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SAMPLING ANALYSIS
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REPORT NO.: MSL-7452
ESC-UAG-87-182

JOB/PROJECT NO.: 760.28-7642

DATE: December 15, 1987

TITLE: ANALYSIS OF ONE RESEARCH SAFETY SAMPLE FOR PCBS.

AUTHORS: B. M. Hughes, D. E. McKenzie and L. R. Minor

ABSTRACT: One oil sample was analyzed for PCBs for Research Safety. This sample was analyzed in order to assist personnel with the determination of proper disposal methodology. The sample was analyzed in duplicate. In both analyses Aroclor 1242 was detected at 3 ug/g. Analyses were conducted with a Level of Detection of 1 ug/g.

TECHNICAL APPROVAL: P. L. Sherman
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Environmental Sciences Center
QC/QA and GLP Supervisor

APPROVAL DATE: January 13, 1988

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TITLE: ANALYSIS OF ONE RESEARCH SAFETY SAMPLE FOR PCBS
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ANALYSIS REPORT

DATE: DECEMBER 15, 1987

LOG NUMBER: U-87-11-10-01 PROJECT NO: 760.28-7642

TO: Randy Parson (W1A)

FROM:

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

Phone No: (314) 694-1464

Title: Analysis of One Research Safety Sample for PCBS

ABSTRACT

One oil sample was analyzed for PCBs for Research Safety. This sample was analyzed in order to assist personnel with the determination of proper disposal methodology. The sample was analyzed in duplicate. In both analyses Aroclor 1242 was detected at 3 ug/g. Analyses were conducted with a Level of Detection of 1 ug/g.

INTRODUCTION

The sample was analyzed using Standard Extraction Method PCEX02 and Standard Analysis Method ECAN06. Summaries of these methods are given in a later section of this report. Calculations were performed using internal standard method of calculation. Results of these analyses are given in Table 1.

SUMMARY

Duplicate analysis showed that Aroclor 1242 was prepsent in the oil at a level of 3 ug/g. All analyses were conducted with a level of detection (LOD) of 1 ug/g. Results of analyses are given in Table 1.

(continued)

QC RESULTS

The oil sample was analyzed with a LOD of 1 ug/g which was determined by spiking the oil with a known amount of Aroclor 1260 Aroclor 1260 was added to the sample at levels corresponding to 1 and 10 ug/g. Recoveries were calculated and were respectively, for low end high level additions, 131 and 127%, respectively. Summaries of recovery data are listed in Table 2. These results indicate that if the oil samples contained PCBs above the LOD they would have been quantifiable. The estimated accuracy of these analyses are on the order of +/- 30%.

DETAILS

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

Standard Extraction Method No(s): PCEX02 (See also flow sheet PCFS02)

Standard Analysis Method No(s): ECAN06 (See also ECFS06)

Sample Identification(s): Hydraulic Fluid- G Bldg Elevator

Final Report File No(s): RA7642

Table File No(s): TA7642,TB7642

Also included in this report are:

(1) Brief descriptions of Standard Extraction and Analysis Methods
(2) Extraction details for all samples.
(3) Chain of Custody and Login Information.
(4) Analytical Request information generated at Login.
(5) Important terminology used in Quality Assurance/Quality Control, Analyte Extraction, and Instrumental Analysis.

ANALYST(S): B. Mason Hughes, La Shawn R. Minor, and David E. McKenzie

ANALYST APPROVAL: B. Mason Hughes

Table 1. Results of capillary GC/EC analyses of Research Safety Samples Using Standard Analysis Method PCEX02.

ESC Project No: 760.28 - 7642 Date DECEMBER 15,1987

SAMPLE I D.	EXTRACT #	LOD(a) (UG/G)	CONCENTRATIONS AROCLOR 1242 DETECTED(UG/G)
HYDRAULIC FLUID-G BLDG ELEVATOR	PCB-2258	1	3
HYDRAULIC FLUID-G BLDG ELEVATOR (DUP)	PCB-2259	1	3
METHOD BLANK	PCB-2260	1	ND (b)

Footnotes:

a. LOD= limic of detection
b. ND= No Aroclor formulations detected above the specified Limit of Detection (1.0 ug/g).

TABLE FILE NO: TA7642:LM

ANALYST(S): B.M. Hughes, D.E. McKenzie, and L.R. Minor

ANALYST APPROVAL: L.R. Minor

MONS 221072

Table 2. Summary of Aroclor 1260 Spiking Concentrations and Recoveries in Research Safety Samples

ESC Project No: 760.28 - 7642 Date: DECEMBER 15,1987

SAMPLE ID	EXTRACT NUMBER	AMOUNT ADDED (ug/g)	RECOVERY (%)
HYDRAULIC FLUID-G BLDG + LOW SPIKE	PCB-2258	1	131
HYDRAULIC FLUID-G BLDG + HIGH SPIKE	PCB-2258	10	127

TABLE FILE NO: TB7642:LM

ANALYST(S): B.M.Hughes, D.E. McKenzie, and L.R. Minor

ANALYST APPROVAL L.R. Minor

MONS 221073

The following pages describe Standard Sampling, Extraction, and/or Analysis Methods used for this project.

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

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

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

EXTRACTION DATE and TIME: .....

SAMPLE COLOR AND DESCRIPTION .....

FINAL WEIGHT .....

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

SAMPLE WEIGHT .....

SURROGATE SPIKE OF SAMPLE [ ] .....

.....

ADD 10 mL BENZENE TO SAMPLE. [ ]

ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH. [ ]

PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS. [ ]

NITROGEN BLOW DOWN IF REQUIRED [ ] FINAL VOLUME ..... mL

EXTRACT NO: .....

DATE/TIME COMPLETED .....

ANALYST: .....

ESC JOB NO: .....

MONS 221075

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

ESC Standard Extraction Method No
PCEX02

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

TYPICAL ANALYTES: Aroclors 1232, 1242, 1248, 1254, 1260, 1262, monochlorobiphenyl isomers through decachlorobiphenyl

ESC Standard Analysis Method Nos ECAN01, ECAN02, MSAN03,MSAN06, MSAN25

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers.

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

APPARATUS AND MATERIALS:
Burdick and Jackson benzene or hexane for sample extraction or dilution
Ultrasonic bath.
Nitrogen for extract volume reduction.
Disposable glassware.
(See flow sheet for this extraction method in PCFS02.)

