Monsanto

MONSANTO COMPANY / DAYTON LABORATORY / DAYTON, OHIO 45407

ESC/DAYTON (Department/Section)

ANALYTICAL (Type of Report)

REPORT NO.: MDA-571

JOB/PROJECT NO.: 484.2320 (309)

DATE: 30 January 1985

PERIOD COVERED:

TITLE: ANALYSIS OF THERMINOL SAMPLES FOR AROCLOR FORMULATIONS

AUTHORS: B. M. Hughes, M. Holliday

ABSTRACT: Seven therminol samples were screened for possible concentrations of Aroclor formulations. One sample was further analyzed by GC MS to determine type and concentration of a detected aroclor. The results of these analyses are shown in this report.

TECHNICAL APPROVAL: P. L. Sherman
Sr. Group Leader

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

APPROVAL DATE: 1-30-85

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.

AUTHORS: B. M. Hughes and M. Holliday
TITLE: ANALYSIS OF THERMINOL SAMPLES FOR AROCLOR FORMULATIONS
REPORT NO.: MDA-571
COPY NO.: 6

DISTRIBUTION

COPY NUMBER

1. M. J. Horner - Krumrich Plant
2. B. M. Hughes
3. R. T. Ivory
4. M. Holliday
5. Technical Reports Library - RIC
6. Central Files

ABSTRACT ONLY

B. A. Woltermann

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: M. Holliday
B. M. Hughes
C. K. Trane

COMPANY CONFIDENTIAL

ANALYSIS REPORT

SAMPLE I.D. 389 LOG NO. 1-84-12-10-01

TITLE: PCB SCREEN OF THERMINOL OILS

TO: H.J. HORNER

SUMMARY

Seven (7) samples from the Krumrich were screened for their possible concentrations of Aroclor formulations. The samples were analyzed in accordance with MAC Method 08-6C-R1. The samples were screened for Aroclors 1232, 1242, 1254, 1248 & 1260. The samples' chromatograms were compared to the chromatograms of standards of these Aroclors, run under similar chromatographic conditions for identification and quantitation. The method has a lower detection limit of 0.1 ug/g (ppm) in oil/hexane. The results of these analyses are shown in the following tables.

RESULTS

SAMPLE I.D.	Aroclor	PCB LEVEL ug/g (ppm)
245 Therminol -66	-	ND (a)
244 Therminol	-	ND
255 Therminol from 82 Luwa	-	ND
247 Therminol	-	ND
255 Therminol from 81 Luwa	-	ND
219 HB42 Therminol	-	ND
718 Used Therminol-60	-	(b)

(a) ND= nondetected above the lower detection limit

(b) This sample showed the presence of electron capturing species which did not align with the screening standards. To confirm the presence or absence of Aroclor formulations this sample was further analyzed by SC/MS. The data for this analysis is shown in the remainder of this report.

QUALITY CONTROL DATA

SPIKES

SAMPLE I.D.	AROCLOR SPIKE	% RECOVERY
244 Therminol	12 ul of Aroclor 1254	95 (c)

(c) Average of duplicate samples

Time Stamp - (850129.1157)

ANALYSIS REPORT

DATE: January 29, 1985

LOG NUMBER: 1-84-12-10-01 PROJECT NO: 760.18-309

TO:
Krumerich/Ivory

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

Phone No: (513) 268-3411

Title: Analysis of 710 Used Therminol-60 (12-4-84) for Aroclor Formulations.

Standard Sampling Method No(s): Sample supplied by Krumerich Plant.

Standard Extraction Method No(s): PCEX01 (See PCFS01 for flow sheet).

Standard Analysis Method No(s): MSAN06

Final Report File No(s): A30918

Table File No(s): none

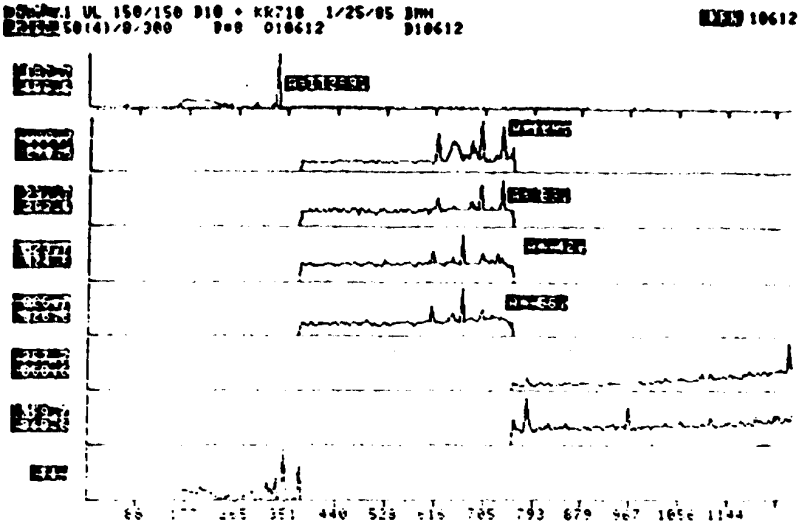
One sample of 710 Used Therminal-60 (12-4-84) was analyzed for Aroclor formulations. Capillary GC/MS analysis techniques showed that this sample contains approximately 3 ug/g Aroclor 1260 or 1262. This report also shows:

