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

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TITLE: ANALYSIS OF AROCLORS IN ENVIRONMENTAL SAMPLES

AUTHORS: B. M. Hughes

ABSTRACT: Various environmental samples from the Trenton plant were analyzed for possible Aroclor concentrations. The results of these analyses are shown in this report.

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APPROVAL DATE: 8-13-84

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ABSTRACT ONLY

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ACKNOWLEDGEMENT

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

Analyses: B. M. Hughes
D. McKenzie
B. Williams

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ANALYSIS REPORT

DATE: August 7, 1984

LOG NUMBER: 1-84-04-30-02 PROJECT NO: 484.21618

TO:
Trenton/Ivory

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

Phone No: (513) 268-3411

Title: Analysis of Aroclor Formulations in Environmental Samples

Standard Sampling Method No(s): Samples supplied by customer.

Standard Extraction Method No(s): WSEX05

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A21618

Table File No(s): B21618,C21618

Tables 1 and 2 summarize results of Aroclor analysis in water samples. Please note that no results could be obtained for Aroclor formulations in Thermainol #82332A, Waste Oil Drum #82332B or Crik West Side #82332E due to large interferrences. These latter three samples may be analyzable using capillary GC/MS methods. This can be done at a later date if the Trenton Plant requests these analyses.

Also included are summaries of methods used and analysis request data generated for this project.

ANALYST(S): B. Mason Hughes, David McKenzie and Barbara Williams

ANALYST APPROVAL: B. Mason Hughes

TABLE 1. Results Capillary GC/EC Analyses of Aroclor Formulations in Trenton Samples

ESC Project No: 484.21618 Date: August 3,1984

	SAMPLE IDENTIFICATION	
	Trenton Iqt process water from clarifier low spike	Trenton Iqt process water from Clarifier High spike
ANALYTE / ESC ANALYTICAL METHOD / LOD	CONCENTRATION (ug/l)	
Aroclor 1254 ECAN02 1.0	.90	9.6
Rng of Values - -	9.3	12.7
No of Values - -	4	24
Std Deviation - -	.17	2.2
% Rel Std Dev - -	20	23
SURROGATE SPIKING COMPOUND / ESC ANALYTICAL METHOD / AMOUNT ADDED (ug/l)	RECOVERY (%)	
CL5-benzene ECAN02 1.0	85	86
Native spiking study Aroclor 1254 1.0,9.9	89	97
DOCUMENTATION		
ESC Extraction Method	WSEX05	WSEX05
Sample Amt extracted (l)	0.1	0.1
Extract final volume (ml)	1.0	1.0
Extract Dilution	1:1	1:1
ESC Extract No.	PCB426	PCB427
Process File Name	BP541	BP542

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: B21618:MH

TABLE 2. Results Capillary GC/EC Analyses of Aroclor Formulations in Trenton Samples

ESC Project No: 484.21618 Date: August 3,1984

	SAMPLE IDENTIFICATION		
	Trenton Iqt process water from clarifier @02332C	Trenton Iqt process water @02332C Duplicate	Method Blank

ANALYTE	ESC ANALYTICAL METHOD	LOD	CONCENTRATION (ug/l)		
Aroclor 1254	ECAN02	1.0	ND	ND	ND
Rng of Values	-	-	-	-	-
No of Values	-	-	-	-	-
Std Deviation	-	-	-	-	-
% Rel Std Dev	-	-	-	-	-
SURROGATE SPIKING COMPOUND		**AMOUNT ADDED (ug/l)**	**RECOVERY (%)**		
CLS-benzene	ECAN02	1.0	67	108	64
DOCUMENTATION					
ESC Extraction Method			WSEX05	WSEX05	WSEX05
Sample Amt extracted (l)			0.1	0.1	0.0
Extract Final volume (ml)			1.0	1.0	1.0
Extract Dilution			1:1	1:1	1:1
ESC Extract No.			PCB424	PCB425	PCB428
Data File Name			BP536	BP537	BP439

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: C21618:RH

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 TBEXD1:HH

MONS 023097

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-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: 10/25/83, 2/3/84

ESC Standard Extraction Method (SEM)
TITLE: Wide scan extraction method for water matrix(s).(ces).

