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ESC/DAYTON
(Department/Section)

ANALYTICAL
(Type of Report)

REPORT NO.: MDA-576

JOB/PROJECT NO.: 506

DATE: 4 February 1985

PERIOD COVERED:

TITLE: ANALYSIS OF SOLID SAMPLES FOR AROCLOR FORMULATIONS

AUTHORS: B. N. Hughes

ABSTRACT: Solid samples received from the Port Plastic Plant were analyzed for possible concentrations of Aroclor formulations. The results of these analyses are shown in this report.

TECHNICAL APPROVAL: [signature]
P. L. Sherman
Sr. Research Group Leader

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

APPROVAL DATE: 2-5-85

RESTRICTED DISTRIBUTION: (see over)

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REPORT NO.: MDA-576
AUTHORS: B. N. Hughes
TITLE: ANALYSIS OF SOLID SAMPLES FOR AROCLOR FORMULATIONS
COPY NO.: 5

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H. A. Woltermann

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ACKNOWLEDGEMENT

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

Analyses: B. M. Hughes
C. K. Trang

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ACKNOWLEDGEMENT

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

Analyses: B. M. Hughes
C. K. Trang

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Time Stamp - <850201.1135>

ANALYSIS REPORT

DATE: February 1, 1985

LOG NUMBER: 1-85-01-08-01 PROJECT NO: 760.18-506

TO:

Port Plastics/Ivory

FROM:

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

Phone No: (513) 268-3411

Title: Analysis of Five Solid Samples for Aroclor Formulations

Standard Sampling Method No(s): Samples Supplied by Customer.

Standard Extraction Method No(s): PCEX01

Standard Analysis Method No(s): ECAM02, MSAM06

Final Report File No(s): A50618

Table File No(s): none

Five samples from the Port Plastics Plant were analyzed for Aroclor formulations using the above referenced Standard Extraction and Analysis Methods. Sample 010709 contained large amounts of Aroclor 1260. Two aliquots of the sample were extracted. Duplicate analyses of the first extract, using ECAM02 Standard Analysis Method (shown in a later section) produced values of 239,000 and 226,000 ug/g Aroclor 1260 and the second extract produced a value of 162,000 ug/g Aroclor 1260. The good agreement of the first pair of values shows the analytical precision of this determination, and the less good agreement of the third value with the first two values is possibly due to sample inhomogeneities.

No Aroclor formulations were detected in the remaining four samples above a concentration of 10 ug/g. In order to lower this level of detection, a capillary GC/MS analysis method (MSAM06) was used. This method is also shown in a later section. The raw data from these four analyses, along with method blank analysis and Aroclor 1254 spiking analyses are shown in later sections of this report. These data show that there is less than 3 ug/g Aroclor formulations in any of the remaining four samples (010710, 010711,

MONS 023303

010712, and 010713).

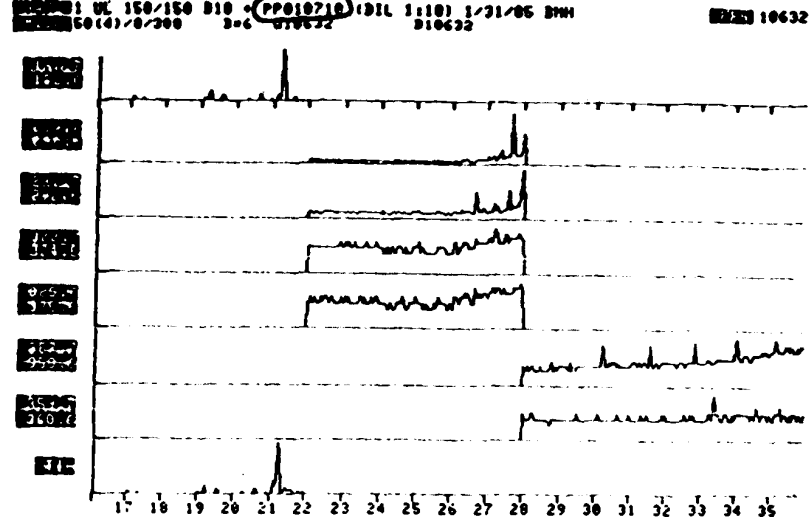
If there are any questions concerning these analyses, do not hesitate to call.