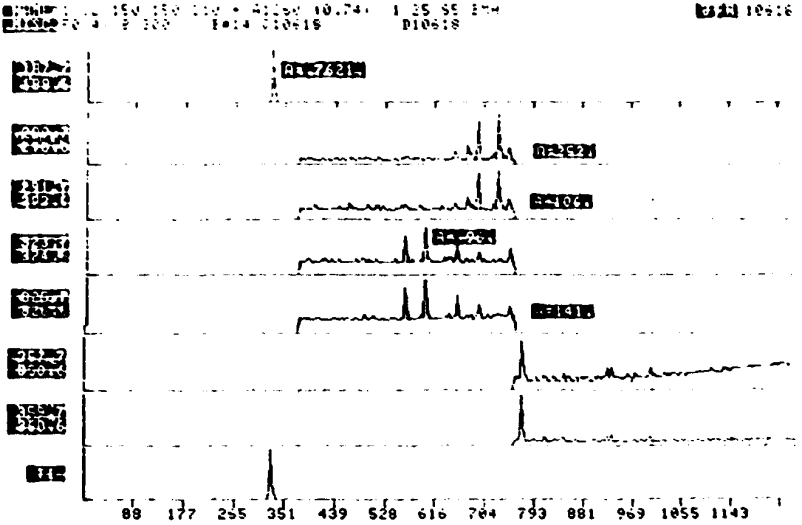
1) Capillary GC/MS chromatograms obtained from extract and standard analyses.
2) Capillary GC/MS chromatograms obtained from the analysis of the sample spiked with 50 ug/g Aroclor 1254.
3) Standard Extraction and Analysis Methods used.
4) Sample login information.