LIMITATIONS: Method applies only to matrices which do not contain interfering electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region for EC analyses. When interferences are present, a mass spectrometric instrumental analysis method should be used.

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

DATES OF METHOD CREATION AND UPDATES: 10/31/83, 2/3/84, 10/5/84

FLOW SHEET ECFS06
FOR ESC ANALYSIS METHOD ECAN06

EXTRACT ID: ..... ESC EXTRACT # .....

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

EC LOGBOOK PAGE NUMBER: .....

EXTRACT COLOR AND DESCRIPTION .....

PLACE 150 uL EXTRACT OR AROCLOR STANDARD, AND 150 uL INTERNAL STANDARD SOLUTION CONTAINING 0.04 ug/mL CL6-BENZENE IN A 1.6 mL AUTOSAMPLER VIAL. [ ]

PLACE VIAL IN AUTOSAMPLER TRAY OF HP-5880. [ ]

RECORD ELECTRON CAPTURE AND GAS CHROMATOGRAPHIC OPERATING PARAMETERS AND 3357-LAS FILE INFORMATION IN INSTRUMENT LOGBOOK. [ ]

DETERMINE WHETHER TO USE INTERNAL OR EXTERNAL QUANTITATION METHODOLOGY. INTERNAL QUANTITATION IS PREFERED, BUT CAN NOT ALWAYS BE USED DUE TO INTERFERENCES. [ ]

[ ] CALCULATE CONCENTRATION OF AROCLOR FORMULATION AVERAGING THE VALUES DETERMINED FOR THE 10 - 30 PCB CONGENERS, USING THE APPROPRIATE METHOD OF CALCULATION LISTED BELOW

INTERNAL STANDARD CALCULATION

$$RRF = \text{Relative Response Factor} = \frac{(\text{congener area in Aroclor standard})}{(\text{CL6-benzene area})(\text{Aroclor concentration})}$$

$$\text{Aroclor Concentration} = \frac{(\text{congener area})}{(\text{CL6-benzene area})\ (RRF)}$$

THESE EQUATIONS ASSUME THAT THE SAME AMOUNT OF INTERNAL STANDARD IS ADDED TO EXTRACTS AND STANDARDS

EXTERNAL STANDARD CALCULATION

Aroclor Concentration = (congener area)/(RF)

RF = Response Factor = (congener area in Aroclor Standard)/(Aroclor Concentration)

Both above calculations are performed for 10 - 30 PCB congenars and the values averaged to yield an average Aroclor formulation concentration. Both methods are also corrected for dilution by multiplying the average concentration by (Extract Volume)/(Sample Amount).

MS FILE NUMBER: ..... DATE/TIME COMPLETED ...../.....

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

MONS 221077

ESC Standard Analysis Method (SAM) ESC Standard Analysis Method No
TITLE: Aroclor Formulations in Solvents ECAN06

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 congeners are potential interferences. Exact retention times of most likely PCB congeners can be obtained from the analysis of Aroclors 1248, 1254, and 1260.

CHROMATOGRAPHIC COLUMN: 25 m fused silica Lee Scientific SB-30 (0.25 uM) with (0.32 mm ID)

COLUMN TEMPERATURE PROGRAM: 130(1)/5/300 INJECTION TYPE: Splitless

---

CALIBRATION AND STANDARDIZATION: Use internal or external standard quantitation technique and calibrate instrument response factors from authentic Aroclor standards. Use a minimum of 10 PCB congeners for the determination of formulation identity and quantity. The external standard quantitation technique is used when chromatographic interferents are present in the retention time region of the internal standard.

CALCULATIONS: Internal Standard Method

$$\text{Formulation concentration} = \frac{\text{(Aroclor Congener Area)}}{\text{(RRF)(Internal Standard Area)}}$$

$$\text{RRF = Relative Response Factor} = \frac{\text{(Aroclor Congener Area)}}{\text{(Internal Standard Area)(Aroclor Concentration)}}$$

External Standard Method

Formulation concentration = (Aroclor Congener Area)/(RF)

RF = Response Factor = (Aroclor Congener Area in Standard)/(Standard Concentration)

The above formulation concentration calculations are performed for 10 - 30 PCB congeners and the values averaged to yield an average Aroclor formulation concentration. Both method concentrations are corrected for dilution by multiplying the average concentrations by (Extract Volume)/(Sample Amount).

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

DATE OF METHOD CREATION: 12/05/87 Time Stamp - <871216.1328>

MONS 221078

The following pages show extraction details for samples associated with this project.

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: HYDRAULIC FLUID - G-BLDG ELEVATOR — PCB-2258

MRC EXTRACT # U-87-11-10-01

LOG NO: .....

EXTRACTION DATE and TIME: DECEMBER 1, 1987

SAMPLE COLOR AND DESCRIPTION: YELLOW

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ..... INITIAL WEIGHT ..... SAMPLE WEIGHT 0.944 g
SURROGATE SPIKE OF SAMPLE	[ ]	HS= 100 uL DEM 092386-01 LS= 100 uL DEM 092386-01
ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

EXTRACT NO: PCB-2258

DATE/TIME COMPLETED: DECEMBER 15, 1987

ANALYST: MINOR

ESC JOB NO: 760.28-7642

MONS 221080

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: HYDRAULIC FLUID - G-BLDG ELEVATOR (DUPLICATE) ---------- PCB-2259

MRC EXTRACT # U-87-11-10-01

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

EXTRACTION DATE and TIME: DECEMBER 1, 1987

SAMPLE COLOR AND DESCRIPTION: YELLOW

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ---------- INITIAL WEIGHT ---------- SAMPLE WEIGHT 0.914 g
SPIKE OF SAMPLE	[ ]	----------
ADD 10 mL BENZENE TO SAMPLE.	[X]	----------
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 mL

PCB-2259

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

DATE/TIME COMPLETED: DECEMBER 15, 1987

ANALYST: MINOR

ESC JOB NO: 760.28-7642

MONS 221081

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

SAMPLE ID: METHOD BLANK ---------- MRC EXTRACT # PCB-2260 ----------

---------- LOG NO ----------

EXTRACTION DATE and TIME: DECEMBER 1, 1987 ----------

SAMPLE COLOR AND DESCRIPTION CLEAR ----------

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[X]	FINAL WEIGHT ---------- INITIAL WEIGHT ---------- SAMPLE WEIGHT ----------
SPIKE OF SAMPLE	[ ]	----------