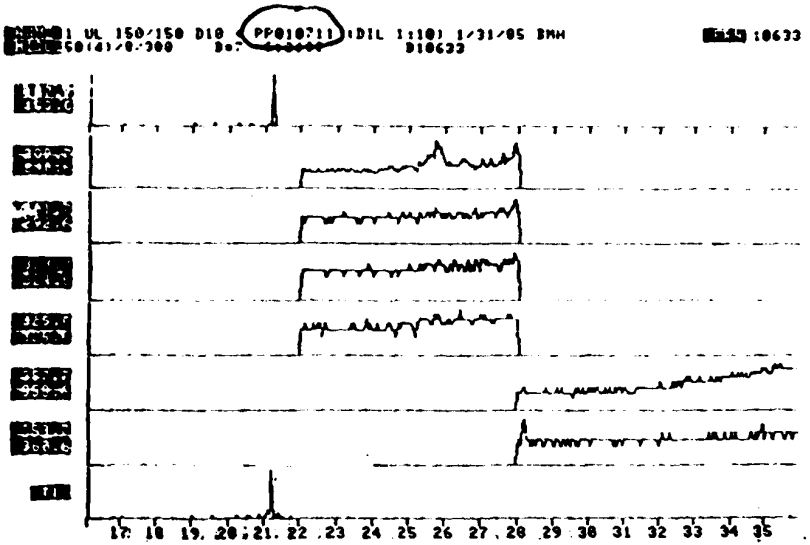
ANALYST(S): B. Mason Hughes and Chi K. Trang

ANALYST APPROVAL: B. Mason Hughes

MONS 023304

The following pages show calculations or raw data documentation of reported results.


PP010710
(DIL 1:10) 1/31/05 BMH
10632
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35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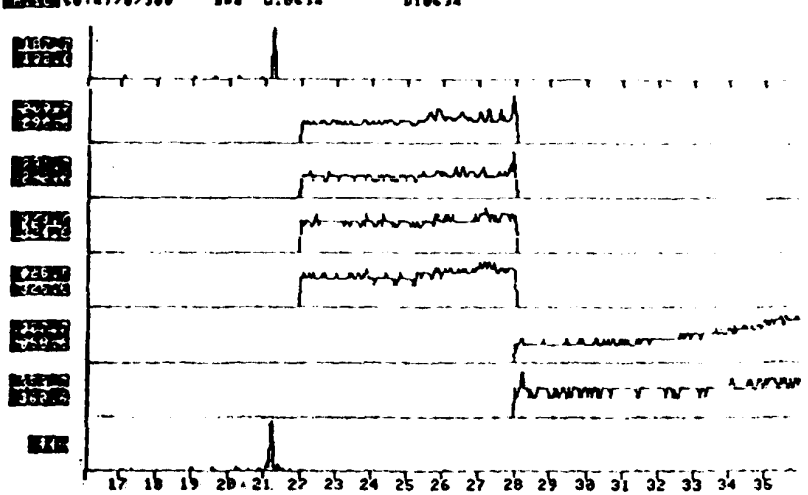
(DIL 1:10) 1/31/05 BMH
10633
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35