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

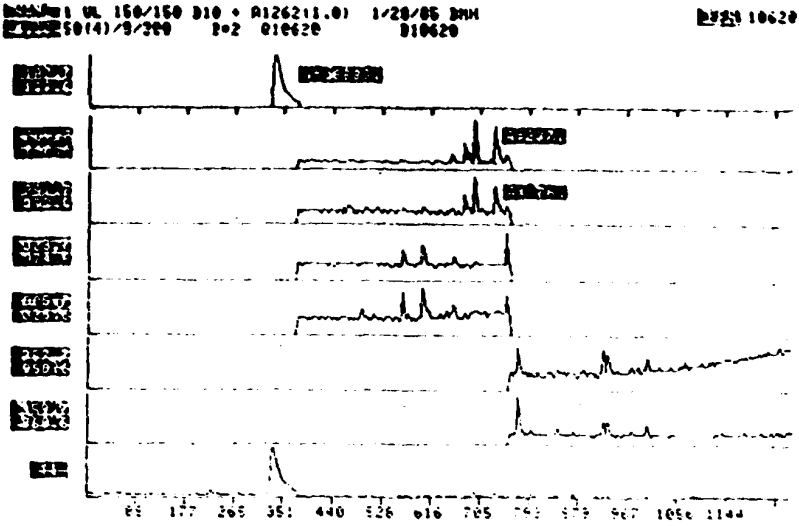
ANALYST APPROVAL: B. Mason Hughes

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


88 177 265 351 440 528 616 705 793 879 967 1056 1144

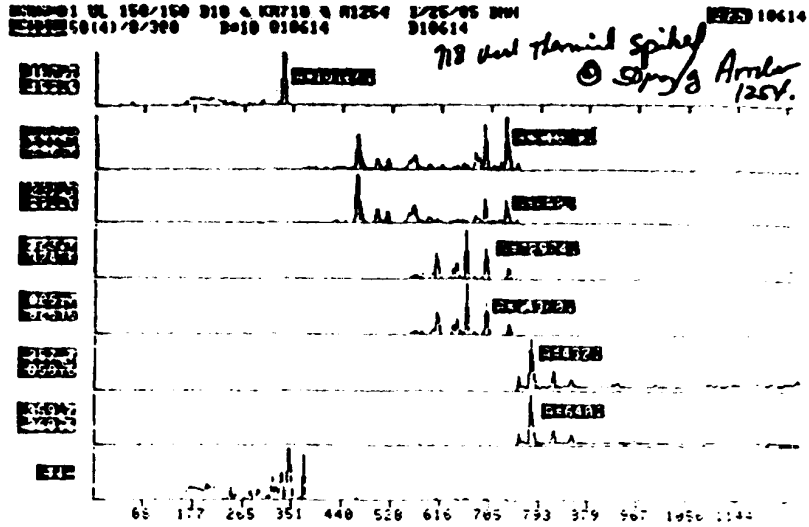

88 177 265 351 439 528 616 704 793 881 969 1055 1143

MONS 023288

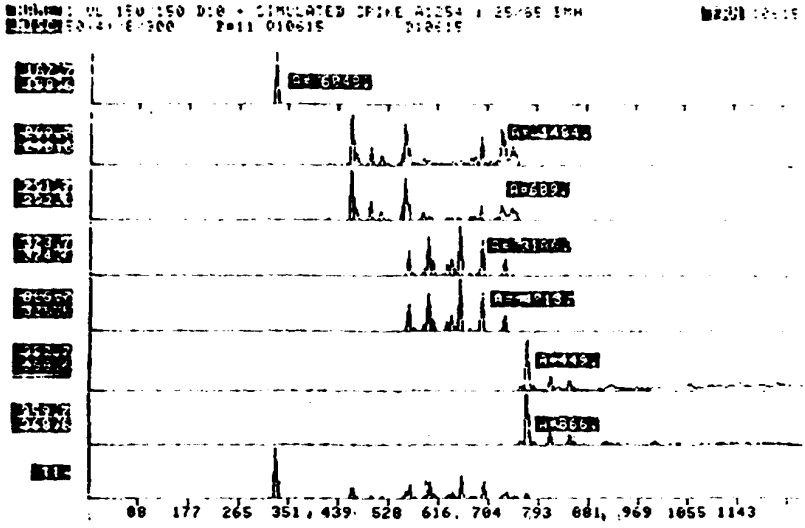

1 UL 150/150 D10 + A1262(1.0) 1/28/85 DMH
50(4)/9/300 B=2 010620 D10620
10620
177 265 351 440 526 616 705 1144

MONS 023289

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



1 UL 150/150 D10 & KR710 & A1254 1/25/85 BMH
50(4)/8/300 D=10 010614 D10614
10614
718 used thermal spike
@ 50μg/g Arrow 1254.
88 177 265 351 440 528 616 705 793 879 967 1056 1144



UL 150/150 D10 + SIMULATED SPIKE A1254 1/25/85 BMH
50(4)/8/300 D=11 010615 D10615
A=6048.
A=1484.
A=689.
A=449.
A=886.
88 177 265 351 439 528 616 704 793 881 969 1055 1143

MONS 023291

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 hydralizable 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

MONS 023293

FLOW SHEET PCP501

FOR ESC EXTRACTION METHOD PCE301

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 023294

ESC Standard Analysis Method (SAM)
TITLE: Aroclor Screen

ESC Standard Analysis Method No:
RSAM06

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 - 2000 eV
Typical Dwell Times:	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. 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
CL2-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: HSAN03, which monitors all molecular ions for all PCB conganers 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 - <850129.1136>

MONSANTO COMPANY/DAYTON LABORATORY

ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 12/10/84 LOG NO: 1-84-12-10-01

REQUESTER: KRUMRICH PROJECT NO: 760.10-309

REPORT RESULTS TO: FILES/HEES,IVORY

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

SAMPLE PICKUP BY: CHI TRANG

SAMPLE LOCATION: LAB 21,BUILDING 1

SAMPLE DESCRIPTION FOR 1 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
710 Used Therminol-60 (12-4-84)	1	1.1364g in 9.0ml of Benzene The above sample was spiked with A1254(59.09PM) (0.1ml A1254+ 0.9ml sample).

RECORDED BY: R. 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: .AA309:LI

ANALYSIS REQUEST | DATE [illegible] | LOG NO. [illegible]

MONSANTO RESEARCH CORPORATION
DAYTON LABORATORY

REQUESTER** [illegible] | CHARGE NO.** [illegible]

REPORT RESULTS TO** FILES/KOP BK [illegible]

SAMPLE DESCRIPTION | NUMBER OF BOTTLES: 1 | NUMBER OF SAMPLES ** 1

[illegible]

ANALYSIS REQUEST TO FILES [illegible]

SAMPLES FOR [illegible]

SAMPLE LOCATION [illegible]

RECORDED BY: [illegible]

REQUIRED ANALYSIS** [illegible]

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

ANALYSI	HOURS	INSTI TIME

REQUIRED QA/QC

**SAMPLE DISPOSAL: AFTER REPORT COMPLETE ☐ DISCARD ☒ RETURN HOLD FOR ☐ 30 DAYS ☐ 60 DAYS _____ OTHER

MONS 023299