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REPORT NO.: MSL-5404
ESC-UAG-86-09

JOB/PROJECT NO.: 5672

DATE: January 27, 1986

TITLE: ANALYSIS OF WORLD HEADQUARTERS WASTEWATER SAMPLES FOR PCBs.

AUTHORS: B.M. Hughes, D. McKenzie, C.K. Trang

ABSTRACT: Samples of wastewater from Monsanto World Headquarters were analyzed for polychlorinated biphenyls. The analyses were done by gas chromatography with electron capture detection. Approximately 0.4 ppm of Aroclor 1248 was detected in both samples.

TECHNICAL APPROVAL: Paul L. Sherman
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Quality Assurance Specialist

APPROVAL DATE: 2-12-86 MONS 023897

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REPT. NO.: ESC-UAG-86-09 AUTHORS: B. M. Hughes, D. McKenzie and C. K. Trang
TITLE: ANALYSIS OF WORLD HEADQUARTERS WASTEWATER SAMPLES FOR PCBs
COPY NO.:

R-1068(A)

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ANALYSIS REPORT

DATE: January 27, 1986

LOG NUMBER: U-85-12-31-01 PROJECT NO: 760.28-5672

TO: Mike Arnold (R3B)

FROM:

Environmental Sciences Center
Monsanto Company
800 N. Lindbergh Blvd.
St. Louis, Mo. 63167

Phone No: (314) 694-1464

Title: Analysis of Aroclor Formulations in Two Water Samples

ABSTRACT

Two water samples were analyzed for Aroclor formulations. A trace of Aroclor 1248 was detected in both samples.

INTRODUCTION

Two water samples were analyzed for Aroclor formulations using liquid-liquid partition extraction techniques and capillary GC/EC analysis techniques.

SUMMARY

Table 1 summarizes analytical results for the analysis of Aroclor formulations in two water samples. Please note that approximately 0.4 ug/g Aroclor 1248 was detected in both samples.

QC RESULTS

Tables 1 and 2 summarize QC results for this project. The duplicate values measured for Aroclor 1248 in the sample labelled CC NBP 3333211 give some indication of the precision of the determination. In addition, the low spike and high spike values for Aroclor 1248 reported in Table 2 show that good recoveries are measured for Aroclor 1248 in the 0.1 to 1.0 ug/g concentration range.

DETAILS

Standard Sampling Method No(s): N.A.

Standard Extraction Method No(s): WSEX05 (Also see Flow Sheet WSFS05.)

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A67218, B67218, C67218

Table File No(s):

Also included in this report are:

1) Brief descriptions of extraction and analysis methods used.

2) Sample custody information recorded at login.

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

ANALYST APPROVAL: B. Mason Hughes

TABLE 1. Summary of PCB Concentrations in Water Samples.

ESC Project No: 760.28 - 67218 Date: January 14, 1985

Sample I.D.	Surrogate Recovaries(%Recovery)		PCB Concentration (ug/g)			
	CL5-banzene	2,4,6-CL3-biphanyl	A1242	A1248	A1254	Total
CC NBP 3333211	121	140	ND(a)	0.31	ND(a)	0.31
CC NBP 3333211 (DUP.)	76	83	"	0.46	"	0.46
Chesterfield NBP 3333211	114	136	"	0.42	"	0.42
Method Blank	51	70	"	ND(a)	"	ND(a)

Footnotes:

a. ND= None Detected (<.01 ug/g)

TABLE FILE NO: B67218:MH

TABLE 2. Summary of Aroclor 1248 Spiking Concentrations and Recoveries in Quality Control Experiments for Water Samples.

ESC Project No: 760.28 - 67218 Date: January 14, 1986

Sample I.D.	Surrogate Recoveries (%Recovery)		Aroclor 1248	
	CL5-benzene	2,4,6-CL3-Biphenyl	Amount Added (ug/g)	Recovery (%)
B&J Water + L. Spike	42	60	.085	117
B&J Water + H. Spike	32	52	.770	111

TABLE FILE NO: C67218:MH

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

FLOW SHEET WSFS05
FOR ESC EXTRACTION METHOD WSEX05

SAMPLE ID: ..... MRC EXTRACT #.....

..... LOG NO: .....

EXTRACTION DATE and TIME: .....

RECORD NATIVE pH ..... WAS pH ADJSTED YES/NO IF YES TO WHAT pH.....

