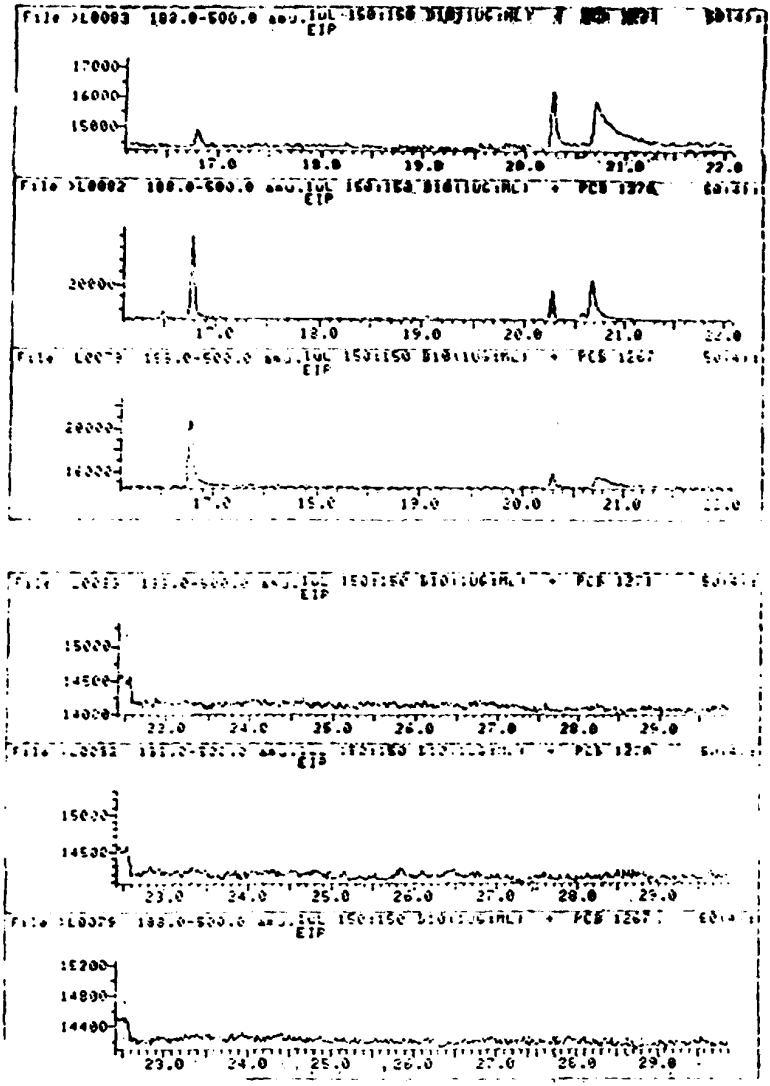
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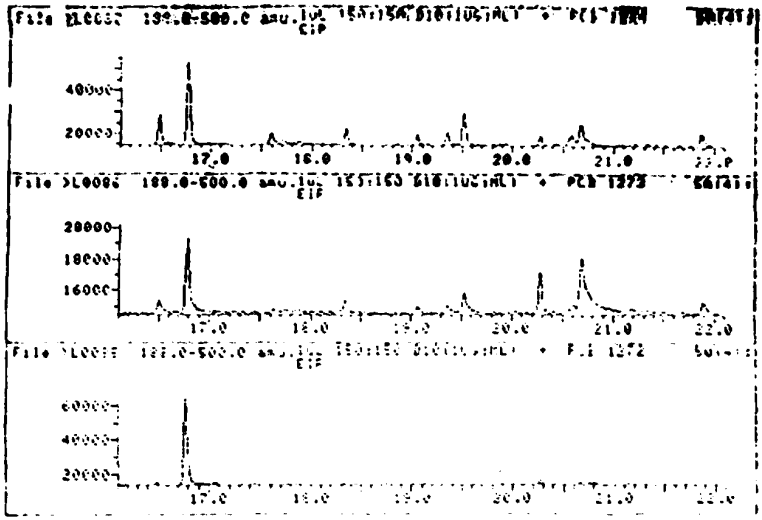
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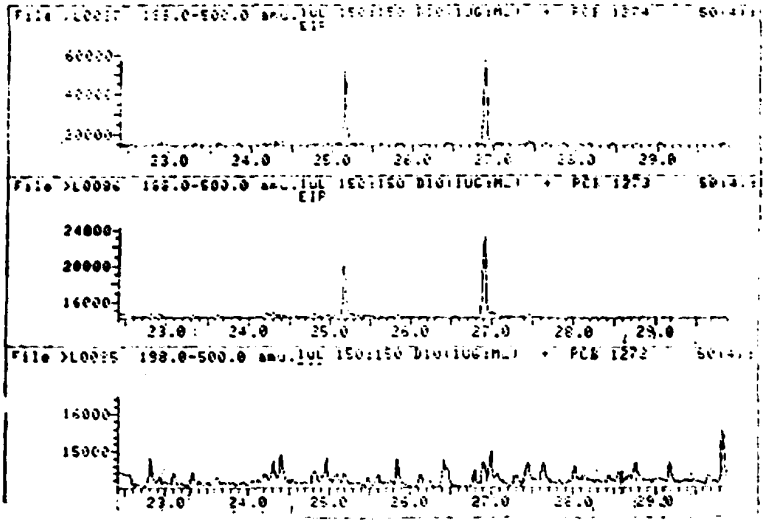
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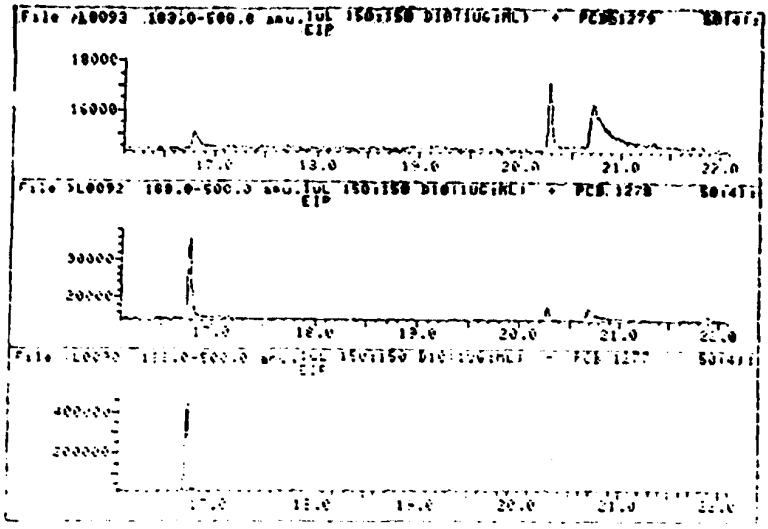
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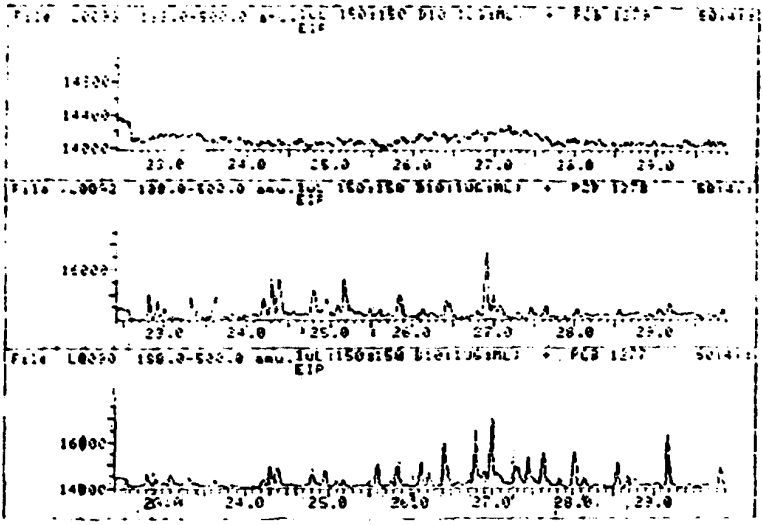
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28.0
29.0
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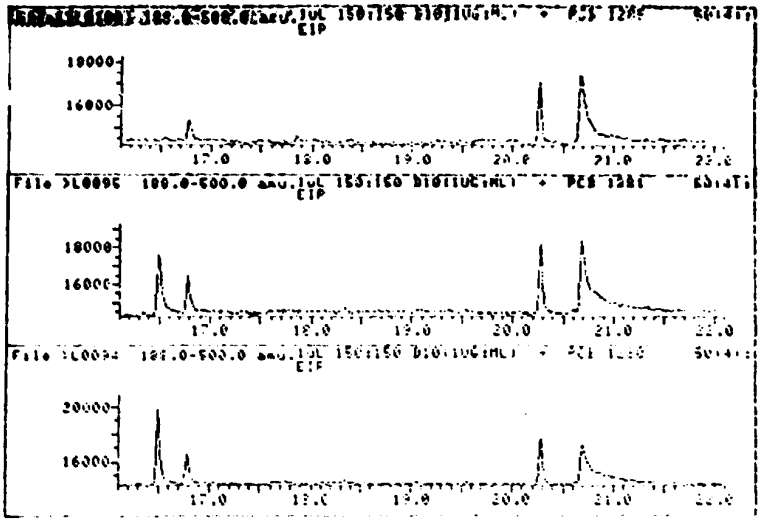
14000
EIP
16000
14000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16000
17.0
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EIP
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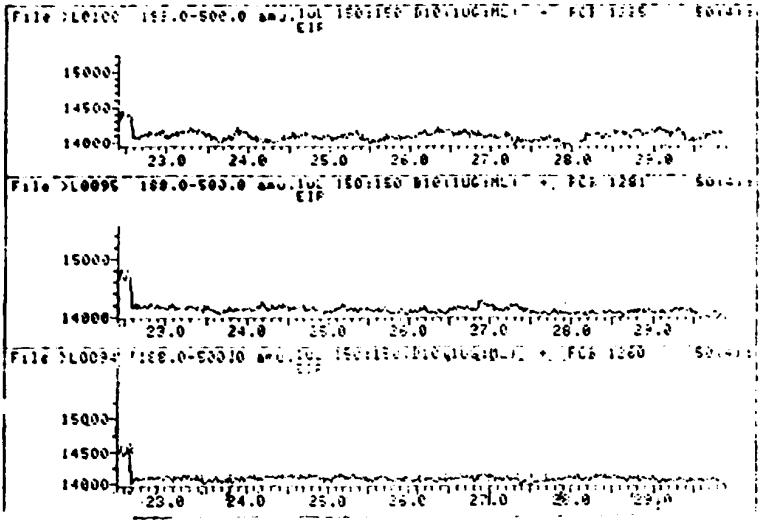
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29.0
15000
14500
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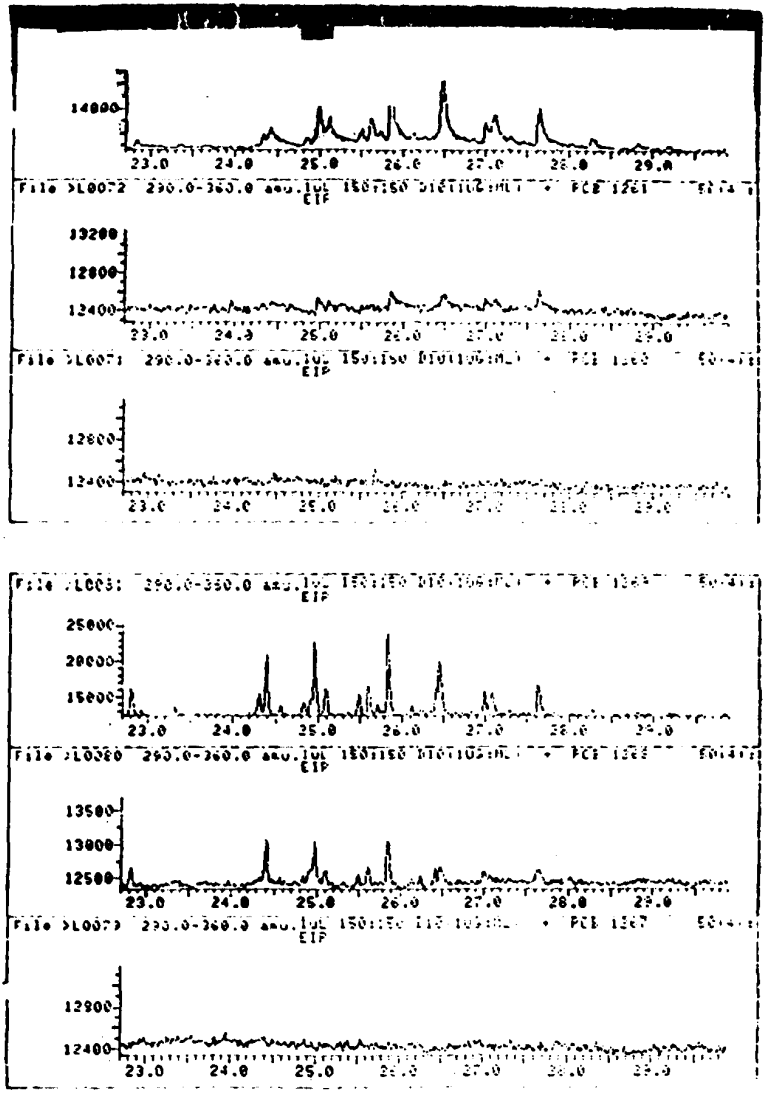
23.0
24.0
25.0
26.0
27.0
28.0
29.0

