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ESC - DAYTON (Department/Section)

ANALYTICAL (Type of Report)

REPORT NO.: MDA - 448

JOB/PROJECT NO.: 484.5070

DATE: 6 February 1984

PERIOD COVERED:

TITLE: ANALYSIS OF SEDIMENT FOR PCB'S

AUTHORS: B. M. Hughes

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ABSTRACT: Sediment samples from Monsanto Triangle Park were analyzed for possible PCB concentrations. The results of the analyses are shown in this report.

Technical Approval:

J. J. Brooks
Research Group Leader

Approved By:

R. F. Ivory
Environmental Sciences Center
Service Project Manager

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Approval Date: 2-6-84

REPORT NO.: MDA - 448 AUTHORS: B. M. Hughes
COPY NO.: 4 TITLE: ANALYSIS OF SEDIMENT FOR PCB'S

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ACKNOWLEDGEMENT

Contributions of the following Dayton Laboratory personnel to this project are gratefully acknowledged:

Analyses:

B. M. Hughes
D. McKenzie
C. Trang

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ANALYSIS REPORT

DATE: February 3, 1984

LOG NUMBER: 01-84-01-24-01 PROJECT NO: 484.50718

TO:
Monsanto Triangle Park/ Bob Ivory.

Results phoned to David Batchelor 81-629-1315 on 1/27/84

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

Phone No: (513) 268-3411

Title: Analysis of Sediment Samples for PCBs.

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX02

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A50718

Table File No(s): B50718, C50718, D50718

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Four sediment samples were received for PCB analyses. Tables 1 - 3 summarize the results and the attached extraction and analysis methods summarize the analysis protocol used. Note that samples labelled 012084-01 and 012084-02 are greater than 50 ug/g and thus must be treated as "PCB-containing" materials.

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

ANALYST APPROVAL: B. Mason Hughes

TABLE 1. Results of Capillary GC/EC Analyses of Aroclor 1232 in Sediment Samples

ESC Project No: 483.50718 Date: January 24, 1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Sample No. 012084-01	SAMPLE IDENTIFICATION: Sample No. 012084-01 Duplicate
			CONCENTRATION (ug/g)	
Aroclor 1232	ECAN02	2(4).1(4)	248846	150366
Rng of Values	-	-	114143	109841
No. of Values			10	11
Std Deviation	-	-	43429	33466
% Rel Std Dev	-	-	17	22

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)		RECOVERY (%)
Pentachlorobenzene	1.0	-	-

DOCUMENTATION

ESC Extraction Method	PCEX02	PCEX02
Sample Amt Extracted (g)	.1468	.1387
Extract Final Volume (ml)	10	10
Extract Dilution	1:500	1:500
ESC Extract No.	PCB264	PCB260
Process File Name	BP131	BP122

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g

ND = none detected with a detection limit given by the LOD value

NA = not analyzed

TABLE FILE NO: B50718

MONS 022774

TABLE 2. Results of Capillary GC/EC Analyses of Aroclor 1232 in Sediment Samples

ESC Project No: 483.50718 Date: January 24, 1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Sample No. 012084-02	Sample No. 012084-02 Duplicate	Sample No. 012084-02 Duplicate
			CONCENTRATION (ug/g)		
Aroclor 1232	ECAN02	7(2),3(2),1(3)	7150	3866	11674
Rng of Values	-	-	6472	3355	11898
No. of Values	-	-	10	10	10
Std Deviation	-	-	2163	1132	4213
% Rel Std Dev			30	29	36
SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)	RECOVERY (%)		
Pentachlorobenzene		1.0	-	-	-
DOCUMENTATION					
ESC Extraction Method			PCEX02	PCEX02	PCEX02
Sample Amt Extracted (g)			1.4083	.1402	1.0186
Extract Final Volume (ml)			10	10	10
Extract Dilution			1:100	1:10	1:100
ESC Extract No.			PCB265	PCB261	PCB266
Process File Name			BP132	BP121	BP133

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g

ND = none detected with a detection limit given by the LOD value

NA = not analyzed

TABLE FILE NO: C50718

TABLE 3. Results of Capillary GC/EC Analyses of Aroclor 1232 in Sediment Samples

ESC Project No: 483.50718 Date: January 24, 1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Sample No. 012084-03	Sample No. 012084-04	Method Blank
			CONCENTRATION (ug/g)		
Aroclor 1232	ECAN02	1.0	ND	ND	ND
Rng of Values	-	-	-	-	-
No. of Values	-	-	-	-	-
Std Deviation	-	-	-	-	-
% Rel Std Dev	-	-	-	-	-

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)	RECOVERY (%)		
Pentachlorobenzene	1.0	89	76	105

DOCUMENTATION

ESC Extraction Method	PCEX02	PCEX02	PCEX02
Sample Amt Extracted (g)	1.0010	.9424	0.0
Extract Final Volume (ml)	10	10	10
Extract Dilution	1:1	1:1	1:1
ESC Extract No.	PCB267	PCB268	PCB269
Process File Name	BP125	BP126	BP127

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g

ND = none detected with a detection limit given by the LOD value

NA = not analyzed

TABLE FILE NO: 050718

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

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-3356-LAS Laboratory Data System

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

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

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

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

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

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

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.