ADD 10 mL BENZENE TO SAMPLE.	[X]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 1 mL OF THE EXTRACT INTO A 1 1/2 mL VIAL FOR ANALYSIS.	[ ]	
NITROGEN BLOW DOWN IF REQUIRED	[ ]	FINAL VOLUME 10 ---------- mL

PCB-2260

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

DATE/TIME COMPLETED DECEMBER 15, 1987 ----------

ANALYST: MINOR ----------

ESC JOB NO: 760.28-7642 ----------

MONS 221082

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

MONS 221083

MONSANTO MEMO

FROM: Randy Parson, Environmental Services, W 101, 4-1282 4-4609

cc: R. J. Edwards R3B
T. W. McMillan W1A 4-831
Mike Arnold 4-804

DATE: November 3, 1987

SUBJECT: Hydraulic Fluid/G-Bldg Elevator
G/W Drum #1

TO: Mr. Mason Huges U4B

The enclosed sample is Hydraulic Fluid from G-building elevator. We would like to have this analysed for PCB.

If you need more information and/or have any questions about the sample, don't hesitate to call me at 4-1282.

Thanks again.

Randy Parson 4-1282
Randy Parson W1A

7642

62-000-760.55-265.005

12/1/87

Septum on the bottom of the Vial

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

Time Stamp - <871217 1351>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE· NOVEMBER 10,1987 LOG NO: U-87-11-10-01

REQUESTER RANDY PARSON PROJECT NO: 760.28-7642

LOGIN NOTEBOOK PAGE (To be filled out at login.): 3759226

REPORT RESULTS TO: FILES/MEES,HUGHES,SHERMAN,MCKENZIE

ANALYSIS REQUESTS SENT TO: FILES MEES,SHERMAN,HUGHES,MCKENZIE,MINOR

SAMPLE PICKUP BY:

SAMPLE LOCATION: U420

SAMPLE DESCRIPTION FOR 1 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
HYDRAULIC FLUID/G-BLDG ELEVATOR G1W DRUM #1	1 X 40 mL	PCB-2258 PCB-2259
NOTE: A SEPTUM WERE FOUND AT THE BOTTOM OF THE SAMPLE		
METHOD BLANK		PCB-2260

RECORDED BY: MINOR

REQUIRED ANALYSES: PCBs

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158642

ESC STANDARD EXTRACTION METHOD(S): PCEX02

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
X- GC/EC ( )	12/3/87	ECAN06	157307

FINAL REPORT NOTEBOOK PAGE: 3745304

OTHER NOTEBOOK PAGES:

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE. X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

MONS 221086

COMMENTS:

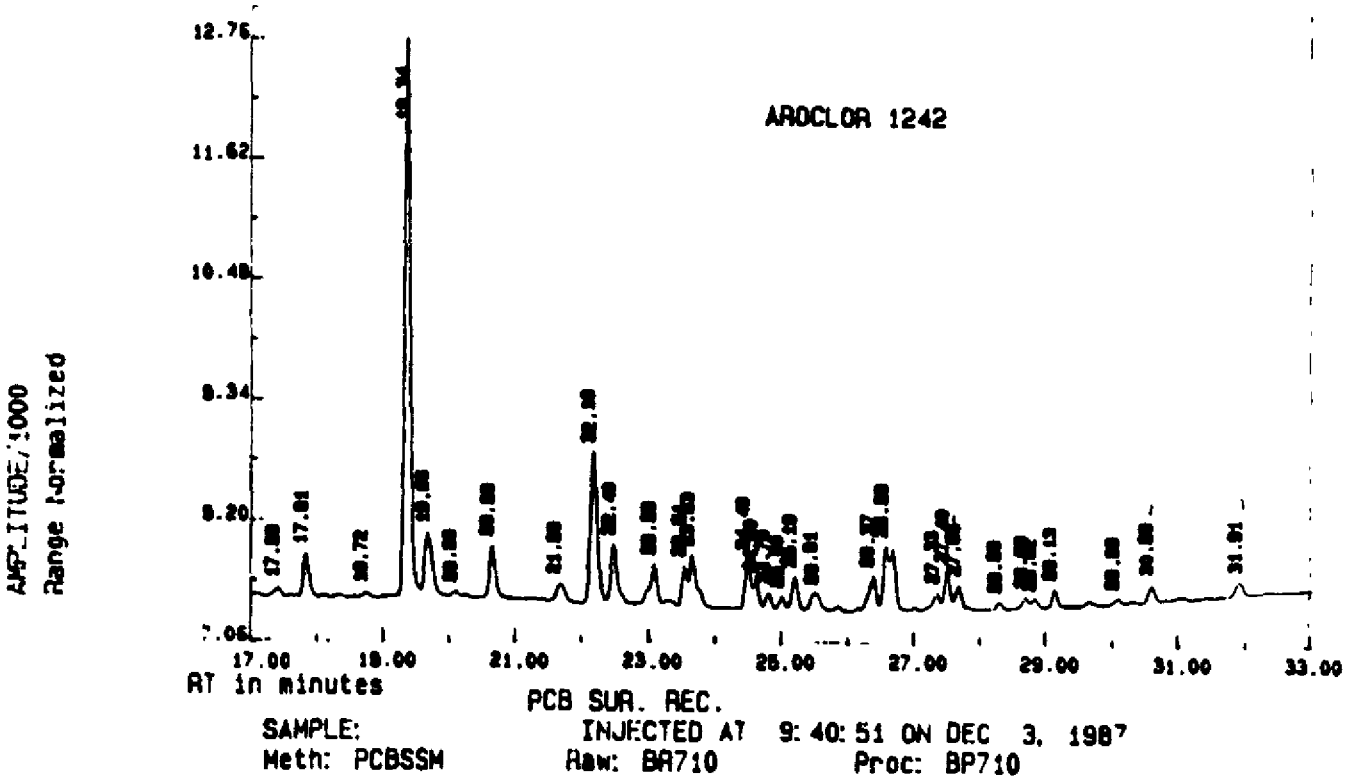
SAMPLE DISPOSITION: HOLD FOR 90 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

