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REPORT NO.: MDA-471

JOB/PROJECT NO.: 484.5150

DATE: 9 April 1984

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TITLE: ANALYSIS OF WATER FOR PCB'S

AUTHORS: B. H. Hughes

ABSTRACT: Three water samples were analyzed for their possible total PCB concentrations. The results of these analyses are shown in this report.

TECHNICAL APPROVAL:

J. J. Brooks
Research Group Leader

APPROVED BY:

R. F. Ivory
Environmental Science Center
Service Project Manager

APPROVAL DATE: 4-5-84

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REPORT NO.: MDA-471
COPY NO.: 5
AUTHORS: B. H. Hughes
TITLE: ANALYSIS OF WATER 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

[redacted]

ANALYSIS REPORT

DATE: March 26, 1984

LOG NUMBER: 1-84-03-20-01 PROJECT NO: 484.51518

TO:

Final Report Sent To Mr. David H. Batchelor
3023 Cornwallis Rd.
P. O. Box 12274
Research Triangle Park, N. C. 27709

FROM:

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

Phone No: (513) 268-3411

Title: Analysis of Three Water Samples for PCBs.

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX01

Standard Analysis Method No(s): ECAN01, ECAN02

Final Report File No(s): A51518

Table File No(s): B51518, C51518, D51518

Three water samples were analyzed for total PCBs using the above referanced extraction and analysis methods. Summaries of these methods are included for more detailed information. In addition to the surrogate spiking compound which was added to each sample in order to determine extraction efficiencies, one sample (identified as 031984-1) was also spiked with 10.5 ug/L Aroclor 1248, to document recovery of an Aroclor formulation. Greater than 59% recovery was measured for all surrogate compounds and the Aroclor 1248 formulation.

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

ANALYST APPROVAL: R. M. Hughes

TABLE 1. Results of Capillary GC/EC Analyses for PCBs in water from Research Triangle Park N.C.

ESC Project No: 404.51510 Date: March 22,1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Research Triangle Park 031984-1	Research Triangle Park 031984-2	Research Triangle Park 031984-2 Duplicate
			CONCENTRATION (ug/l)		
Total PCBs	ECAN01	1.0	ND	2.1	2.0
SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/l)		RECOVERY (%)	
CLS-benzene		1.0	66	63	71
DOCUMENTATION					
ESC Extraction Method			PCEX01	PCEX01	PCEX01
Processed Data File			BP290	BP288	BP293
Extract Dilution Factor			1:1	1:1	1:1
ESC Extraction Number			PCB347	PCB348	PCB349
Sample Wt. Extracted (l)			.1	.1	.1

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/l

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

NA = not analyzed

TABLE FILE NO: 851510:MH:22

MONS 022813

TABLE 2. Results of Capillary GC/EC Analyses for PCBs in water from Research Triangle Park N.C.

ESC Project No: 404.51518 Date: March 22.1984

	SAMPLE IDENTIFICATION	
	Research Triangle Park 031984-3	Research Triangle Park Method Blank
ANALYTE / ESC ANALYTICAL METHOD / LOD	CONCENTRATION (ug/l)	
Total PCBs / ECAN01 / 1.0	1.94	ND
SURROGATE SPIKING COMPOUND / AMOUNT ADDED (ug/l)	RECOVERY (%)	
CLS-benzene / 1.0	59	61
DOCUMENTATION		
ESC Extraction Method	PCEX01	PCEX01
Processed Data File	BP289	BP295
Extract Dilution Factor	1:1	1:1
ESC Extraction Number	PCB350	PCB352
Sample Wt. Extracted (l)	.1	.1

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/l

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

NA = not analyzed

TABLE FILE NO: D51518:MH:22

MONS 022814

TABLE 3. Results of Capillary GC/EC Analyses for PCBs in water from Research Triangle Park N.C.

ESC Project No: 404.51518 Date: March 22,1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Research Triangle Park 031984-1 Spiked with Aroclor 1248 CONCENTRATION (ug/l)
Aroclor 1248	ECAN02	1.0	13.2
Rng of Values	-	-	9.1
No of Values	-	-	16
Std Deviation	-	-	3.06
% Rel Std Dev	-	-	23

SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/l)	RECOVERY (%)
CL5-benzene	ECAN02	1.0	74
Native Spiking Study A1248	ECAN02	10.5	125

DOCUMENTATION

ESC Extraction Method	PCEXO1
Sample Amt extracted (l)	.1
Extract Dilution	1:1
ESC Extract No.	PCB351
Process File Name	BP294

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/l

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

NA = not analyzed

Rng 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: C51518:MH

MONS 022815

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 an 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 from the average value of the Aroclor formulation, based upon the analysis of a number of PCB isomers. 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 "Rng 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 TBEX01:MH

MONS 022816

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

ESC Standard Extraction Method (SEM) TITLE: PCBs in Various Matrices	ESC Standard Extraction Method No: PCEX01

MATRIX(X).(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Still bottoms, soils, beef fat, beef serum, whole milk, beef feces	0.5 - 5.0	1 - 10

TYPICAL ANALYTES: Aroclors 1232, 1242, 1248, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers	ESC Standard Analysis Method Nos: ECAN01, ECAN02, MSAN03

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers for ECAN01 and ECAN02. Any species in the appropriate retention time region which give the same relative characteristic mass responses as PCB isomers for MSAN03.	TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
	pentachlorobenzene	50 - 120
	2,4,6-trichlorobiphenyl	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson hexane, benzene and methylene chloride for sample extraction.
Concentrated sulfuric acid for sample digestion or sample clean-up.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Activated basic alumina.
Disposable glassware.

LIMITATIONS: Method applies only to matrices which do not contain non-hydrolizable electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region for EC detection methods.

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

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

ESC Standard Analysis Method (SAM) ESC Standard Analysis Method No:
TITLE: PCBs in Solvents ECAN01

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 (%)
Trichlorobiphenyl isomers through octachlorobiphenyl isomers	0.01	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 (0.32 mm)

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 PCB isomer standards.

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

where: RRF is the relative response factor for the appropriate PCB isomer.

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

DATES OF METHOD CREATION AND METHOD UPDATES: 10/23/83

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

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

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

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

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

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

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

CALCULATIONS: Formulation concentration = (isomer 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: 10/23/83, 2/3/84

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

MRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: MARCH 20, 1984 LOG NO: 1-84-03-20-01

REQUESTER: RTP/HUGHES PROJECT NO: 484.51518

REPORT RESULTS TO: FILES/MEES, IVORY, BROOKS

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

SAMPLE PICKUP BY: D. MCKENZIE

SAMPLE LOCATION:

SAMPLE DESCRIPTION FOR 3 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
RTP 031984-1	1	PCB347
RTP 031984-2	2	PCB348, PCB349
RTP 031984-3	1	PCB350
	Method Blank	PCB352
	RTP 031984-1 spiked with Aroclor 1248	PCB351

RECORDED BY: P. HUPP

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES:

	DATE COMPLETED
X OC/EC (IB)	3/27/84

QUALITATIVE: YES SEMIQUANTITATIVE: QUANTITATIVE: YES

REQUIRED QA/QC:

COMPLETION DATE REQUESTED: 3/21/84

COMMENTS: Results phoned to David Batchelor on 3/21/84 and 3/22/84.

SAMPLE DISPOSITION: X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: Samples may contain PCBs.

ANALYSIS REQUEST FILE NAME: AA5:[50,1]LI

MONS 022822