MONS 023306


PP010712
DIL 1:10) 1/31/85 BMH
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35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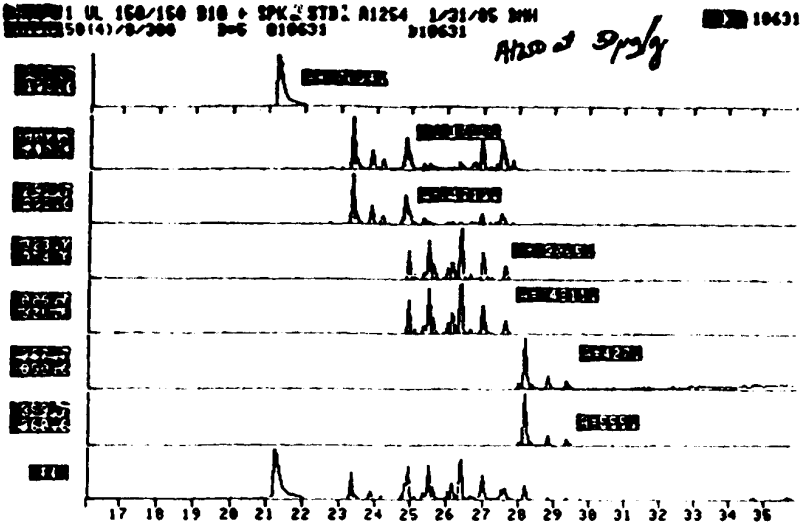
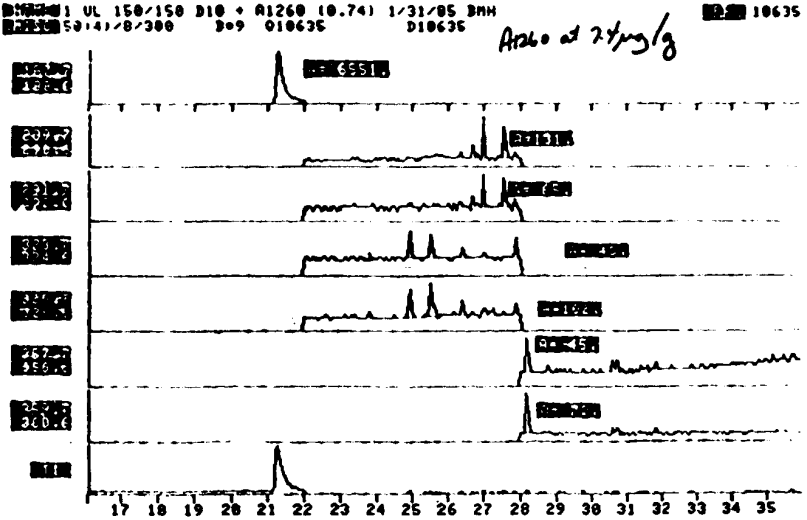
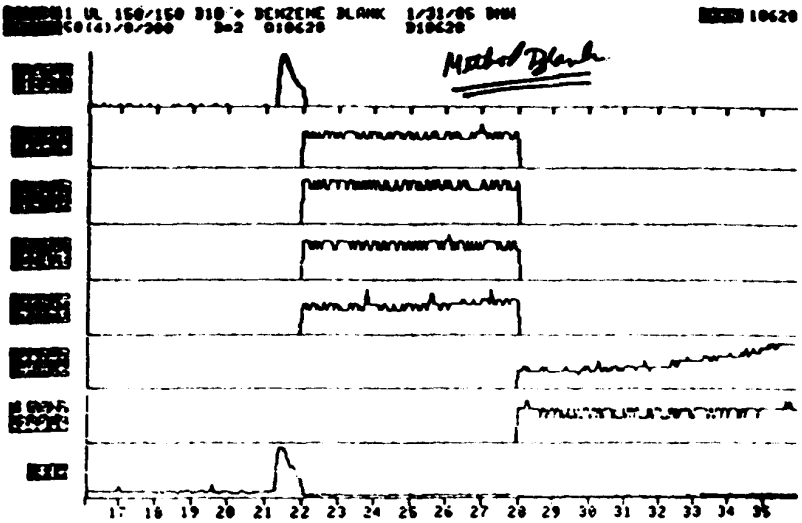

1 UL 150/150 D10 + SPK STD A1254 1/31/85 BMH
50(4)/8/300 D=6 Q10631 D10631
10631
A1250 at 50µg/g
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35

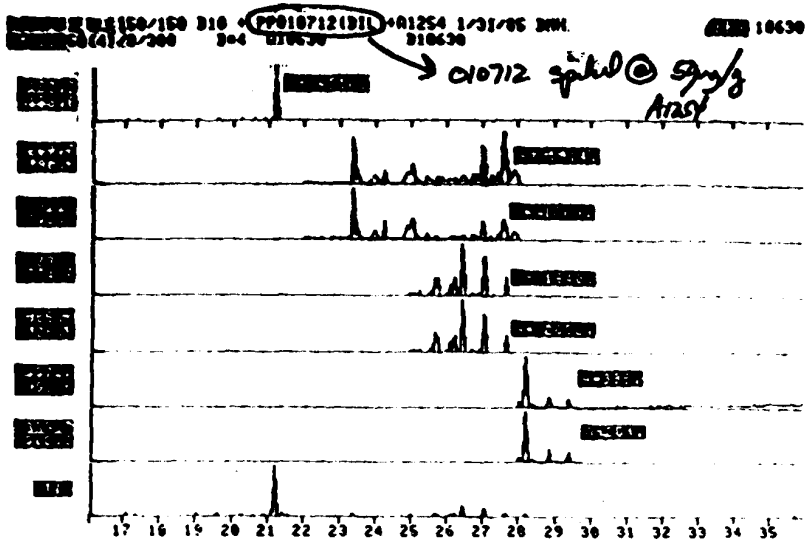

1 UL 150/150 D10 + A1260 (0.74) 1/31/85 BMH
50(4)/8/300 D=9 Q10635 D10635
10635
A1260 at 74µg/g
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35