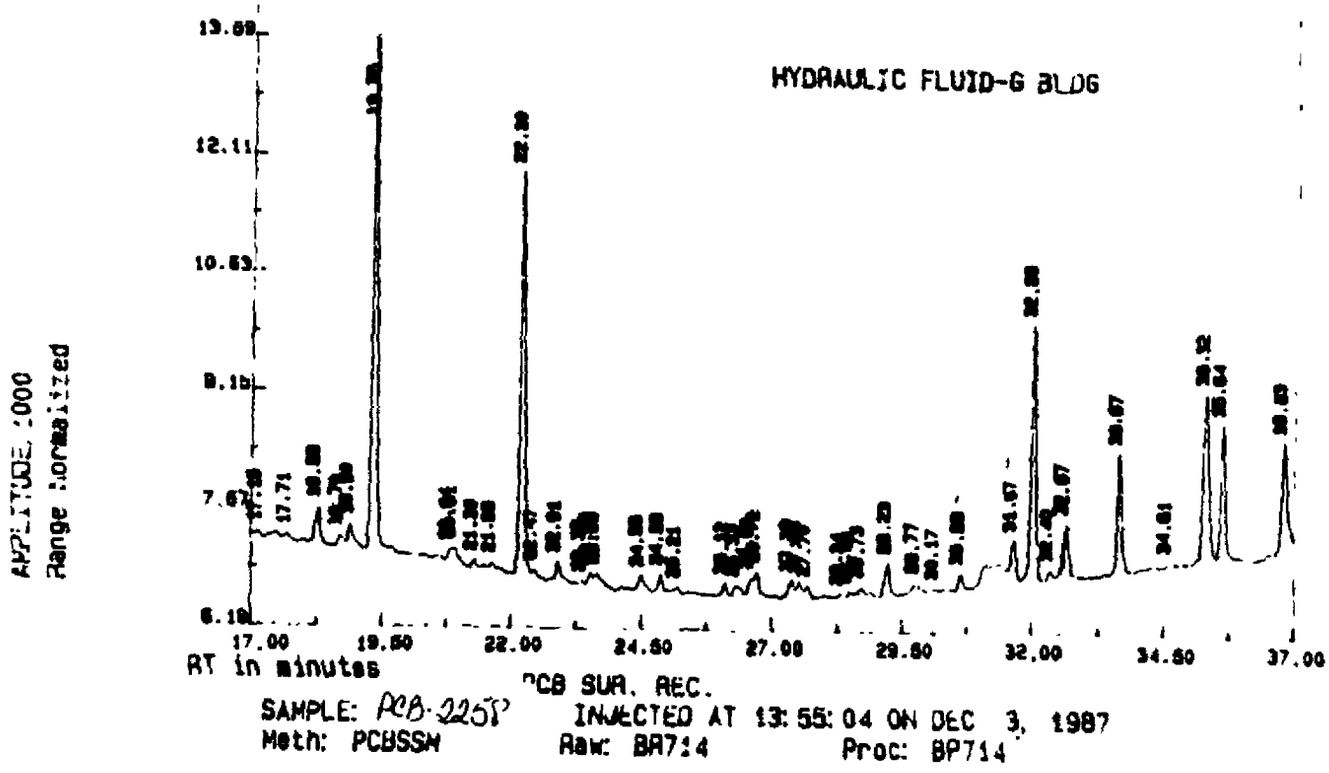
ANALYSIS REQUEST FILE NAME: AA7642·LI

MONS 221087

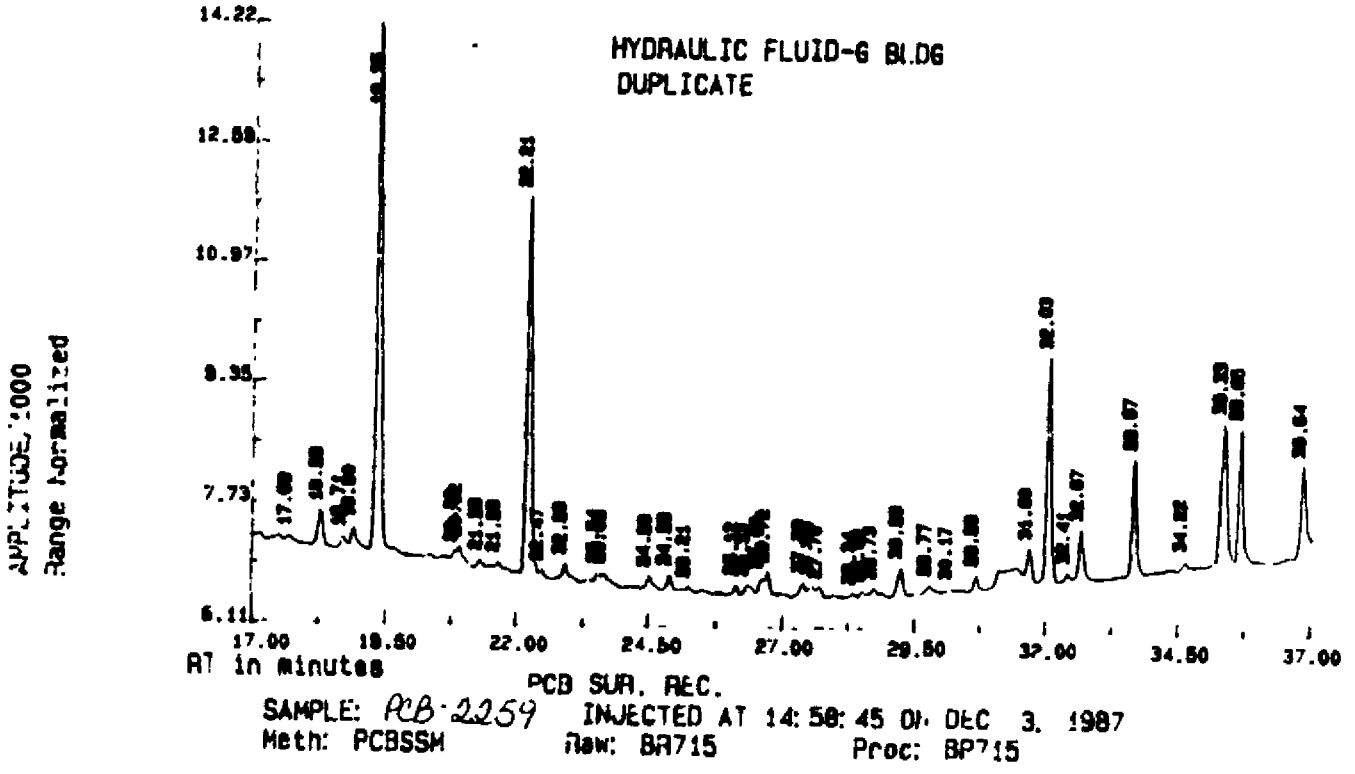
The following pages show QA/QC data for selected samples.