The following pages show QA/QC data for selected samples.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24.0
25.0
26.0
27.0
28.0
29.0
EIP
13200
12800
12400
EIP
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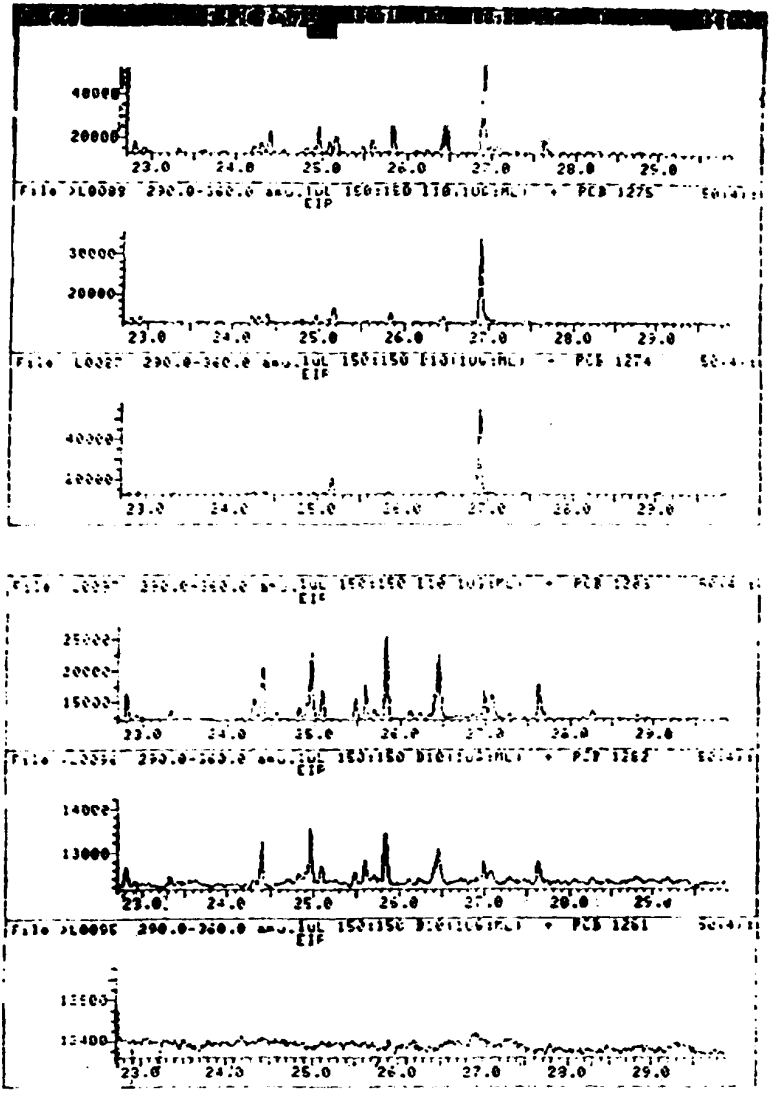
13000
12500
EIP
12800
12400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26.0
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28.0
29.0
EIP
PCB 1275
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PCB 1274
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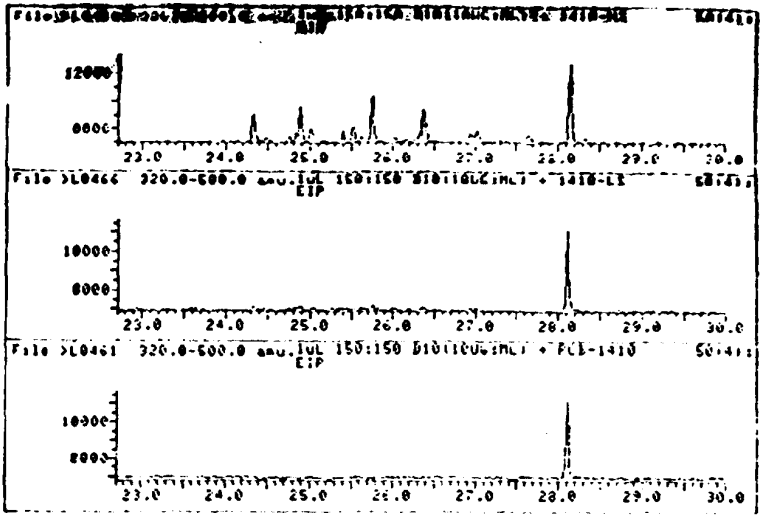
13000
PCB 1261
13500
13400


