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REPORT NO.: MDA-478

JOB/PROJECT NO.: 484.5180

DATE: 30 April 1984

PERIOD COVERED:

TITLE: ANALYSIS OF PCB'S IN SAND AND CHARCOAL

AUTHORS: B. M. Hughes

ABSTRACT: Sand and charcoal samples from the Monsanto RTP facility were analyzed for their possible PCB content. The results of these analyses are shown in this report.

TECHNICAL APPROVAL:

J. J. Brooks
J. J. Brooks
Senior Research Group Leader

APPROVED BY:

R. F. Ivory
R. F. Ivory
Environmental Science Center
Service Project Manager

APPROVAL DATE: *May 1, 1984*

AUTHORS: B. M. Hughes
TITLE: ANALYSIS OF PCB'S IN SAND AND CHARCOAL

REPORT NO.: MDA-478

COPY NO.: 5

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MONS 022866

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<u>ABSTRACT ONLY</u>
H. A. Woltermann

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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: April 18, 1984

LOG NUMBER: 1-84-03-29-01

PROJECT NO: 484.51818

TO: Research Triangle Park/Bob Ivory

FROM:

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

Phone No: (513) 268-3411

Title: Analysis of PCBs in Sand and Charcoal Samples

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX02

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A51818

Table File No(s): C51818, B51818, E51818, D51818

One sand and one charcoal sample were analyzed for PCBs. Tables 1 and 3 summarize the results and tables 2 and 4 summarize QA/QC results. Approximately 1 ug/g Aroclor 1254 was found in these two samples. A summary of the extraction and analytical methods are also included for your information. The extraction method PCEX02 was modified slightly in order to improve the extraction efficiencies for the charcoal sample. This modification involved multiple ultrasonic extractions using benzene, methylene chloride, acetone and hexane, followed by extract concentration to 1 ml.

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

ANALYST APPROVAL: B. Mason Hughes

MONS 022868

**TABLE 1/ Results of Capillary GC/EC Analyses of Aroclor 1254 in Charcoal
Filter Sample from Research Triangle Park, N.C.**

ESC Project No: 484.51018

Date: April 9, 1984

SAMPLE IDENTIFICATION

Charcoal
Filter
032884-2

ANALYTE	ESC ANALYTICAL METHOD	LOD	CONCENTRATION (ug/g)
Aroclor 1254	ECAN02	0.5	1.15
Ring of Values	-	-	.50
No of Values	-	-	9
Std Deviation	-	-	.19
± Rel Std Dev	-	-	16

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)	RECOVERY (%)
CLS-benzene	ECAN02 1.0	76
Native Spiking	ECAN02 -	-
Study A1248 (See Table 2 for results from spiking charcoal with Aroclor 1248.)		

DOCUMENTATION

ESC Extraction Method PCEX02
Sample Amt extracted (g) 1.1463
Extract Dilution 1:1
ESC Extract No. PCB365
Process File Name BP371

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

Ring of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values

No of Values = Number of PCB isomers used to measure Aroclor concentration

± Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.

TABLE FILE NO: CS1018:101

MONS 022869

TABLE 2. QA/QC Results of Capillary GC/EC Analysis of Aroclor 1248 in Charcoal Filter Samples from Research Triangle Park, N.C.

ESC Project No: 484.51018

Date: April 9, 1984

SAMPLE IDENTIFICATION					
Charcoal Filter 032004-2 Method Blank		Charcoal Filter 032004-2 Low Spike		Charcoal Filter 032004-2 High Spike	
ESC ANALYTICAL METHOD					
ANALYTE	ANALYTICAL METHOD	LOD	CONCENTRATION (ug/g)		
Aroclor 1248	ECAN02	1.0	ND	1.62	6.67
Ring of Values	-	-	-	1.21	13.3
No of Values	-	-	-	20	19
Std Deviation	-	-	-	.27	2.60
% Rel Std Dev	-	-	-	32	31
=====					
SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)		RECOVERY (%)	
CLS-benzene	ECAN02	1.0	79	77	66
=====					
Native Spiking Study A1248	ECAN02	0..07,0.38	-	106	103
A1248(Corrected For A1254)	-	-	-	83	93
=====					
DOCUMENTATION					
ESC Extraction Method		PCEX02		PCEX02	PCEX02
Sample Amt extracted (g)		0		1.2671	1.4060
Extract Dilution		1:1		1:1	1:1
ESC Extract No.		PCB355		PCB367	PCB366
Process File Name		BP350		BP375	BP373
=====					
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					
Ring of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values					
No of Values = Number of PCB isomers used to measure Aroclor concentration					
% Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.					

TABLE FILE NO: 051018:NM

MONS 022870

TABLE 3. Results of Capillary GC/EC Analyses of Aroclor 1254 in Sand Filter Sample from Research Triangle Park, N.C.