AMPLITUDE/1000
Range Normalized
12.76
11.62
10.48
9.34
8.20
17.00
19.00
21.00
23.00
25.00
27.00
29.00
31.00
33.00
RT in minutes
AROCLOR 1242
PCB SUR. REC.
SAMPLE:
INJECTED AT 9:40:51 ON DEC 3, 1987
Meth: PCBSSM
Raw: BR710
Proc: BP710

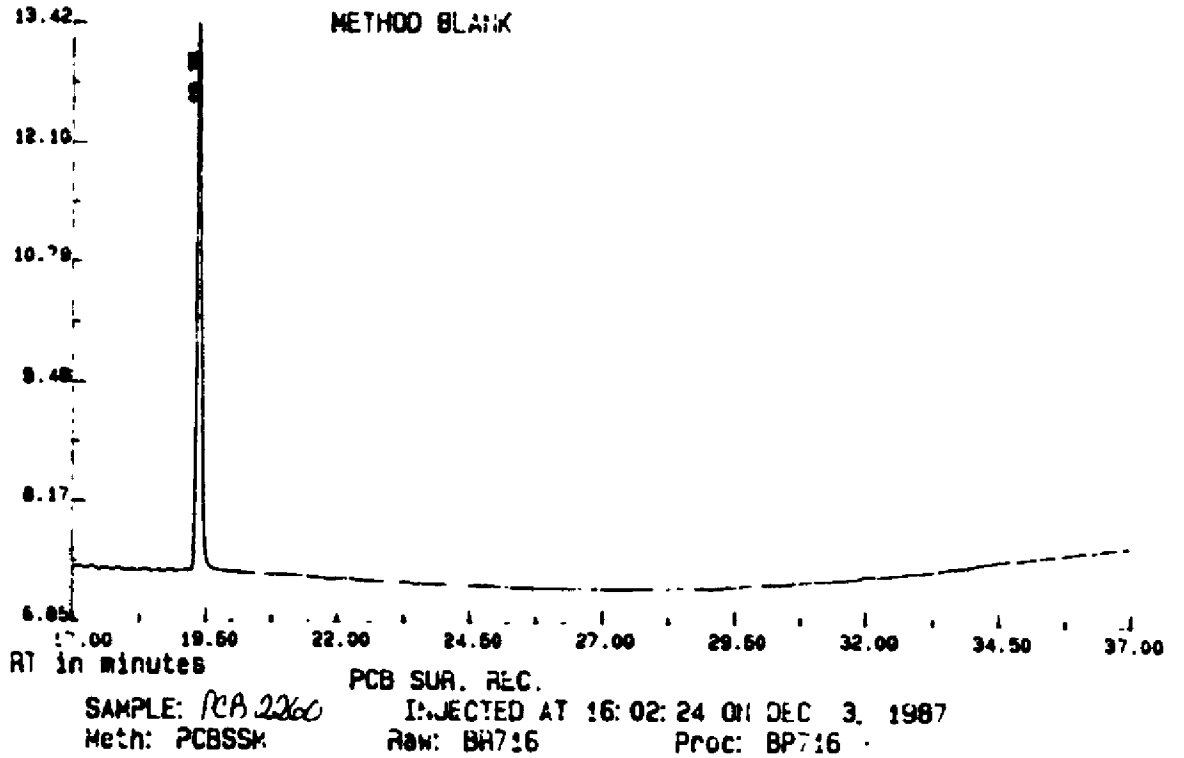
MONS 221089


HYDRAULIC FLUID-G BLDG
AMPLITUDE 1000
Range Normalized
13.69
12.11
10.53
8.95
7.37
6.10
17.00
19.50
22.00
24.50
27.00
29.50
32.00
34.50
37.00
RT in minutes
PCB SUR. REC.
SAMPLE: PCB-225
INJECTED AT 13:55:04 ON DEC 3, 1987
Meth: PCBSSW
Raw: BA714
Proc: BP714