EIP
23.0 24.0 25.0 26.0 27.0 28.0 29.0 30.0
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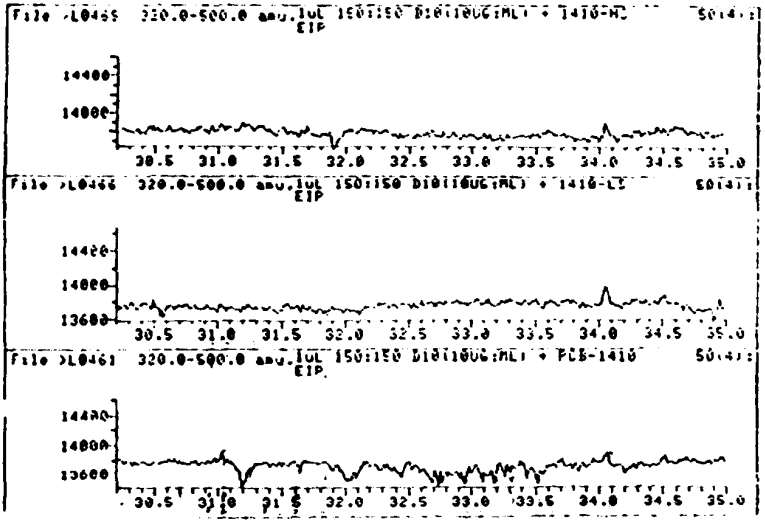
EIP
30.5 31.0 31.5 32.0 32.5 33.0 33.5 34.0 34.5 35.0
EIP
30.5 31.0 31.5 32.0 32.5 33.0 33.5 34.0 34.5 35.0
EIP
30.5 31.0 31.5 32.0 32.5 33.0 33.5 34.0 34.5 35.0

The following pages describe Standard Extraction Methods and Standard Analysis Methods used for this project.

ESC Standard Extraction Method (SEM)
TITLE: Wide scan extraction method for water.