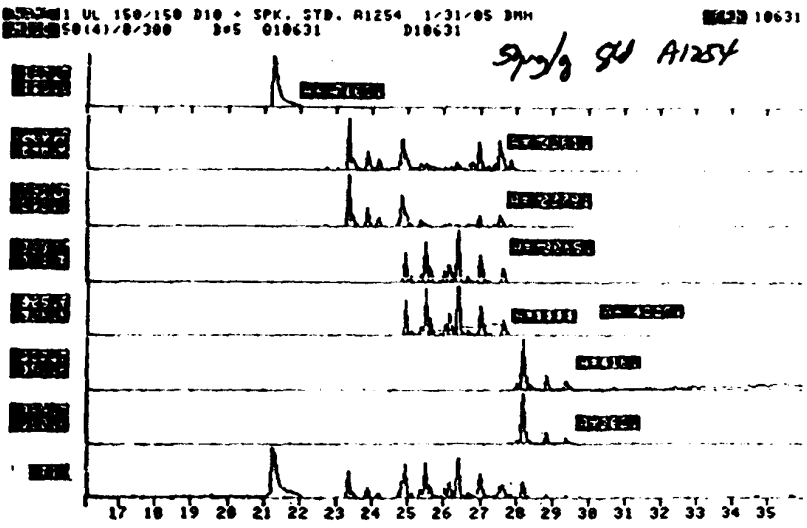
MONS 023308

The following pages show QA/QC data for selected samples.



MONS 023310


150/150 D10 + PP010712(D1L) +A1254 1/31/05 DHH
50(4)/0/300 D=4 D10630 D10630
10630
010712 spiked @ 5ppm/g
Aroclor
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35


UL 150/150 D10 + SPK. STD. A1254 1/31/05 DHH
50(4)/0/300 D=5 010631 D10631
10631
5ppm/g QC A1254
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35

MONS 023311

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: PCEM01

MATRI(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: ECAM01, ECAM02, HSAM03

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers for ECAM01 and ECAM02. Any species in the appropriate retention time region which give the same relative characteristic mass responses as PCB isomers for HSAM03.	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.
Ultrasonic bath to agitate sample.
Centrifuge to settle fine particles.
Disposable glassware.
(See flow sheet for this extraction method in PCFS01.)

LIMITATIONS: Method applies only to matrices which contain 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, 9/25/84

FLOW SHEET PCF001

FOR ESC EXTRACTION METHOD PCE001

SAMPLE ID: ---------- NRC EXTRACT # ----------

---------- LOG NO: ----------

EXTRACTION DATE and TIME: ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL [ ]

SAMPLE WEIGHT ----------

SURROGATE SPIKE OF SAMPLE [ ]

ADD 10 mL SOLVENT TO SAMPLE [ ]

CIRCLE SOLVENT USED : BENZENE

METHYLENE CHLORIDE

HEXANE

ULTRASONICATE FOR 1 HR IN A SOUND BATH. [ ]

CENTRIFUGE DOWN PARTICULATE MATTER IN SAMPLE. [ ]

REMOVE 1 mL AND PLACE IN 1 1/2 mL VIAL FOR ANALYSIS. [ ]

EXTRACT NO: ----------

DATE/TIME COMPLETED:----------/----------

ESC JOB NO: ----------

ANALYST: ----------

MONS 023314

ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No:
TITLE: Aroclor Formulations in Solvents — ECAM02

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 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

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: 10/23/83, 2/3/84 Time Stamp - <850201.1123>

RSC Standard Analysis Method (SAM) **RSC Standard Analysis Method No:**
TITLE: Aroclor Screen **RSAM04**

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:	1900 - 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, two characteristic masses which are monitored are the molecular ion containing all 35-CL isotopes and the molecular ion containing one 37-CL isotope. The following summarizes the masses and approximate retention time regions monitored for Aroclor formulation screening.

ANALYSIS #1

Analyte	Masses	Retention Times (minutes)
CL-BP isomers, anthracene-d10	188,190	16 - 22
CL3-BP isomers	222,224	16 - 22
CL4-BP isomers	289.8,291.8	22 - 28
CL5-BP isomers	323.9,325.9	22 - 28
CL6-BP isomers	357.8,359.8	28 - 36
CL7-BP isomers	391.8,393.8	28 - 36

CALCULATIONS: Analyte concentration = (Analyte area)/[(Analyte RRF)(IS area)]

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)/[(analyte conc.)(IS area)]

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 formulation 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 for each chlorine class in the above RRF equation. The RRFs for the corresponding PCB isomer classes are used to calculate total PCB isomer concentrations of each class in unknown samples.

NOTE: MSAM03, which monitors all molecular ions for all PCB congeners must be used if no Aroclor formulation can be identified from this analysis.

