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DATE: 5 April 1984

PERIOD COVERED:

TITLE: ANALYSIS OF WATER FOR PCB'S

AUTHORS: B. M. Hughes

ABSTRACT: Four water samples from the Monsanto RTP facility were analyzed for possible PCB concentrations. The results of these analyses are shown in this report.

TECHNICAL APPROVAL:

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

APPROVED BY:

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Environmental Science Center
Service Project Manager

APPROVAL DATE:

7-3-84

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AUTHORS: B. M. Hughes
TITLE: ANALYSIS OF WATER FOR PCB'S

REPORT NO.: MDA-469
COPY NO.: 1

MONS 022799

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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: March 29, 1984

LOO NUMBER: 1-84-03-13-01

PROJECT NO: 484.51318

TO: Bob Ivory/Research Triangle Park

FROM:

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

Phone No: (513) 268-3411

Title: Analysis of Water 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): A51318

Table File No(s): B51318, C51318

Four water samples containing sediment were analyzed for PCBs. Tables 1 and 2 summarize the results. Note that these samples contain Aroclor 1248 rather than the Aroclor 1232 formulation found in previous samples.

ANALYST(S): B. Mason Hushes, David McKenzie and Chi Trans

ANALYST APPROVAL: B. Mason Hushes

MONS 022801

TABLE 1. Results of Capillary GC/EC Analyses of Aroclor 1248 in water Samples from Research Triangle Park, N.C.

ESC Project No: 484.51318

Date: March 27, 1984

SAMPLE IDENTIFICATION					
Research Triangle Park		Research Triangle Park		Research Triangle Park	
030784-1		030784-1		030784-2	
		Duplicate			

ANALYTE	ESC ANALYTICAL METHOD	LOD	CONCENTRATION (ug/g)		
Aroclor 1248	ECAN02	0.1	.60	.67	.97
Rng of Values	-	-	.86	.91	1.43
No of Values	-	-	21	19	21
Std Deviation	-	-	.19	.20	.33
% Rel Std Dev	-	-	32	29	34

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)	RECOVERY (%)		
CL5-benzene	ECAN02 1.0	103	120	85

DOCUMENTATION

ESC Extraction Method	PCEX02	PCEX02	PCEX02
Sample Amt Extracted (g)	10	10	10
Extract Final Volume (ml)	1	1	1
Extract Dilution	1:1	1:1	1:1
ESC Extract No.	PCB333	PCB343	PCB335
Process File Name	BP246	BP263	BP248

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

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: B51318:PM

MONS 022802

TABLE 2. Results of Capillary GC/EC Analyses of Aroclor 1248 in water Samples from Research Triangle Park, N.C.

ESC Project No: 484.51318

Date: March 27, 1984

SAMPLE IDENTIFICATION		
Research Triangle Park 030784-3	Research Triangle Park 030784-4	Research Triangle Park Method Blank

ANALYTE	ESC ANALYTICAL METHOD	LOD	CONCENTRATION (ug/g)		
Aroclor 1248	ECAN02	0.1	3.3	2.35	ND
Rng of Values	-	-	4.4	2.58	-
No of Values	-	-	21	21	-
Std Deviation	-	-	1.1	.76	-
X Rel Std Dev	-	-	34	32	-

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)	RECOVERY (%)		
CL5-benzene	ECAN02 1.0	100	104	104

DOCUMENTATION

ESC Extraction Method	PCEX02	PCEX02	PCEX02
Sample Amt Extracted (g)	10	10	0
Extract Final Volume (ml)	1	1	1
Extract Dilution	1:10	1:10	1:1
ESC Extract No.	PCB269	PCB270	PCB265
Process File Name	BP337	BP339	BP341

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

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

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

TABLE FILE NO: C51318:PM

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 3337-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 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 as "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 regulator, 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 TBEX011MH

MONS 022804

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

MONS 022805

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

ESC Standard Extraction Method No:
PCEX02

MATRIX(ES):

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, 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

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

ESC Standard Analysis Method No.
ECAN02

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 1232, 1242, 1248, 1254, 1260, and 1262	0.1	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the reten-
tion 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 µm) 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 formu-
lation 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 concentra-
tion is obtained from the average of at least 10 such cal-
culations.

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 022608

NRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: MARCH 20, 1984

LOG NO: 1-84-03-13-01

REQUESTER:

RTP/MUGHES

PROJECT NO:

484.51318

REPORT RESULTS TO: FILES/NEES, IVORY, BROOKS

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

SAMPLE PICKUP BY: D. MCKENZIE

SAMPLE LOCATION:

SAMPLE DESCRIPTION FOR 4 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
RTP SAMPLE NO. 030784-1	1	PCB333
	DUPLICATE EXTRACTION	PCB343
RTP SAMPLE NO. 030784-2	1	PCB335
RTP SAMPLE NO. 030784-3	1	PCB269
RTP SAMPLE NO. 030784-4	1	PCB270
	METHOD BLANK	PCB265

RECORDED BY: P. HUPP

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES:

X GC/EC (18) DATE
COMPLETED
3/29/84

QUALITATIVE: YES

SEMIQUANTITATIVE:

QUANTITATIVE: YES

REQUIRED QA/QC:

COMPLETION DATE REQUESTED:

COMMENTS: Results phoned to David Batchelor on 3/15.

SAMPLE DISPOSITION:

X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS:

ANALYSIS REQUEST FILE NAME: AAS130-11

MONS 022809