ESC Standard Extraction Method No: WSEX05

MATRIX:	TYPICAL SAMPLE SIZE (Liters)	TYPICAL EXTRACT VOLUME (ml)
water	0.01 - 4	1 - 40

TYPICAL ANALYTES: Aroclors, PCBs, priority pollutants

ESC Standard Analysis Method Nos: ECANxx and MSANxx

INTERFERENCES: Various: See individual interferences for individual analytes.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
phenol-d5	20-80
Cl-phenol-d4	20-80
CL2-benzene-d4	50-120
naphthalene-d8	50-120
pyrene-d10	50-120
chrysene-d12	50-120
pentachlorobenzene	50-120
2,4,6-trichloro-biphenyl	50-120

APPARATUS AND MATERIALS:
Burdick and Jackson methylene chloride
Separatory funnels
Sodium sulfate for extract drying
Sodium hydroxide and sulfuric acid for pH adjustments
Nitrogen for extract volume reduction
Water bath and K-D apparatus for extract volume reduction

LIMITATIONS: Sufficient sample amount to provide GC-MS LOD.

SAFETY PRECAUTIONS: See Safe Laboratory Practices.

DATES OF METHOD CREATION AND UPDATES: 5/16/84, 10/5/84, 12/7/84

FLOW SHEET USE505
FOR ESC EXTRACTION METHOD USE005

SAMPLE ID: ---------- NRC EXTRACT #----------

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

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

RECORD NATIVE pH ---------- WAS pH ADJUSTED YES/NO IF YES TO WHAT pH----------

PLACE SAMPLE INTO AN APPROPRIATELY SIZED SEPERATORY FUNNEL (1/2 HEAD SPACE SUGGESTED FOR ADEQUATE MIXING) [ ]

SAMPLE VOLUME ----------

SURROGATE SPIKE

IF pH ADJUSTED ( )

ADJUST WITH 50% NaOH OR H SO
2 4

ADD METHYLENE CHLORIDE IN THE APPROPRIATE AMOUNT (1 L SAMPLE 30 mL SOLVENT, 100 mL SAMPLE 15 mL SOLVENT) [ ]

RECORD AMOUNT METHYLENE CHLORIDE ADDED FOR EACH EXTRACTION

1)----------

2)----------

3)----------

SHAKE SAMPLE ONE TIME AND VENT THROUGH STOPCOCK REPEAT DOUBLING THE NUMBER OF SHAKES BETWEEN VENTING SAMPLES UNTIL THE NUMBER OF SHAKES EXCEED 8! [ ]

OBSERVATIONS

ALLOW PHASES TO SEPARATE [ ]

REPEAT EXTRACTION PROCEDURE TWO [ ] [ ] TIMES

DRY EXTRACT WITH 4 INCH SODIUM SULFATE COLUMN (COLUMN PREPARED WITH 30 mL METHYLENE CHLORIDE) [ ]

[ ] EXTRACT DRYING NOT REQUIRED

RINSE SODIUM SULFATE COLUMN WITH 2 X 30 mL [ ]

REDUCE EXTRACT VOLUME TO 1 mL BY K-D TECHNIQUE AND OR NITROGEN SOLVENT VOLUME REDUCTION [ ]

[ ] TRANSFER EXTRACT INTO VIAL FOR ANALYSIS

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

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

MONS 023663

ESC Standard Extraction Method (SEM) **ESC Standard Extraction Method No:**
TITLE: PCBs in base hydrolizable or ionizable matices **PCEX15**

MATRIX(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Chemical products which are base hydrolizable or ionizable, ie. chloro acetyl chloride, chloro phenols, formic acid, etc.	0.5 - 1g	1 - 10

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

INTERFERENCIES:	TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
Any electron capturing species in the retention time region of monochlorobiphenyl isomers through decachlorobiphenyl isomers for ECANI and ECANII. Any specier in the appropriate retention time region which give the same relative characteristic mass responses as PCB isomers for MSANII.	pentachlorobenzene	50 - 120
	2,4,6-trichloro-biphenyl	50 - 120
	3,3',4,4'-tetra-chlorobiphenyl-de	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson hexane for extraction solvent.
NCH solutions of various strengths for hydrolysis and ionization of chemical species such as ethers, acid chlorides, acids, esters, etc.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Disposable glassware.
(See flow sheet for this extraction method in PCFS15.)

LIMITATIONS: Method applies only to matrices which contain hydrolizable or ionizable electron capturing species in the monochlorobiphenyl through decachlorobiphenyl retention time region for EC dection methods.

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

DATES OF METHOD CREATION AND UPDATES: 7/25/84

FLOW SHEET PCFS15

FOR ESC EXTRACTION METHOD PCEX15

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

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

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

WEIGH OUT SAMPLE INTO A 14
OR 40 mL VIAL [ ] WEIGHT SAMPLE (1 g) ----------

SURROGATE SPIKE OF SAMPLE [ ] SPIKE ----------

ADD ---- mL ---- KOH AND [ ] -- RECORD AMOUNT AND CONC. OF BASE ADDED
SHAKE
NOTE - base should be added
slowly or gradually
to avoid violent
reactions

[ ] RINSE COLUMN WITH 2 X 20 mL
HEXANE

COLUMN DESCRIPTION
25 mL DISPO PIPET

REMOVE EXTRACT QUANTITATIVELY [ ]
WITH 3 X 4 mL HEXANE RINSES

1 cm Na_2SO_4

4 cm 1% POTASSIUM
PERMANGANATE
SILICA GEL

REDUCE SAMPLE VOLUME TO
3-4 mL WITH NITROGEN AND [ ]
APPLY TO COLUMN QUANTITATIVLY

1 cm SILICA GEL

ELUTE WITH 40 mL HEXANE [ ]

4 cm ACID SILICA
GEL

NITROGEN BLOW DOWN TO 1 mL [ ]

1 cm SILICA GEL

4 cm BASE SILICA
GEL

\XXX/ GLASS WOOL
\ /

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

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

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

MONS 023665

ESC Standard Extraction Method (SEM) TITLE: PCBs in Non-EC Active Matrices	ESC Standard Extraction Method No: PCEX21

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Soils and other non-EC active solids and solvents	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 that is non acid hydrolyzable	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.
Pierce vials.
Sulfuric acid at varying concentrations.
Ultrasonic bath.
Disposable glassware.
(See flow sheet for this extraction method in PCFS21.)