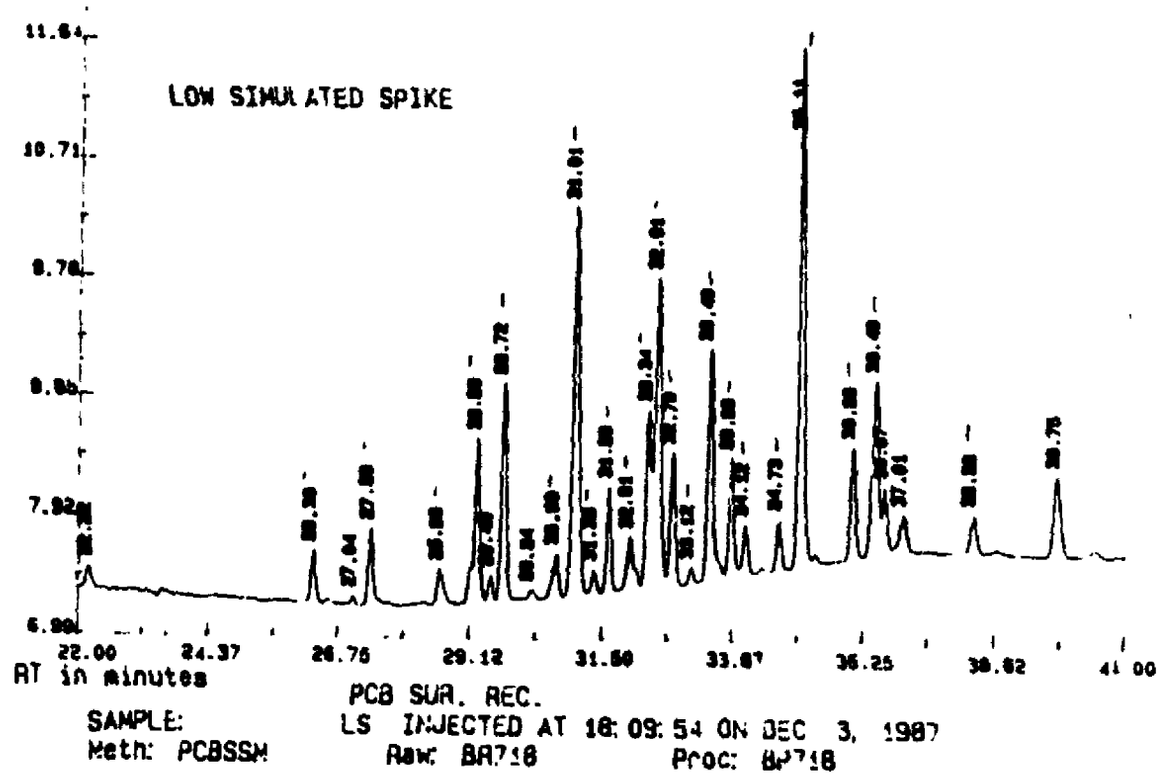
MDNS 221090


HYDRAULIC FLUID-G BLDG
DUPLICATE
14.22
12.89
10.97
9.35
7.73
6.11
AMPLITUDE, 1000
Range Normalized
17.00
19.50
22.00
24.50
27.00
29.50
32.00
34.50
37.00
RT in minutes
PCB SUR. REC.
SAMPLE: PCB-2259 INJECTED AT 14:58:45 ON DEC 3, 1987
Meth: PCBSSM Raw: BR715 Proc: BP715

MONS 221091

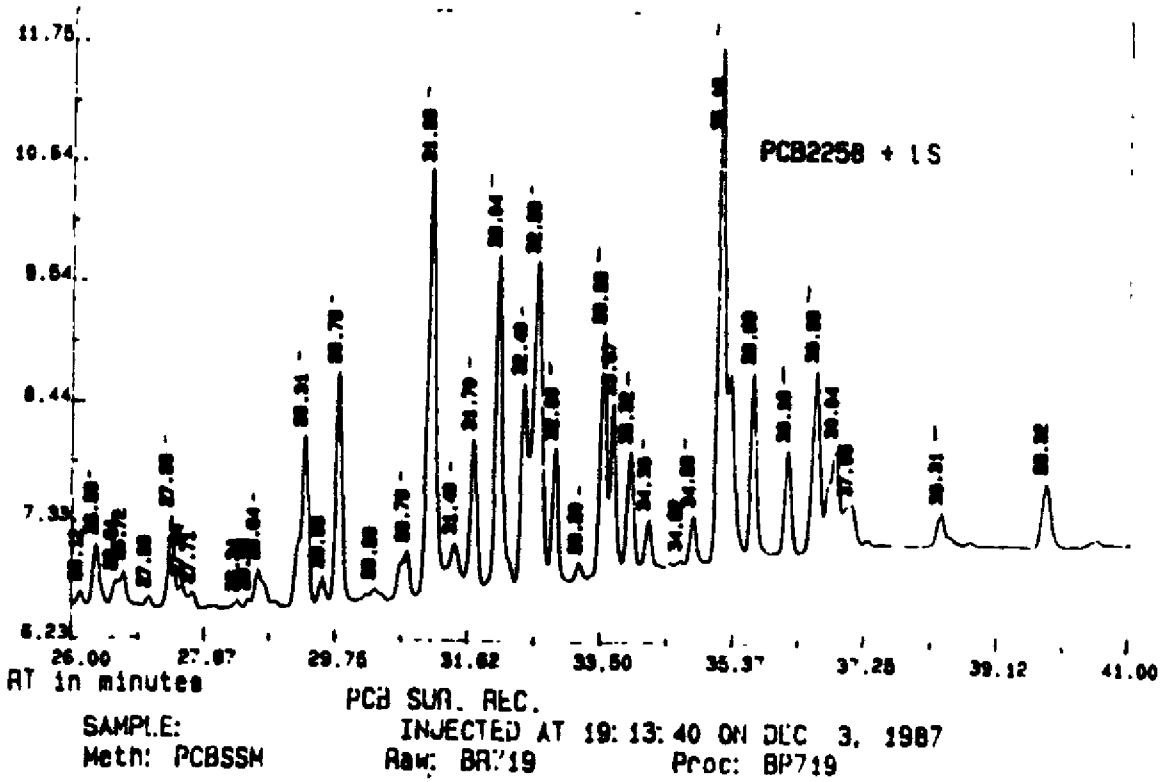

METHOD BLANK
13.42
12.10
10.79
8.48
6.17
AMPLITUDE/1000
Range Normalized
17.00
19.50
22.00
24.50
27.00
29.50
32.00
34.50
37.00
RT in minutes
PCB SUR. REC.
SAMPLE: PCB 2260
INJECTED AT 16:02:24 ON DEC 3, 1987
Meth: PCBSSK
Raw: BH716
Proc: BP716