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

DATES OF METHOD CREATION/UPDATES: 1/23/85 Time Stamp - <850129.1257>

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

Time Stamp - <850201.1142>

MONSANTO COMPANY/DAYTON LABORATORY

ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 1/8/85 LOG NO: 01-85-01-08-01

REQUESTER: PORT PLASTIC PROJECT NO: 760.18-506

REPORT RESULTS TO: FILES/HEES,IVORY,SHERMAN

ANALYSIS REQUESTS SENT TO: FILES/HEES,IVORY,HUGHES,SHERMAN

SAMPLE PICKUP BY: CHI TRANG

SAMPLE LOCATION: LAB 21,BUILDING 1

SAMPLE DESCRIPTION FOR 5 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
#010709	1	1.1262 g in 9.0 ml of Benzene
#010709(DUP)		0.1177 g in 9.0 ml of Benzene
#010710	1	1.1144g in 9.0 ml of Benzene
#010711	1	1.0800 g in 9.0 ml of Benzene
#010712	1	1.1614 g in 9.0 ml of Benzene The above sample was spiked with A1254(0.1 ml A1254 (59PPM)+0.9 ml of sample).
#010712(DUP)		0.0741 g in 9.0 ml of Benzene
#010713	1	1.1320g in 9.0 ml of Benzene The above sample was spiked with A1254(0.1ml A1254 (59PPM)+0.9ml of sample).

MONS 023319

Method Blank

150 ul Anthracene
Dilute 150ul Benzene

RECORDED BY: B. F. IVORY

REQUIRED ANALYSES: PCBs

ANALYTICAL TECHNIQUES: GC/EC,GC/MS

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: HOLD FOR 30 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA506:LI

The following pages show chain-of-custody information for this project.

ANALYSIS REQUEST | DATE: 11-8-84 | LOG NO. 01-85-01-08-01

MONSANTO RESEARCH CORPORATION, DAYTON LABORATORY | REQUESTER** Paul Plotts | CHARGE NO.** 5016

SAMPLE DESCRIPTION | NUMBER OF BOTTLES: 5 | NUMBER OF SAMPLES = 5

- 010709 1.1262
- 010710 1.1144
- 010711 1.0800
- → 010712 1.1614
- 010713 1.1320

REPORT RESULTS TO** FILES/ANALYSE: Moss, Ivey, Skinner

ANALYSIS REQUEST TO FILES: Hughes, Ivey, Skinner

SAMPLES FOR: Hughes

SAMPLE LOCATION: Log 21

RECORDED BY:

REQUIRED ANALYSIS**

C PCB

KNOWN HEALTH HAZARD PRESENTED BY SAMPLE: **

REFERENCE TO PREVIOUS WORK (REQUEST #, JOURNAL, ETC.) **

COMPLETION DATE

ANALYTICAL TECHNIQUE **

- ☐ GAS CHROMATOGRAPHY
- ☐ ULTRAVIOLET
- ☐ VISIBLE
- ☐ INFRARED
- ☐ EMISSION
- ☐ EDAX XRF
- ☐ MICROSCOPY
- ☐ OTHER ____
- ☐ NMR
- ☐ HPLC
- ☐ XRD
- ☐ ICP
- ☐ ATOMIC ABSORPTION
- ☐ MASS SPEC
- ☐ GC/MS
- ☐ AUTO ANALYZER

QUAL. ____ SEMIQUANT. ____ QUANT. ____

TIME ALLOTED FOR ANALYSES

ANALYST	HOURS	INST. TIME

REQUIRED QA/QC

**SAMPLE DISPOSAL: WITH REPORT COMPLETE ☐ DISCARD ☐ RETURN HOLD FOR ☒ 30 DAYS ☐ 60 DAYS ___ THEREAFTER

THIS INFORMATION MUST BE SUPPLIED BY ANALYSIS REQUEST ORIGINATOR

MONS 023322

$500

MONSANTO

INDUSTRIAL HYGIENE LAB
Addyston, Ohio

SAMPLE INFORMATION

DATE January 8, 1985

LABORATORY (Outside) MONSANTO RESEARCH CORPORATION
1515 Nicholas Rd. Box 8 Station B
Dayton, OH 45407
Attn: Bob Ivory

SAMPLE #	VOLUME	SUBSTANCE
010709 Bldg 1 Transformer Drips	NA	PCB
010710 Bldg 30 Therminol	NA	PCB
010711 Dirt DN Therminol Furnace	NA	PCB
010712 Dirt DN Therminol Furnace	NA	PCB
010713 Dirt DN Therminol Furnace	NA	PCB

Charge: 914.37-692,JE Transaction

SIGNATURE [signature]
Se Tordah

(21) 552-0
2358

MONS 023323