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ANALYTICAL
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1984
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REPORT NO.: MDA - 454

JOB/PROJECT NO.: 484.2020

DATE: 22 February 1984

PERIOD COVERED:

TITLE: ANALYSIS OF OIL FOR PCB

AUTHORS: B. M. Hughes

ABSTRACT: A sample of oil from the Queeny Plant was analyzed for its possible PCB concentration. The result of this analysis is shown in this report.

Technical Approval: Joseph J. Brooks

J. J. Brooks
Research Group Leader

Approved By: R. F. Ivory

R. F. Ivory
Environmental Sciences Center
Service Project Manager

Approval Date: 2-27-84

REPORT NO.: MDA - 454 AUTHORS: B. M. Hughes
COPY NO.: 5 TITLE: ANALYSIS OF OIL FOR PCB

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ACKNOWLEDGEMENT

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

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

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ANALYSIS REPORT

DATE: February 14, 1984

LOG NUMBER: 1-84-01-25-02 PROJECT NO: 484.20218

TO:
St. Louis (Queeny)/Ivor:

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

Phone No: (513) 268-3411

Title: Analysis of Fluids for PCBs.

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX03

Standard Extraction Method Flow Sheet(s): PCFS03

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A20218

Table File No(s): B20218

One heat transfer oil was analyzed for PCBs. Results are given in Table 1. Brief descriptions are given for the ESC Extraction and Analytical Methods used for this analysis. In addition a description of table format is supplied.

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

ANALYST APPROVAL: B. M. Hughes

TABLE 1. Results of Capillary GC/EC Analyses of PCBs in Oil Samples

ESC Project No: 483.20218 Date: February 14, 1984

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Heat Transfer Oil WW-B S-771 CONCENTRATION (ug/g)	SAMPLE IDENTIFICATION: Method Blank CONCENTRATION (ug/g)
Aroclor 1248	ECAN02	5	ND	ND
Aroclor 1254	ECAN02	5	ND	ND
Aroclor 1260	ECAN02	5	ND	ND
Total Aroclors	-	5	ND	ND

SURROGATE SPIKING COMPOUND	AMOUNT ADDED (ug/g)	RECOVERY (%)	
Pentachlorobenzene	1.0	184	125

DOCUMENTATION

ESC Extraction Method	PCEX03	PCEX03
Sample Amt Extracted (g)	1.0075	0
Extract Final Volume (ml)	10	10
Extract Dilution	1:1	1:1
ESC Extract No.	PCB318	PCB323
Process File Name	BP183	BP188

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: B20218

MONS 022791

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 given by the average value of a number of 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 "Rnge 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 requir extract dilution, no effort is made to quantify this compound. This could be done if ESC knows that this data is 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 TBEXO1:MH

MONS 022792

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

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Non-EC Active Matrices

ESC Standard Extraction Method No:
PCEX03

MATRIX(S) (CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Soils and other non-EC active solids, solvents and oils	0.5 - 5.0	10

TYPICAL ANALYTES: Aroclors 1232, 1242, 1248, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers

ESC Standard Analysis Method Nos:
ECAN01, ECAN02

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers which are not removed by the three-phase silica-gel column.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
Pentachlorobenzene	50 - 120
2,4,6-trichlorobiphenyl	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson hexane for sample extraction or dilution. If extracts contain electron-capturing interferences, a three-phase silica-gel column may be used. See flow sheet for this extraction method in PCFS03.

LIMITATIONS: Method applies only to matrices which do not contain interferring electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region when electron capture analytical methods are used. This limitation does not apply to mass spectrometric methods.

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

DATES OF METHOD CREATION AND UPDATES: 2/13/84

FLOW SHEET PCFS03

FOR ESC EXTRACTION METHOD PCEX03

SAMPLE ID: --(See Tables)---------- MRC EXTRACT # -(See Tables)-

---------- LOG NO: --(See Tables)----

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

WEIGH OUT SAMPLE INTO A 1 1/2 VIAL ( )

SURROGATE SPIKE OF SAMPLE ( )

--(See Tables)----

DRY SAMPLE IF NEEDED BY ADDING ABOUT 0.1 g Na SO TO VIAL ( )
2 4

DILUTE SAMPLE IN VIAL WITH HEXANE AND PLACE ONTO CLEAN-UP COLUMN. ( )

QUANTITATIVELY RINSE VIAL ONTO COLUMN WITH 4 X 1 mL RINSES. ( )

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. ( )

NITROGEN BLOW DOWN TO --(See Tables)--mL ( )

EXTRACT NO: --(See Tables)--

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

ANALYST: ---(See Tables)-----

ESC JOB NO: --(See Tables)--

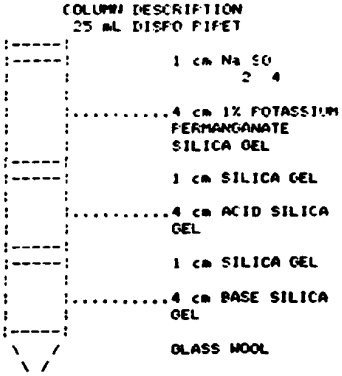
FINAL WEIGHT ----------

INITIAL WEIGHT ----------

SAMPLE WEIGHT -(See Tables)--

( ) RINSE COLUMN WITH 2 X 20 mL HEXANE

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET



MONS 022795

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

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

MRC ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: JANUARY 25, 1984 LOG NO: 1-84-01-25-02

REQUESTER: ST. LOUIS/IVORY/QUEENY PROJECT NO: 434.2020

REPORT RESULTS TO: FILES/MEES.IVORY

ANALYSIS REQUESTS SENT TO: FILES.IVORY.SNYDER.HOLLIDAY

SAMPLE PICKUP BY: David McKenzie

SAMPLE LOCATION: Bldg 2, Lab 28

SAMPLE DESCRIPTION FOR 1 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
HEAT TRANSFER OIL MM-B S-771	1	FCB310
METHOD BLANK		FCB323

RECORDED BY: P. MUPP

REQUIRED ANALYSES: PCB IN OIL

ANALYTICAL TECHNIQUES:

	DATE COMPLETED
X GC/EC (18)	2/14/84

QUALITATIVE: YES SEMIQUANTITATIVE: YES QUANTITATIVE:

REQUIRED QA/QC:

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: X HOLD FOR 30 DAYS

SAFETY PRECAUTIONS:

ANALYSIS REQUEST FILE NAME: AA2020:LI

MONS 022798