MONS 221092

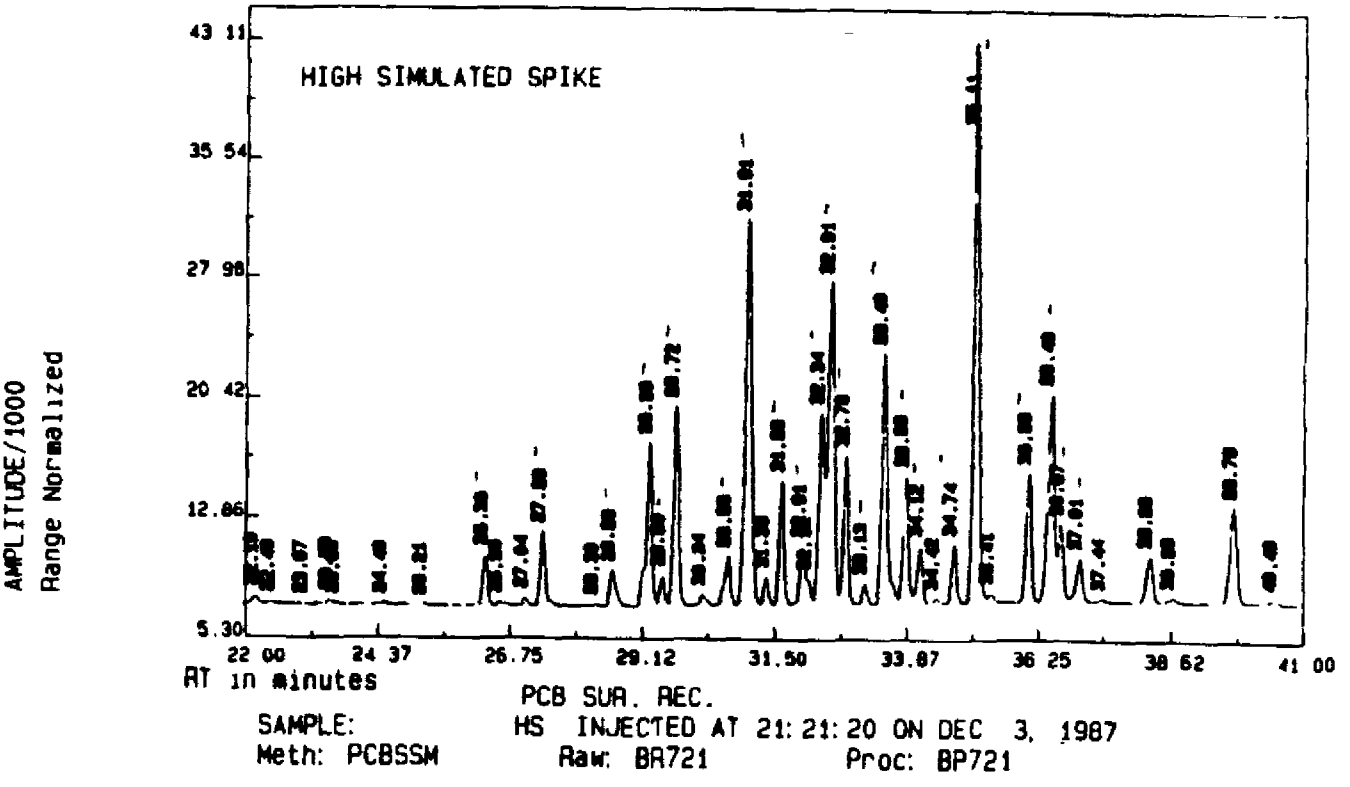


AMPLITUDE/1000
Range :ormalized

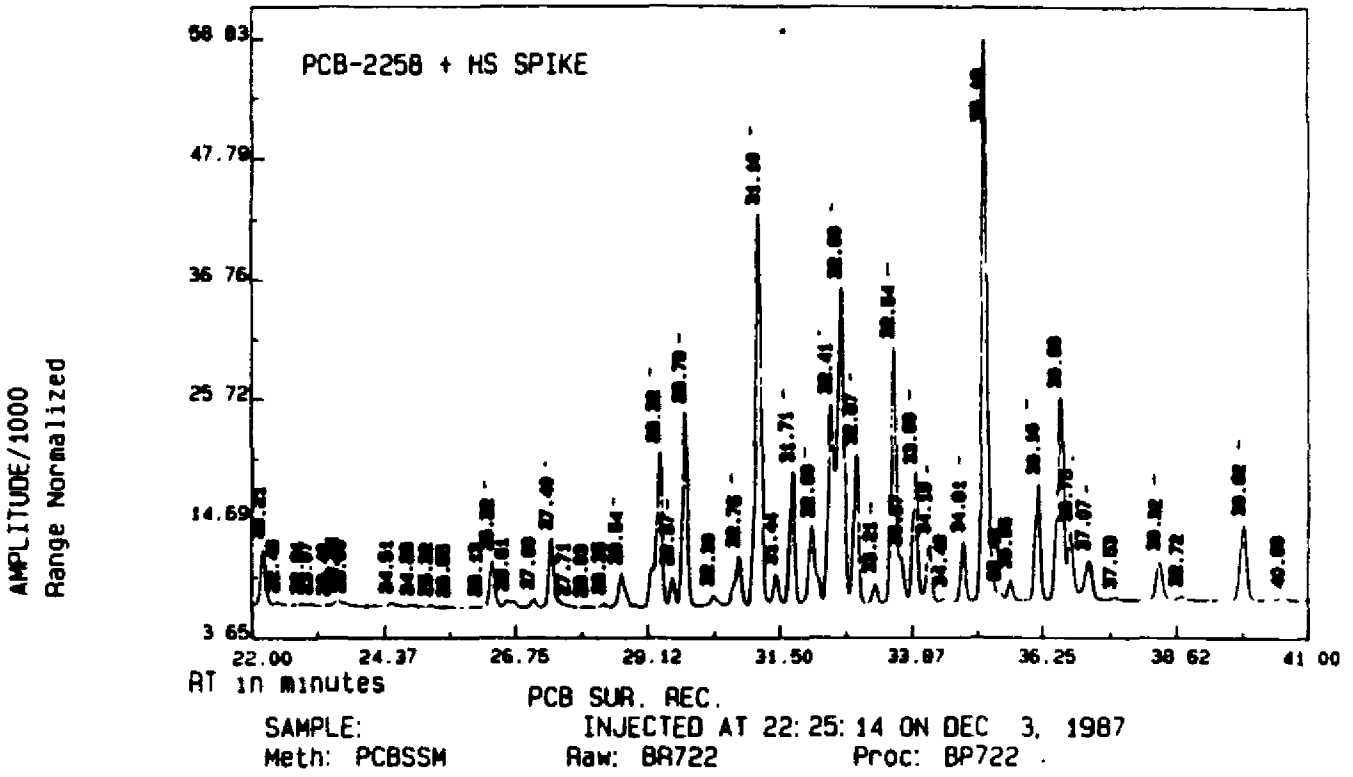
MONS 221093


PCB2258 + LS
11.75
10.54
9.64
8.44
7.33
6.23
26.00
27.87
29.75
31.62
33.50
35.37
37.25
39.12
41.00
RT in minutes
PCB SUR. REC.
SAMPLE:
INJECTED AT 19:13:40 ON DEC 3, 1987
Meth: PCBSSM
Raw: BR719
Proc: BP719

MONS 221094


HIGH SIMULATED SPIKE
AMPLITUDE/1000
Range Normalized
43 11
35 54
27 98
20 42
12.86
5.30
22 00
24 37
26.75
29.12
31.50
33.87
36 25
38 62
41 00
RT in minutes
PCB SUR. REC.
SAMPLE:
HS INJECTED AT 21:21:20 ON DEC 3, 1987
Meth: PCBSSM
Raw: BR721
Proc: BP721

MONS 221095


PCB-2258 + HS SPIKE
58 83
47.79
36 76
25 72
14.69
3 65
AMPLITUDE/1000
Range Normalized
22.00
24.37
26.75
29.12
31.50
33.87
36.25
38 62
41 00
RT in minutes
PCB SUR. REC.
SAMPLE:
INJECTED AT 22: 25: 14 ON DEC 3, 1987
Meth: PCBSSM
Raw: BR722
Proc: BP722

MONS 221096

The following sections describe important terminology used in Quality Assurance/Quality Control, Analyte Extraction and Instrumental Analysis ESC and ESC/UAG Good Laboratory Practices Manuals can be obtained by contacting Janet Lee (U4D) METS 544-1273, GLP and QA/QC Supervisor.

