Monsanto

MONSANTO COMPANY / DAYTON LABORATORY / DAYTON, OHIO 45407

ESC/DAYTON	ANALYTICAL
(Department/Section)	(Type of Report)

REPORT NO.: MDA-519

JOB/PROJECT NO.: 484.2150

DATE: 7 August 1984

PERIOD COVERED:

TITLE: ANALYSES FOR PCB's

AUTHORS: B. M. Hughes

RECEIVED
AUG 1 9 1984
MONSANTO COMPANY
CENTRAL REP. ...
ST. LOUIS

RESTRICTED DISTRIBUTION

ABSTRACT: Three samples from the Trenton Plant were analyzed for their possible PCB concentrations. The results of these analyses are shown in this report.

PERMANENT COPY

TECHNICAL APPROVAL: J J Brooks / HBW
J. J. Brooks
Senior Research Group Leader

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

APPROVAL DATE: 8-8-84

RESTRICTED DISTRIBUTION: (see over)

COMPANY CONFIDENTIAL

This document is the property of Monsanto Company and the recipient is responsible for its safekeeping and disposition. It contains CONFIDENTIAL INFORMATION which must not be reproduced, revealed to unauthorized persons or sent outside Monsanto Company without proper authorization.

REPORT NO.: MDA-519 AUTHORS: B. M. Hughes
COPY NO.: TITLE:

DISTRIBUTION

COPY NUMBER			ABSTRACT ONLY
1	A. Schroeder	1910	B. A. Woltermann
2	B. M. Hughes	1250	
3	R. F. Ivory	1250	
4	Central Files	1250	
5	Technical Reports Library	R2C	

NOTE: Distribution of this report is restricted. Requests for additional copies should be made through the principal contact at the Monsanto location involved or the Manager, Environmental Sciences Center, Dayton Laboratory, Dayton, Ohio.

ACKNOWLEDGEMENT

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

Analyses: B. M. Hughes
D. McKenzie
C. Trang

COMPANY CONFIDENTIAL

ANALYSIS REPORT

DATE: March 30, 1984

LOG NUMBER: 1-84-04-02-01 PROJECT NO: 484.21518

TO: Bob Ivory/Trenton

FROM:

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

Phone No: (513) 268-3411

Title: Analysis of PCBs in Three Samples

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX01

Standard Analysis Method No(s): ECAN02, MSAN04

Final Report File No(s): A21518

Table File No(s): B21518, C21518, D21518, E21518

One soil, one oily deposit and one waste oil drum sample were analyzed for PCBs using the above referenced extraction and analysis methods. Copies of these methods are included for further technical information. Tables 1 - 3 summarize these results and Table 4 summarizes QA/QC results. Note that greater than 50 ug/g total PCBs were detected in the oily deposit. This sample, and duplicate and the soil sample which contains approximately 10 ug/g total PCBs were also analyzed for PCBs using a capillary GC/MS method which monitors selected masses to detect PCB isomers. This latter instrumental technique (summarized in SAM MSAN04) confirmed the high levels of Aroclor 1254 and Aroclor 1260 in the oily deposit. Concentrations of Aroclor 1254 detected in the oily deposit were 223 and 135 ug/g and concentrations of 1260 detected were 205 and 301 ug/g. Mass spectrometric analysis also confirmed the presence of approximately 35 ug/g (4 ug/g Aroclor 1254 and 31 ug/g Aroclor 1260) total PCBs in the soil sample. No PCBs (<1 ug/bottle) were detected in either of the two blank sample bottles.

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

ANALYST APPROVAL: _____

MONS 023042

TABLE 1. Results of Capillary GC/EC Analyses of PCBs in Trenton Ditch Soil Sample

ESC Project No: 484.21510 Date: April 30,1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Trenton Ditch Soil at Water Level CONCENTRATION (ug/g)
Aroclor 1249	ECAN02	0.1	ND
Aroclor 1254	-	-	1.67
Aroclor 1260	-	-	11.14
Total Aroclors	ECAN02	0.1	12.81

SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)	RECOVERY (%)
CL5-benzene	ECAN02	1.0	142

DOCUMENTATION

ESC Extraction Method	PCEX01
Sample Amt extracted (g)	1.1723
Final Extract Volume (ml)	1
Extract Dilution	1:2
ESC Extract No.	PCB393
Process File Name	BP463

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: B21510:MH

MONS 023043

TABLE 2. Results of Capillary QC/EC Analyses of PCBs in Trenton Oily Deposit on Floor Samples

ESC Project No: 484.21518 Date: April 30,1984

	SAMPLE IDENTIFICATION		
	Trenton Oily Deposit on Floor	Trenton Oily Deposit on Floor Duplicate	Trenton Oily Deposit on Floor Method Blank

ANALYTE	ESC ANALYTICAL METHOD	LOD	CONCENTRATION (ug/g)		
Aroclor 1248	ECAN02	0.1	ND	ND	ND
Aroclor 1254	-	-	258	101	-
Aroclor 1260	-	-	163	192	-
Total Aroclors	-	-	421	293	-

SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)	RECOVERY (%)		
CL5-benzene	ECAN02	1.0	-	-	117

DOCUMENTATION

ESC Extraction Method	PCEX01	PCEX01	PCEX01
Sample Amt extracted (g)	1.1092	1.2429	0
Final Extract Volume	1	1	1
Extract Dilution	1:100	1:100	1:1
ESC Extract No.	PCB396	PCB397	PCB399
Process File Name	BP468	BP472	BP471