DATES OF METHOD CREATION AND METHOD UPDATES: 10/25/83, 2/3/84

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

ESC Standard Extraction Method No: PCEX02

MATRI(X),(CES):

Soils and other non-EC active solids and solvents

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

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

ESC Standard Analysis Method Nos: ECAN01, ECAN02

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

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

APPARATUS AND MATERIALS:
Burdick and Jackson benzene for sample extraction or dilution.
Disposable glassware.

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

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

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

ANALYSIS REPORT

DATE: February 14, 1984

LOG NUMBER: 1-84-02-07-01 PROJECT NO: 484.50718

TO:

Monsanto Triangle Park/Ivory

FROM:

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

Phone No: (513) 268-3411

Title: Analysis of One Sediment for PCBs

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX02

Standard Analysis Method No(s): ECAN02

Final Report File No(s): F50718

Table File No(s): E50718

One sample labelled 20384-1 was received from Monsanto Triangle Park for PCB analyses. Table 1 gives the results. Also included are brief descriptions of ESC Standard Extraction and Analysis Methods used and a description of the table format. Please note that the sample contains more than 50 ug/g Aroclor 1232 and thus must be treated as a PCB-containing material.

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

ANALYST APPROVAL: B. M. Hughes

TABLE 1. Results of Capillary GC/EC Analyses of Aroclor 1232 in Sediment Samples

ESC Project No: 483.50718 Date: February 10,1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Sample No. 20384-1 Duplicate	SAMPLE IDENTIFICATION: Sample No. 20384-1
			CONCENTRATION (ug/g)	
Aroclor 1232	ECAN02	10	249	477
Rng of Values	-	-	217	398
No. of Values			10	10
Std Deviation	-	-	68	123
% Rel Std Dev	-	-	2.7	25.8
SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)		
			RECOVERY (%)	
Pentachlorobenzene		1.0	-	-
DOCUMENTATION				
ESC Extraction Method			PCEX02	PCEX02
Sample Amt Extracted (g)			1.0023	1.0295
Extract Final Volume (ml)			10	10
Extract Dilution			1:1	1:1
ESC Extract No.			PCB324	PCB325
Process File Name			BP101	BP101

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g

ND = none detected with a detection limit given by the LOD value

NA = not analyzed

TABLE FILE NO: E50718

MONS 022781

DETAILED EXPLANATION OF TABLES

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

Each table includes a general title, ESC Project Number and Date on which the table was generated. The sample identification is the customer identification which was on the sample bottle. The next section of the table gives results for the particular analyte listed. For Aroclor formulations, the formulation is identified either visually or from ESC developed data analysis programs. The concentration of the formulation is given by the average value of a number of PCB isomers in the formulation. The number of isomers used in this calculation is given as "No. of Values" and the range of Aroclor concentrations for this number of PCB isomers is given by "Rnge of Values". The standard deviation and % relative standard deviation for this number of determinations of the Aroclor formulation are given in the following two lines of the tables.

The next section of the table deals with quality control used in each sample analysis. The surrogate spiking compound which is added to the sample before extraction and analysis is listed, along with its amount and % Recovery measured for each sample. In cases where PCB concentrations are very high and thus require extract dilution, no effort is made to quantify this compound. This could be done if ESC knows that this data is important, but typically 50 ug/g is the important level to quantify accurately so that surrogate data for concentrations larger than this value, from a regulatory standpoint, are not important.

The next section documents how each sample was extracted and analyzed. The ESC Extraction Method, amount of sample extracted, extract final volume, extract dilution factor, ESC extract number and the computer data file in which the processed data is stored are given in this section. The last section gives a brief description of abbreviations or gives any footnotes that give additional information about specific samples.

This table format is both compact and precise. It was developed in an attempt to quickly and accurately generate data which the customer can quickly and accurately evaluate. If there are any suggestions concerning this format, please contact me at extension 396.

DESCRIPTION STORED IN TBEXO1:MH

MONS 022782

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

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

ESC Standard Extraction Method No: PCEX02

MATRIX (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, ELAN02

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

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

APPARATUS AND MATERIALS:
Burdick and Jackson benzene for sample extraction or dilution.
Disposable glassware.

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

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

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

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

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

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

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

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

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

---

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 area)/(isomer RRF)

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.

DATES OF METHOD CREATION AND METHOD UPDATES: 10/25/83, 2/3/84

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

MRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: FEBRUARY 7, 1984 LOG NO: 1-84-02-07-01

REQUESTER: RTP/HUGHES PROJECT NO: 484.50718

REPORT RESULTS TO: FILES/MEES.IVORY.BROOKS

ANALYSIS REQUESTS SENT TO: FILES.IVORY.BROOKS.HUGHES

SAMPLE PICKUP BY: D. MCKENZIE

SAMPLE LOCATION:

SAMPLE DESCRIPTION FOR 1 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	NUMBER(S)
SAMPLE NO. 20384-1	1	PCB324 PCB325

RECORDED BY: P. HUFF

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES:

	DATE COMPLETED
X GC/MS (18)	2/14/84

QUALITATIVE: YES SEMIQUANTITATIVE: QUANTITATIVE: YES

REQUIRED QA/QC:

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS:

ANALYSIS REQUEST FILE NAME: AB5070:LI