ESC Standard Extraction Method No:
WSEX05

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
water	10.0 mL -4 L	10 mL- 400 mL

TYPICAL ANALYTES: Aroclors, PCBs, priority pollutants

ESC Standard Analysis Method Nos:

INTERFERENCES: Various: See individual interferences for individual analytes.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
deuterated surrogates for individual analytes	50-120
pentachlorobenzene	50-120
2,4,6-trichloro-biphenyl	50-120

APPARATUS AND MATERIALS:
Burdick and Jackson methylene chloride
Separatory funnels
Sodium sulfate for extract drying.
Sodium hydroxide and sulfuric acid for pH adjustments.
Nitrogen for extract volume reduction.
Water bath for K-D extract volume reduction

LIMITATIONS: Sufficient sample amount to provide GC-MS LOD.

SAFETY PRECAUTIONS: See Safe Laboratory Practices.

DATES OF METHOD CREATION AND UPDATES: 5/16/84

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

MRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: APRIL 30, 1984 LOG NO: 1-84-04-30-02

REQUESTER: TRENTON/IVORY

PROJECT NO: 484.21618

REPORT RESULTS TO: FILES/MEES,IVORY,BROOKS

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

SAMPLE PICKUP BY: M. HUGHES

SAMPLE LOCATION: LAB 28 BUILDING 2

SAMPLE DESCRIPTION FOR 9 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
20Z THERMINOL #82332A	1	PCB-429,PCB-430 DUP. PCB-431-LS,PCB-432-MS PCB-442,PCB-443 DUP. PCB-444-LS,PCB-445-MS
20Z WASTE OIL DRUM #82332B	1	PCB-433,PCB-434 DUP. PCB-435-LS,PCB-436-MS PCB-446,PCB-447 DUP PCB-448-LS,PCB-449-MS
METHOD BLANK(S)		PCB-441,PCB-450
1 QT PROCESS WATER #82332C	1	PCB-424,PCB-425 DUP. PCB-426-LS,PCB-427-MS
METHOD BLANK		PCB-428
100Z CRIK WEST SIDE #82332E	1	PCB-437,PCB-438 DUP. PCB-439-LS,PCB-440-MS
20Z EMPTY BOTTLE FOR OIL SAMPLES	1	
100Z EMPTY BOTTLE-SAMPLING VESSEL FOR THERMINOL	1	
100Z EMPTY BOTTLE FOR "CREEK" SAMPLE	1	
1 QT EMPTY BOTTLE FOR EFFLUENT SAMPLE	1	
1QT SQUARE EMPTY BOTTLE FOR RESIDUE SAMPLE	1	

RECORDED BY: P. HUPP

REQUIRED ANALYSES: PCB

ANALYTICAL TECHNIQUES:

DATE COMPLETED

MONS 023102

X GC/EC (10) 8/7/84

QUALITATIVE: YES SEMIQUANTITATIVE: QUANTITATIVE: YES

REQUIRED QA/QC: DUPLICATE OF EACH SAMPLE, AND NATIVE SPIKE AT TWO LEVELS

COMPLETION DATE REQUESTED:

COMMENTS: METHOD BLANK PCB-441 AND PCB-450 APPLIES TO THERMINOL, WASTE OIL DRUM AND CRIK WEST SIDE SAMPLES. METHOD BLANK PCB-428 APPLIES TO PROCESS WATER.

SAMPLE DISPOSITION: X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS:

ANALYSIS REQUEST FILE NAME: AA2160:L1

FIELD SAMPLING REPORT
&
CHAIN OF CUSTODY RECORD

[redacted]: TO MONSANTO RESEARCH CORP

ant Location: TRENTON, MI (MONSANTO)

te Description: RESINS PCB INSPECTION

te: 4/27/84

mple	Quantity	Time	By	Comments
[illegible]	2oz Thermimel	4/23/84	ME/ARS	
[illegible]	2oz. Waste Oil	"	"	
[illegible]	1QT. Waste Water	"	"	
[illegible]	10oz. Creek Mud	4/25/84	"	
[illegible]	Empty 2oz bottle		ARS	
[illegible]	" QT " (brown)		"	
[illegible]	" 10oz "		"	
[illegible]				
[illegible]	Empty SQUARE QT BOTTLE (white)		ARS	
10				

spached By:	Date	Time	Received at Lab by:	Date	Time
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ANALYSIS OF FOOD AND WATER

EXHIBIT I

MONS 023104