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: C21518:MH

MONS 023044

TABLE 3. Results of Capillary GC/EC Analyses of PCBs in Trenton Waste Oil Drum Sample

ESC Project No: 484.21518 Date: April 30,1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Trenton Waste Oil Drum CONCENTRATION (ug/g)
Aroclor 1248	ECAN02	10	ND
Aroclor 1254	-	-	-
Aroclor 1260	-	-	-
Total Aroclors	-	-	-

SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)	RECOVERY (%)
CL5-benzene	ECAN02	1.0	102

DOCUMENTATION

ESC Extraction Method	PCEX01
Sample Amt extracted (g)	1.1741
Final Extract Volume (ml)	2
Extract Dilution	1:10
ESC Extract No.	PCB398
Process File Name	BP473

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: D21518

MONS 023045

TABLE 4. QC/QA Results of Capillary GC/EC Analyses of PCBs in Trenton Ditch Soil Samples

ESC Project No: 484.21518 Date: April 30,1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Trenton Ditch Soil at Water Level Low Spike	SAMPLE IDENTIFICATION: Trenton Ditch Soil at Water Level High Spike
			CONCENTRATION (ug/g)	
Aroclor 1248	ECAN02	0.1	1.11	11.7
Aroclor 1254	-	-	2.70	9.24
Aroclor 1260	-	-	5.86	5.76
Total Aroclors (Excluding A1248 in Native Spiking Study)	-	-	8.56	15.0
SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)	RECOVERY (%)	
CL5-benzene	ECAN02	1.0	100	104
Native Spiking Study Aroclor 1248		.74,8.4	150	139
DOCUMENTATION				
ESC Extraction Method			PCEX01	PCEX01
Sample Amt extracted (g)			1.1538	1.0313
Extract Final Volume (ml)			1	1
Extract Dilution			1:2	1:2
ESC Extract No.			PCB394	PCB395
Process File Name			BP465	BP467

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: E21518:MH

MONS 023046

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 "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 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 TDEXO11MH

MONS 023047

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: (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:	TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
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.	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: 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

---

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 1242, 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

ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No: MSAN04

TITLE: Organic Compounds in Solvents

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5985B Capillary GC/MS-Selected Ion Monitoring with Revision-E Multi-drive software.

Typical Run Time:	3 - 40 min.
Typical Multiplier Voltage:	1800 - 2800 eV
Typical Dwell Time:	200 - 500 msec/mass
Maximum # of Masses:	20
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.

CALCULATIONS: Analyte concentration = (Analyte area)/(Analyte RRF)(area of IS)

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)/(conc. of analyte)(area of IS)

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 for-

mulation 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 in the above RRF equation. The RRFs for the corresponding PCB isomer classes are used to calculate total PCB isomer concentrations in unknown samples.

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

DATES OF METHOD CREATION AND METHOD UPDATES: 4/30/84

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

MRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: APRIL 2, 1984 LOG NO: 1-84-04-02-01

REQUESTER: TRENTON/IVORY

PROJECT NO: 484.21518

REPORT RESULTS TO: FILES/MEES,IVORY,BROOKS

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

SAMPLE PICKUP BY: M. HUGHES

SAMPLE LOCATION:

SAMPLE DESCRIPTION FOR 3 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
OILY DEPOSIT - 6S SUBSTATION	1	PCB396,PCB397
DITCH SOIL - PRIOR TO POND #1	1	PCB398
WASTE OIL DRUM ONE HALF FULL	1	PCB398
*PCB SAMPLE BLANK (EMPTY 40ML VIAL)	1	PCB401
*PCB SAMPLE BLANK (EMPTY BOTTLE)	1	PCB400
Method Blank		PCB399

*EMPTY VIAL AND BOTTLE TO BE RINSED AND ANALYZED ONLY IF PCB'S FOUND IN THE FIRST THREE SAMPLES.

RECORDED BY: P. HUPP

REQUIRED ANALYSES: PCB BY GC/EC, GC/MS

ANALYTICAL TECHNIQUES:

	DATE COMPLETED		DATE COMPLETED
X GC/EC (18)	4/30/84	X GC/MS (18)	4/30/84

QUALITATIVE: YES SEMIQUANTITATIVE: QUANTITATIVE: YES

REQUIRED QA/QC:

COMPLETION DATE REQUESTED: DATA NEEDED BY 4-27-84

COMMENTS: GC/EC results phoned to on 4/26/84.

SAMPLE DISPOSITION: X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: Sample may contain PCBs.

ANALYSIS REQUEST FILE NAME: AA2150:LI

MONS 023054

FIELD SAMPLING REPORT
&
CHAIN OF CUSTODY RECORD

Customer: Monsanto Trenton Mi

Plant Location:

Site Description: Phosphates Plant

Date: 3/26/84

Sample	Quantity	Time	By	Comments
1	Waste Oil Sample (1)	1608	MF/ARS	
2	Main Dikh Mud (1)	1625	MF/ARS	
3	65 Flmr Dewit (1)	3/27/84	ARS	
4				
5				
6				
7				
8				
9				
10				
11	Delivered to JD Lane	A R Schmidt	1630 on	3/30/84
12				
Composite				

Dispatched By: J.D.Lane	Date 3/30/84	Time 1630	Received at Lab by:	Date (Y-M-D)	Time 0800
Method of Shipment: Private Auto					
Relinquished By: Signature			Received by: Signature		
Relinquished By: Signature			Received By: Signature		
Relinquished By: Signature			Received By: Signature		

EXHIBIT 1

MONS 023055