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: 12/17/84

MONS 023666

FLOW SHEET PCPS21

FOR ESC EXTRACTION METHOD PCE123

SAMPLE ID: ---------- NRC EXTRACT # ----------

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

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

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

WEIGH OUT SAMPLE INTO A 1+ OR 40 mL PIERCE VIAL	[ ]	SAMPLE WEIGHT----------
SURROGATE SPIKE OF SAMPLE	[ ]	----------

ADD 10.0 mL BENZENE TO SAMPLE.	[ ]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 2 mL OF THE EXTRACT INTO A 7 mL VIAL	[ ]	
ADD 2 mL ------ SULFURIC ACID AND SHAKE	[ ]	
ALLOW PHASES TO SEPARATE REMOVE 1 mL FOR ANALYSIS.	[ ]	FINAL VOLUME ---------- 10.0 mL

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

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

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

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

MONS 023667

ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No: MSAM03

TITLE: Organic Compounds in Solvents

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

Typical Run Time:	3 - 40 min.
Typical Multiplier Voltage:	1800 - 2800 eV
Typical Dwell Times:	200 - 500 msec/mass
Maximum # of Masses:	20 masses x 5 ranges = 100 masses
Electron Energy:	70 eV

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	0.01 - 0.1	30	30

INTERFERENCES AND LIMITATIONS: Analyte characteristic mass must be chromatographically resolved from other eluting compounds which produce the same mass.

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 internal standard quantitation technique and calibrate instrument response factors from standard solutions of analytes of interest.

Qualitative: When multiple characteristic masses are monitored for a given analyte, the ratio of characteristic mass responses must be within 20% of those for the authentic standard. For PCB analyses, the characteristic masses which are monitored are the molecular ion containing all 35-CL isotopes and the molecular ion containing one 37-CL isotope. The following table summarizes the masses and approximate retention time regions monitored for the analysis of internal standard, surrogate standards, and chlorobiphenyl (CL-BP) through decachlorobiphenyl (CL10-BP) PCB congeners.

Analyte	Masses	Retention Times (minutes)	Relative Retention Times (a)
CL-BP isomers, anthracene-d10	188.190	16 - 22.5	0.60 - 0.84
CL2-BP isomers	222.224	16 - 22.5	0.60 - 0.84
Pentachlorobenzene surrogate	247.9,249.9	16 - 22.5	0.60 - 0.84
CL3-BP isomers	255.9,257.9	16 - 22.5	0.60 - 0.84
CL4-BP isomers	289.8,291.8	22.5 - 30	0.84 - 1.12
3,4,3',4'-CL4-BP-d6 surrogate	296,298	22.5 - 30	0.84 - 1.12
CL5-BP isomers	323.9,325.9	22.5 - 30	0.84 - 1.12
CL6-BP isomers	357.8,359.8	22.5 - 30	0.84 - 1.12
CL7-BP isomers (b)	323.9,325.9	22.5 - 30	0.84 - 1.12
	391.8,393.8	30 - 50	1.12 - 1.87
CL8-BP isomers	427.7,429.7	30 - 50	1.12 - 1.87
CL9-BP isomers	461.6,463.6	30 - 50	1.12 - 1.87
CL10-BP	497.6,499.6	30 - 50	1.12 - 1.87

(a) Relative Retention Times calculated using a retention time of 26.78 minutes for 3,4,3',4'-CL4-BP-d6.

(b) CL7-BP isomers are analyzed using either CL2-loss characteristic ions (m/e 323.9 and 325.9) or molecular ions (m/e 391.8 and 393.8).

CALCULATIONS: Analyte concentration = (Analyte area)/[(Analyte RRF)(IS area)]

where: Analyte area is the characteristic mass area of the analyte.
RRF is the relative response factor for the analyte.
IS is the internal standard and is usually anthracene-d10.

RRF = (analyte area in standard)/[(analyte conc.)(IS area)]

PCB concentrations are calculated in one of two ways:

1) If an Aroclor formulation can be identified from the patterns of PCB isomers, selected PCB isomers or classes of isomers (i. e. all CL4-biphenyl isomers) are used to quantify the Aroclor concentration. Relative Response Factors are calculated using the concentration of the total Aroclor formulation in the above RRF equation.

2) If no unique Aroclor formulation is present, then the total PCB concentration is calculated by using standard solutions containing known amounts of chlorobiphenyl through decachlorobiphenyl isomers to calculate Relative Response Factors for each chlorine class in the above RRF equation. The RRFs for the corresponding PCB isomer classes are used to calculate total PCB isomer concentrations of each class in unknown samples.

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

DATE OF METHOD CREATION: 12/9/83 Time Stamp -.(850719.0936)

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

Time Stamp: 19850805.1944

MONSANTO COMPANY/DAYTON LABORATORY

ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 3/29/85 LOG NO: 1-85-03-29-01

REQUESTER: Kumerich/Ivory/ J.A. Gloeckner PROJECT NO: 760.10-317

REPORT RESULTS TO: FILES/Mees, Ivory

ANALYSIS REQUESTS SENT TO: FILES/ Ivory, Sherman, Hughes

SAMPLE PICKUP BY: Chi Trang

SAMPLE LOCATION: Lab. 28, BUILDING 2

SAMPLE DESCRIPTION FOR 11 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER S)
02926-1 Still Bubble-Soil	1	PCB-1270
0292 -2 " Concrete	1	PCB-1272
0292 -3 " Soil	1	PCB-126?
		PCB-1268-LS,PCB-126?-MS
0292 -4 " Concrete	1	PCB-1273
0292 -5 " Soil	1	PCB-1264,PCB-126?-DUP
0292 -6 " Concrete	1	PCB-1273,PCB-1274-DUP
		PCB-1275-LS,PCB-1276-MS
0292 -7 " Soil	1	PCB-1266
0292 -8 " Concrete	1	PCB-1277
Blank Soil		PCB-1271
Method Blank, Bubble		PCB-1279
02927-1 Thermino1 60	1	PCB-1410
		(1.0700 g. + 9 mls benzene)
" " QA/QC		PCB-1410-LS and PCB-1410-MS
02931-1 MODCB Waste Solid	1	PCB-1259,PCB-126?-DUP
		PCB-1261-LS,PCB-126?-MS
Blank for MODCB sample		PCB-126?
02931-2 ODCB Waste Solid & Water	1	PCB-1280,PCB-1281-DUP
		PCB-1282-LS,PCB-1283-MS
Blank for ODCB waste solid (solid part)		PCB-1284
Water from 02931-2		PCB-1285,PCB-128?-DUP
		PCB-1287-LS,PCB-1288-MS
Blank for water		PCB-1289

RECORDED BY: W. Mees

REQUIRED ANALYSES: PCBs & Chlorinated Benzene

ANALYTICAL TECHNIQUES:

TECHNIQUE,(EN)	DATE COMPLETED	TECHNIQUE(EN)	DATE COMPLETED

-X- GC/MS (18) 8/7/83

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED: As soon as possible.

COMMENTS:

SAMPLE DISPOSITION: -- DISCARD -- RETURN X- HOLD FOR 30 DAYS -- OTHER----

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA317:LI

MONS 023672

The following is a copy of the ESC/DAC Good Laboratory Practices Manual.