ESC Project No: 484.51818

Date: April 16, 1984

SAMPLE IDENTIFICATION	
Sand Filter 032084-1	Sand Filter 032084-1 Duplicate

ANALYTE	ESC ANALYTICAL		CONCENTRATION (ug/g)	
	METHOD	LOD		
Aroclor 1254	ECAN02	0.1	.85	.74
Ring of Values	-	-	.97	.76
No of Values	-	-	12	11
Std Deviation	-	-	.34	.26
% Rel Std Dev	-	-	40	35

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)		RECOVERY (%)
CL3-benzene	ECAN02	1.0	100
Native Spiking Study A1248	ECAN02	-	-

(See Table 4 for A1248 spiking study results.)

DOCUMENTATION

ESC Extraction Method	PCEX02	PCEX02
Sample Amt extracted (g)	1.1032	1.1643
Extract Dilution	1:1	1:1
ESC Extract No.	PCB382	PCB383
Process File Name	BP442	BP443

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

Ring of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values

No of Values = Number of PCB isomers used to measure Aroclor concentration

% Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.

TABLE FILE NO: E51818INH

MONS 022871

TABLE 4. Results of Capillary GC/EC Analyses of Aroclor 1248 in Sand Filter Samples from Research Triangle Park, N.C.

ESC Project No: 484.51010

Date: April 16, 1984

SAMPLE IDENTIFICATION		
Sand Filter 032804-1 Method Blank	Sand Filter 032804-1 Low Spike	Sand Filter 032804-1 High Spike

ANALYTE	ESC ANALYTICAL METHOD	LOD	CONCENTRATION (ug/g)		
Aroclor 1248	ECAM02	1.0	ND	2.38	10.18
Ring of Values	-	-	-	3.57	14.86
No of Values	-	-	-	15	17
Std Deviation	-	-	-	.72	2.62
% Rel Std Dev	-	-	-	30	26

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)	RECOVERY (%)	
CLS-benzene	ECAM02 1.0	88	133
Native Spiking	ECAM02 0.1, 2.2, 10.76	-	195
Study A1248			
A1248(Corrected For A1254)	-	-	125

DOCUMENTATION

ESC Extraction Method	PCEX02	PCEX02	PCEX02
Sample Amt extracted (g)	0	1.0282	1.2172
Extract Dilution	1:1	1:1	1:1
ESC Extract No.	PCB386	PCB384	PCB385
Process File Name	BP444	BP447	BP449

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

Ring of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values

No of Values = Number of PCB isomers used to measure Aroclor concentration

% Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.

TABLE FILE NO: 051818:MM

MONS 022872

DETAILED EXPLANATION OF TABLES

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

Each table includes a general title, ESC Project Number and Date on which the table was generated. The sample identification is the customer identification which was on the sample bottle. The next section of the table gives results for the particular analyte listed. For Aroclor formulations, the formulation is identified either visually or from ESC developed data analysis programs. The concentration of the formulation is calculated as the average value of the Aroclor formulation, based upon the analysis of a number of individual PCB isomers in the formulation. 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 "Ring 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 these data are 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 TWF001:RM

MONS 022873

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

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

ESC Standard Extraction Method No:
PCEN02

MATRIX(X),(CES):

Solids 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, trichloro-
biphenyl isomers through octachloro-
biphenyl 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
2,4,6-trichloro-
biphenyl

50 - 120

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 interfering
electron capturing species in the trichlorobiphenyl through octachloro-
biphenyl 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

MONS 022875

ESC Standard Analysis Method (SAM)
TITLE: Aroclor Formulations in Solvents

ESC Standard Analysis Method No:
ECAM02

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

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Aroclor formulations 1237, 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 μ m) 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.

MONS 022877

NRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: MARCH 29, 1984

LOG NO: 1-84-03-29-01

REQUESTER:
RTP/HUGHES

PROJECT NO:
484.51818

REPORT RESULTS TO: FILES/NEES, IVORY, BROOKS

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

SAMPLE PICKUP BY: D. MCKENZIE

SAMPLE LOCATION:

SAMPLE DESCRIPTION FOR 2 SAMPLES		
SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	NUMBER(S)
RTP NUMBER 032884-1 SAND FILTER	1	PCB382, PCB383
Sand Filter-low spike		PCB384
Sand Filter-high spike		PCB385
Sand Filter-method blank		PCB386
RTP NUMBER 032884-2 CHARCOAL FILTER	1	PCB365
Charcoal Filter-low spike		PCB367
Charcoal Filter-high spike		PCB366
Charcoal Filter-method blank		PCB355

RECORDED BY: P. HUPP

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES:

K GC/EC (18) DATE
 COMPLETED
 4/18/84

QUALITATIVE: YES

SEMIQUANTITATIVE:

QUANTITATIVE: YES

REQUIRED QA/QC:

COMPLETION DATE REQUESTED:

COMMENTS: Result phoned to David Batchelor, RTP.

SAMPLE DISPOSITION:

X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: May contain PCBs.

ANALYSIS REQUEST FILE NAME: AAS180.L1

MONS 022878