PLACE SAMPLE INTO AN APPROPRIATELY SIZED SEPERATORY FUNNEL (1/2 HEAD SPACE SUGGESTED FOR ADEQUATE MIXING) [ ]

SAMPLE VOLUME .....

SURROGATE SPIKE

.....

.....

[ ] .....

IF pH ADJUSTED ( ) ADJUST WITH,50% NaOH OR H_2SO_4

ADD METHYLENE CHLORIDE IN THE APPROPRIATE AMOUNT (1 L SAMPLE 30 mL SOLVENT, 100 mL SAMPLE 15 mL SOLVENT) [ ]

RECORD AMOUNT METHYLENE CHLORIDE ADDED FOR EACH EXTRACTION

1).....

2).....

3).....

SHAKE SAMPLE ONE TIME AND VENT THROUGH STOPCOCK REPEAT DOUBLING THE NUMBER OF SHAKES BETWEEN VENTING SAMPLES UNTIL THE NUMBER OF SHAKES EXCEED 80 [ ]

OBSERVATIONS

.....

.....

.....

.....

ALLOW PHASES TO SEPARATE [ ]

REPEAT EXTRACTION PROCEDURE TWO MORE TIMES [ ] [ ]

IF ACID OR BASE WASHED RECORD TYPE AND AMOUNT. [ ]

ACID/BASE WASH COMENTS

.....

DRY EXTRACT WITH 4 INCH SODIUM SULFATE COLUMN (COLUMN PRERINSED WITH 30 mL METHYLENE CHLORIDE) [ ]

.....

[ ] EXTRACT DRYING NOT REQUIRED

RINSE SODIUM SULFATE COLUMN WITH 2 X 30 mL [ ]

REDUCE EXTRACT VOLUME TO 1 mL BY K-D TECHNIQUE AND OR NITROGEN SOLVENT VOLUME REDUCTION [ ]

[ ] TRANSFER EXTRACT INTO VIAL FOR ANALYSIS

EXTRACT NO: ..... DATE/TIME COMPLETED...../.....

ANALYST: ..... ESC JOB NO: .....

MONS 023904

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

ESC Standard Extraction Method No: WSEX05

MATRIX:	TYPICAL SAMPLE SIZE (Liters)	TYPICAL EXTRACT VOLUME (ml)
water	0.01 - 4	1 - 40

TYPICAL ANALYTES: Aroclors, PCBs, priority pollutants

ESC Standard Analysis Method Nos: ECANxx and MSANxx

INTERFERENCES: Various; See individual interferances for individual analytes.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
phenol-d5	20-80
CL-phenol-d4	20-80
CL2-benzene-d4	50-120
naphthalene-d8	50-120
pyrene-d10	50-120
chrysene-d12	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 and K-D apparatus for 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, 10/5/84, 12/7/84

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

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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 RRF)(isomer area/internal standard area)

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.

DATE OF METHOD CREATION: 10/25/83, 2/3/84 Time Stamp - <860127.1257>

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

Time Stamp - <860127.1253>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 12/31/85 LOG NO: U-85-12-31-01

REQUESTER: Mike Arnold PROJECT NO: 760.28 - 672

REPORT RESULTS TO: FILES/MEES, Sherman, Hughes, McKenzie, Trang

ANALYSIS REQUESTS SENT TO: FILES, Hughes, McKenzie, Trang, Sherman

SAMPLE PICKUP BY: McKenzie

SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 2 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
Monsanto Company Creve Couer Dilutiuon Basin West Pump, NBP 3333211	1 X 40 mL	PCB-1502 PCB-1503-DUP
Monsanto Company Chesterfield Village Dilution Basin - Outfall NBP 3333211	1 X 40 mL	PCB-1504
Burdick and Jackson Water Blank		PCB-1505
Burdick and Jackson Water Blank Plus low level native spike.		PCB-1506
Burdick and Jackson water Blank Plus high level native spike.		PCB-1507

RECORDED BY: McKenzie

REQUIRED ANALYSES: PCBs

ANALYTICAL TECHNIQUES:

TECHNIQUE (EM)	DATE COMPLETED
x GC/EC (18)	1/27/86

QUALITATIVE: x SEMIQUANTITATIVE: QUANTITATIVE: x

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

-- HOLD FOR 30 DAYS

MONS 023908

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA672:11

MONS 023909