[redacted]

GOOD LABORATORY PRACTICES MANUAL

A Guide to Quality Control/Quality Assurance

Ultratrace Analysis Group

March 1984

Joseph J. Brooks
Group Leader, Ultratrace Analysis

H. A. Waltermann
Manager, Environmental Sciences Center

Issued to: _____

Date: _____

Environmental Sciences Center
Dayton Laboratory
Dayton, Ohio 45407

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II. Objectives

The main objectives of the Ultratrace Analysis Group quality assurance/quality control program are: 1) to assure that our laboratory generates high quality results; and 2) to maintain the necessary records that document laboratory performance.

II. Scope

The scope of this manual includes areas of GLP not specifically addressed in the ESC section manual and expands upon certain areas where QA/QC practices differ from those described in the section manual. It is intended to be used along with the section manual to provide a complete picture of the GLP within the Ultratrace Analysis Group.

III. Analysis

The generation of analytical results within the Ultratrace Analysis Group routinely involves two processes: sample preparation and instrumental analysis. Sample preparation involves one or more of a variety of techniques including extraction, dilution, acid/base washes, column chromatography, concentration and numerous other techniques to render the sample in a suitable condition for instrumental analysis. Instrumental analysis takes the suitably prepared sample and introduces it into an analytical instrument to measure the desired parameter(s). Both of these processes can be described by clearly defined and separable methods called sample preparation methods (SPMs) and instrumental analysis methods (IAMs). SPMs and IAMs may be combined in numerous ways (depending upon the nature of the samples and the needs of the customer) to produce a successful analytical protocol. For example, several different SPMs may be used to produce samples that are suitable for analysis by a single IAM. The measures incorporated into each of these experimental processes to ensure that an assessment of the quality of the results can be made constitute the QA/QC program for a project. The following subsections describe the elements of the analysis process:

A. Methods

Methods are the written documentation that describe the analytical process. They contain sufficient detail such that a competent scientist or technician can readily perform the required analyses. The usual practice is to have methods that describe the sample preparation process (SPMs) and methods that describe the instrumental analysis process (IAMs). Each method developed within the Ultratrace Analysis Groups is assigned a method number as described in the ESC Section Manual.

Methods are obtained from various sources including in-house development, modification/adaptation of literature methods, or specific protocol methods dictated by government agencies. Methods may be formally validated by a series of experiments that describe a classical validation protocol or may be less vigorously proven owing to factors such as frequency or use, customer needs and

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cost/timing restrictions. In any case a QA/QC program is applied to the analysis process to provide a measure of the quality of the results. Unless a QA/QC program is prescribed by a required protocol (e.g., EPA Dioxin in Soil protocol), certain minimum QA/QC measures (depending upon the analysis type) are incorporated into all Ultratrace Analysis projects. The following subsection describes the Ultratrace QA/QC program:

B. QA/QC Program

An effective QA/QC program incorporates experiments that provide information relative to each of the following questions:

(1) Are there any impurities, background or contamination that will influence the results?

(2) How reproducible are the results?

(3) How well can the analyte be recovered from the sample matrix?

(4) What is the limit of detection for the analysis?

(5) What is the dynamic range of the analysis?

(6) What is the accuracy of the analysis?

All of these questions are addressed by properly selecting/applying the use of blanks, replicates, spikes and standards in a QA/QC program. The discussion of the use of these QA/QC techniques as they apply to Ultratrace Analysis Group activities is best done by considering the sample preparation and instrumental analysis processes separately.

1. Sample Preparation QA/QC

   a. Method blanks - Representative glassware, solvents and reagents are used with sample matrices which are similar to those for which results are being reported, yet do not contain the analytes of interest. Method blanks are analyzed at a frequency equal to 10% of the analyzed samples for sample sets which are multiples of 10. At least one method blank is analyzed for sample sets containing between 1 and 9 samples.

   b. Replicates - If sufficient sample amounts have been submitted for analysis, replicate frequency is the same as the method blank frequency. If at all possible, replicates should be obtained from the same sample bottle in order to minimize sample inhomogeneity problems due to samples being taken at slightly different times.

   c. Spikes - If sufficient sample is available for each sample type, the sample is split and spiked at two levels with

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the native (unlabeled) analyte(s). The levels are typically 2x (low) and 20x (high) the anticipated detection limit and serve as a technique for estimating the limit of detection for each matrix. These low and high spikes are conducted at a frequency equal to the Method Blank frequency. Obviously spiking with the native analyte cannot be done for widescan analysis since this presupposes knowledge of the identity of the analyte(s) in the sample.

Other spiking techniques are used to evaluate the extraction/recovery efficiencies for the analyte(s) of interest. Ideally extraction/recovery efficiencies are determined for each sample, since, in principle, each sample may represent a different matrix. This can be done very easily when the detection technique is mass spectrometry if an isotopically labeled version of the analyte(s) is available. Documentation of extraction/recovery efficiencies is not required for each sample if the sample matrix for a set of samples is constant. However, for a sample set for which the similarity of samples is not known, and for which adequate sample is available, each sample must be split and spiked with a known amount of the analyte(s) of interest to determine extraction efficiency. If adequate sample size is not available or the identity of the analyte(s) is not known before the fact (i.e., widescan analysis), surrogate spiking compounds may be used to assess extraction efficiencies in each sample without actually spiking each sample with the analyte(s) of interest. The choice of the surrogate spiking compound is tailored for each analyte. This surrogate must be chosen so that it is either chemically identical to the analyte of interest (for example using isotopically labeled analogues of the analytes), or is closely similar in terms of liquid/liquid partition properties, chemical reaction properties, column elution properties or other matrix isolation properties which are used to isolate the analyte(s) from the sample matrix. Another property of the surrogate spiking compound is that it not be present in the sample being analyzed.

2. Instrumental Analysis QA/QC

The final step in obtaining analytical results involves the use of an appropriate instrumental technique to produce the analytical data. Several QA/QC practices must be incorporated in this process to ensure validity of the data.

a. Instrumentation

Careful documentation of instrument usage, calibration, and maintenance is essential for the generation of high quality results. Each major instrument within the Ultratrace Analysis Group will be the assigned responsibility of a professional employee designated as the Instrument Steward. The Instrument Steward is responsible for making sure that logbooks are available and properly completed by instrument users. It is the

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responsibility of each individual using the equipment to record all the pertinent data required in the logbook. An example of a page from a GC/MS logbook is given in Figure 1 and that of a GC/EC logbook is given in Figure 2.

Instruments will be calibrated according to the instrument manufacturers' specifications or according to the method being used with the instrument. A log of instrument calibration will be maintained with the instrument.

The Instrument Steward will be responsible for ensuring that preventative and corrective maintenance programs are carried out on all instruments under his/her responsibility. If a specific problem is noted by an instrument user, it should be noted in the logbook and the Instrument Steward notified so that appropriate corrective action can be taken.

b. Blanks

A solvent blank will be analyzed at a frequency equal to the method blank. The method blank can serve as the solvent blank provided it is clean in the area of the analyte(s) of interest. This practice eliminates the possibility of background instrumental contamination.

c. Quantitation Internal Standard

A quantitation internal standard will be added to each sample following sample preparation but prior to instrumental analysis. This compound will serve for determining relative response factors and for correcting for variations in sample injection volumes and evaporation of solvents.

d. Standards

Standards are solutions of the analyte(s) of interest used to determine the response of the detector over the range of analyte concentrations found in the samples. For Quantitative analyses, sufficient standards and replication of standards will be run to demonstrate linearity and precision of the detector response over the range of analyte concentrations. Should sample concentrations fall outside the demonstrated linear range, either additional standards are prepared to expand the range, or appropriate dilution/concentration of the sample is done to place it in the proper range.

C. Degrees of Quantitation

The information provided for any particular analysis is tailored to the needs of the customer. In actual practice these needs vary across the broad continuum from the grossly qualitative to the precisely quantitative. It is informative to examine what is involved in providing the varying degrees of quantitation required by our customers.

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HP 5985A

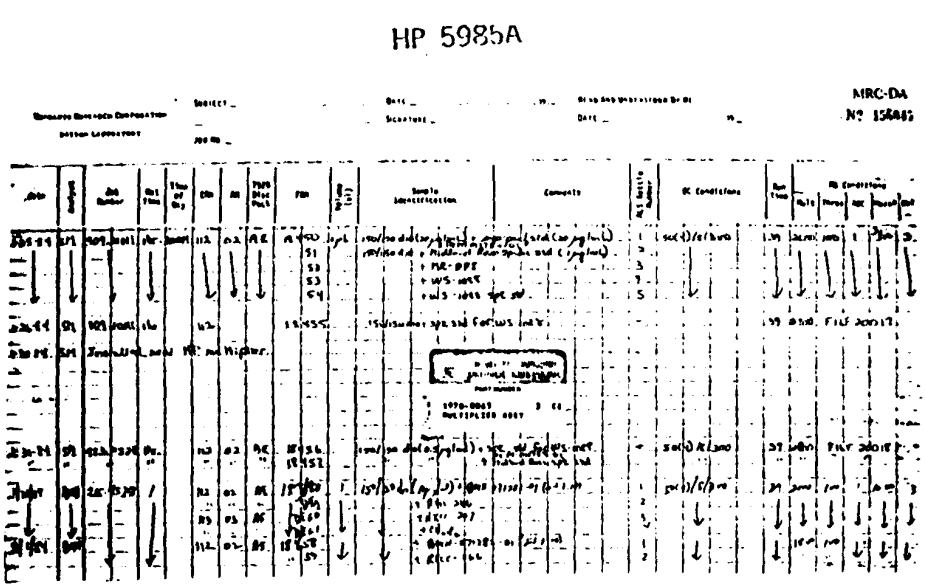

FIGURE 1. Example of GC/MS logbook page.

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HP 5880 GC/EC

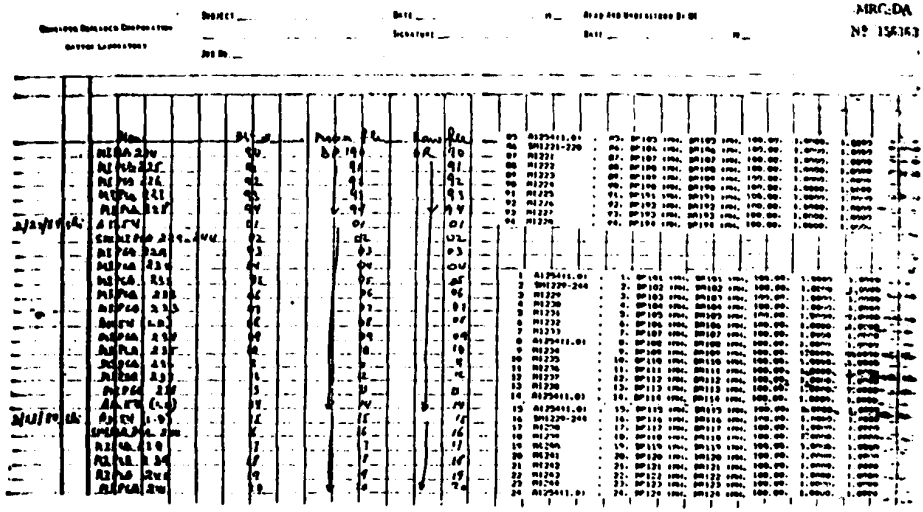

FIGURE 2. Example of GC/EC logbook page.

1. Qualitative Analysis

Qualitative analysis implies no quantitation at all but rather seeks only the identity of the compound(s) present. GC/MS is particularly applicable to this type of analysis problem. Two types of identifications can be made:

a. Tentative

A tentative identification is made on the basis of the analyst's judgement of a match between the mass spectrum from the sample and a mass spectrum from a reference library. This is often done for the large number of compounds found in a widescan organic analysis.

b. Confirmed

A confirmed identification requires that the chromatographic retention time and the mass spectrum of the unknown compound match those of a known standard of the tentatively identified compound. This is often done in widescan analyses when standards can be obtained for tentatively identified compounds that are of concern.

The normal QA/QC requirements for qualitative analysis are (1) analysis of a blank, (2) analysis of duplicate samples and (3) use of a calibration compound (usually Decafluorotriphenylphosphine, DFTPP) to assure that the mass spectrometer will produce mass spectra that can be compared with library spectra.

2. Semi-Quantitative Analysis

Semi-Quantitative Analysis extends the information provided by a qualitative analysis to include some estimate of the amount of analyte(s) present. The method of semi-quantitation involves the comparison of detector response of the analyte(s) with that of an internal standard. The estimated concentration is obtained by assuming a unit response factor between the analyte(s) and the internal standard(s). An indication of recovery from the sample matrix is obtained from the recovery of the surrogate internal standard(s).

3. Quantitative Analysis

Quantitative Analysis provides for the accurate assessment of the amount of analyte(s) present in a sample. To be able to do quantitative analysis one must know the identity of the analyte so that standards can be prepared for spiking and for the generation of calibration curves. The method of quantitation is based on a three-point calibration curve using standards of the analyte(s) of interest over the range of concentrations in the samples. A one-point calibration method can be used (as with the laboratory data

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system) provided that linearity has been demonstrated over the concentration range of the samples using standards of the analyte(s) at three concentrations in the range. This calibration curve is then used to provide concentrations of the analyte(s) in the samples via an external standards method or more preferably via an internal standards method. For the internal standards method, the quantitation internal standard is added to each of the standards (just as with the samples) prior to instrumental analysis and relative response factors are determined.

For quantitative analysis the mass spectrometer is operated as a gas chromatographic detector. The MS is therefore tuned and operated in a manner specified in the approved method rather than using a calibration compound as required for qualitative analyses.

2. Quality Criteria and Remedial Action

The inclusion of QA/QC measures into an analysis program has little meaning unless the results are interpreted and appropriate action is taken when necessary. The ability to do this assumes some set of criteria that will trigger a response or remedial action. The following subsections describe the criteria that are used in the Ultratrace Analysis Group to evaluate when quality control data indicate a need for corrective measures and what remedial action will be taken.

1. Quality Criteria

The overriding quality criterion above all others is: Do the results meet the needs of the customer. This determination will be made jointly between the customer and the analyst. The analyst must exercise caution in not allowing the customer to overstate, misinterpret or unintentionally misrepresent the data. In addition to this prime quality criterion, the following conditions must be met:

a. Blanks - In the event that a blank produces a positive value, it may not be used as a valid blank if it exceeds 10% of any reported value for a sample.

b. Recoveries - Recoveries for all surrogate and native spikes must lie in the range of 50%<Recovery<150%.

c. Precision - Precision will be calculated based on the recoveries of surrogate internal standards. Average recoveries will be calculated with ± standard deviation for each sample set. Replicate analyses and low and high native spike recoveries may also be used to provide additional information/insight into the precision of the analysis.

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d. Limit of Detection - The limit of detection (LOD) will normally be stated as ½ the value of the low level native spike if it is recovered and quantifiable. Should the low level native spike not be recovered, the LOD will be stated as ½ the value of the high level native spike if it is recovered and quantifiable. Should neither spike be recovered, a decision must be made regarding the advisability of reprocessing the sample. Should all samples contain such large quantities of the analyte(s) that the levels of the low and high spikes are insignificant (≤10%) by comparison, the value of the LOD becomes less important and is simply stated as the LOD for the instrumental analysis process determined by the analysis of standards and the ability to recover the surrogate spikes.

e. Accuracy - The accuracy of the analyses is best determined by analyzing some sample which contains the analyte at some predetermined level (e.g., an NBS standardized sample). If this type of standard is not available then the low level and high level spikes can be used as a measure of the accuracy of the analyses. Results must fall within a range of 75% to 120% of the excepted value for the analyses to be considered valid.

If the above quality criteria are not met the analyst will immediately assess the need for and extent of remedial action that may be necessary.

2. Remedial Action

The first course of action will be for the analyst and/or group leader to contact the customer and discuss the results pointing out the limitations of the data indicated by the quality control results. At this point the overriding quality criterion of customer needs will be addressed first. It could be that the customer's needs are met without meeting one or more of the other criteria. In such a case no remedial action is necessary. It could also be that the customer has such a demand for tightness in the results that his needs are not met even though the other quality criteria are. This would trigger further analyses with tighter quality control designed around the results obtained (e.g., replications of positives to better establish precision or spikes of non-detected samples to better establish the limit of detection).

In consultation with the customer additional experiments will be designed including the appropriate quality control to ultimately provide the results of sufficient quality to meet the customer's needs.

IV. Data

This section deals with the subject of data and how they are obtained, recorded and reduced so as to be useable in a report and yet readily available for referral and inspection.

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The samples will be labeled with a log number at sample login according to the procedure described in the ESC Section GLP Manual. When delivered to the responsible technical person, the samples may then receive additional labeling as deemed necessary. This additional labeling will be documented in a laboratory logbook and/or notebook. These logbooks and notebooks will be used by the analysts to record all pertinent work and results obtained from the samples. The laboratory notebooks will be filled out in accordance with Monsanto Uniform Notebook Procedures.

All raw data generated for a given project, which cannot be conveniently placed in the laboratory notebook (e.g., chromatograms, computer output, etc.), will be filed under some prescribed order (i.e., log number) in a central file under the supervison of the Project Leader and/or QA Specialist.

Most of the data generated by the Ultratrace Analysis Group are computer data stored on magnetic discs. In order to archive these data the specific file name and disc number associated with the analysis will be written down in the laboratory notebook and/or instrument logbook. When the disc is completely full of data an entire unedited disc image or copy of the files on the disc will be transferred to magnetic tape. A logbook will be kept for recording the disc number, name and prefix associated with it, along with the associated tape and file number to which the disc was transferred. An example of such a logbook is given in Figure 3. When the data are transferred a verification program is used to check all transferred records for correctness. When tapes are filled (they typically hold seven to ten disc images) they should be stored in a fireproof vault.

All calculations of the results of the data will be specified in the method used. These calculations will be entered in the analysts laboratory notebook unless they are computer generated. In this case, a copy of the computer output will be filed with the raw data. The Project Leader will be responsible for checking the correctness of the data.

V. Sample Disposal

Samples will be disposed of in accordance with the instructions on the Analysis Request form. The only exception to this would result when the analysis reveals a problem which requires sample disposal in a manner other than that originally specified. If the Project Leader notes from the results of the analysis that a potentially toxic product was present in the samples then the samples will be disposed of in a manner deemed safe by the Safety Department. The date of sample disposal and the manner should be noted on the request form stored with the raw data completing the chain of custody of the sample.

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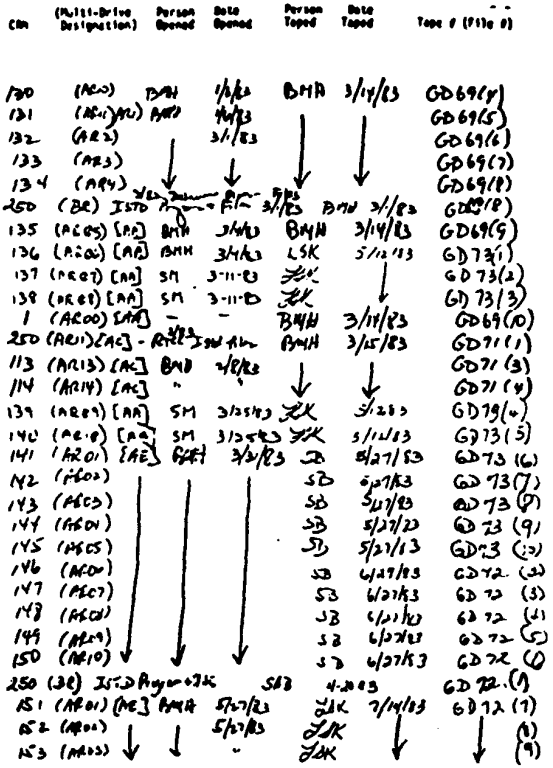

FIGURE 3. Example of DISC/Tape Logbook page.

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