Quality Assurance/Quality Control Terminology

This section describes commonly used terminology and phrases which are used in conjunction with Quality Assurance/Quality Control (QA/QC) This information may be helpful in understanding some details of QA/QC procedures which have been incorporated in the present report. The following two sections also give useful information concerning terminology used in analyte extraction and instrumental analysis of samples and sample extracts

Quality Assurance/Quality Control - Terminology used to describe activities which are designed to produce analytical results of known quality In an effort to minimize costs of environmental analyses, specific QA/QC elements have been incorporated into ESC/UAG extraction and analysis steps which should not unduly burden analytical studies with large development and method validation costs. This approach has been taken so that sets of samples can be analyzed for various analytes without extensively validating methods over wide concentration ranges and without using methods which are designed for only one matrix.

One price which one pays for this approach is that analytical precision and accuracy are sacrificed, somewhat. However, in many cases, when clearly defined regulatory limits have been set, confirming that the analyte is present at levels well below or well above the limit does not need the type of accuracy that is required when the regulated analyte concentration is in the vicinity of the regulatory limit. In these latter cases, more extensive QA/QC procedures are required than are included in the present study

Precision - Terminology used to evaluate the reproducibility of a certain determination. Parameters which affect analysis precision range from sample acquisition, to sample storage, to sample extraction, and finally to sample extract analysis. Normally, the major factor which affects the precision of the analysis is the sample extraction step This step normally results in sample analysis precision on the order of + or - 30%.

The precision of a result can be determined in a number of ways Replicate analyses of the same sample, followed by the calculation of a percent relative standard deviation for that determination is the most popular method of assessing precision. However, this calculation should not be made when there are less than three replicate points from which to calculate the percent relative standard deviation. In addition, replicate extractions and instrumental analyses must be performed. Therefore obtaining percent relative standard deviation data can be very expensive and is not normally obtained unless large studies are being conducted. A second method of assessing precision is with the use of duplicate analyses The present report includes results of at least one sample which was analyzed in duplicate. NOTE!! It is the responsibility of the analysis requestor to review these data and draw his own conclusions concerning the precision of the determination, and, more importantly, if the precision of the determination meets his needs.

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Accuracy - Terminology used to determine the "correctness" of the data The accuracy of most steps in the analysis of samples is typically on the order of 10%. However, when analyte concentrations in the sample extracts approach the level of detection of a determination, accuracy as well as precision can approach 100% or greater. The accuracy of a determination of an analyte which is present at concentrations which are much greater than the instrumental level of detection is most strongly affected by the homogeniety of the sample matrix and the precision of the extraction step, which results in typical accuracies in environmental analyses to be on the order of 30%. Normally, the accuracy of standard solutions which are used to calibrate analytical instrumentation is well below this amount, and therefore is normally not an important factor in producing inaccurate data. From time to time, analyte responses of standard solutions from different sources will be compared to standard solutions used for instrument calibration. This information is normally included in the section entitled "QA/QC Results".

The accuracy of a result can be estimated in a number of ways The current study uses surrogate standard recovery and native analyte recovery information to aid in assessing the accuracy of a result This recovery information, normally reported as percent recovery, gives you information concerning the percent of surrogate standards and native analytes which were recovered after adding to your sample matrix and performing the various extractions, clean-ups, and instrumental analyses associated with the determination. The use of surrogates also assumes that the spiked surrogates are bound in the matrix in the same way as the analytes of interest. Surrogate standards are added to every sample while native analytes are added to only 10% of each set of samples. Therefore surrogate standard recovery data could be used to correct the reported results of an analyte concentration, if the user of the data feel such corrections are warranted. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Bias - Terminology used to describe how the accuracy of a determination may be affected. A low bias means that values below the correct value are normally measured. A high bias means that values above the correct value are normally measured. The instrumental analysis step of extract analysis normally does not contribute significantly to analysis bias. Most bias in environmental analysis is introduced in the sample extraction step. A low bias is introduced when the sample is not completely extracted and the extract does not contain 100% of the analyte. A high bias is introduced when the final volume of the extract is less than the correct volume. Surrogate Standards (SS), which are discussed in the next section, can be used to determine the amount of bias which is incorporated in the reported result. NOTE!! No data reported in this report includes any correction for this bias. It is the responsibility of the person requesting the analyses to apply appropriate bias corrections, if he/she feels that such corrections are warranted.

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Level of Detection (LOD) - Terminology used to indicate what analyte concentration will produce a signal-to-noise ratio of 10. For the present report, this value is not experimentally determined Normally, a Limit of Quantitation is used to determine the threshold concentration above which an analyte can be detected.

Limit of Quantitation (LOQ) - Terminology used to indicate the lowest analyte concentration which can reliably be quantified. The precision of the determination of the analyte concentration at the LOQ is normally on the order of 100%. The LOQ is normally determined experimentally by adding a known amount of analyte to the sample matrix and measuring the percent recovery at the LOQ. In some cases, when there is insufficient sample quantity, or for convenience, an LOQ will be determined using other methods. These methods should be clearly described in this report.

Further Information

The general area of Quality Assurance/Quality Control, along with much of the terminology described above, is very complex. The following references may provide further information which may be useful in understanding the validity of the results reported in this study.

ASTM Standard Practice D4210-83 entitled "Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data".

ASTM Standard Practice D2777-77 entitled "Determination of Precision and Bias of Methods of Committee D-19 on Water".

An article in Analytical Chemistry, authored by 29 well-known environmental scientists. This article was funded by a grant from the Environmental Protection Agency and acknowledges 24 additional scientists in industry and government who were involved in the review of the article. Its citation is "Principles of Environmental Analysis", L. H. Keith, et. al., Anal. Chem. 1983, 55, 2210 - 2218.

40 CFR Part 136 entitled "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule". This document includes details of EPA's 600-series analysis methods. A current copy of these methods can be found in the October 26, 1984 Federal Register.

QAQCTE:MH

Analyte Extraction Terminology

This section describes commonly used terminology and phrases which are used in conjunction with analyte extraction. This information may be helpful in understanding some details of how analytes in your samples were isolated for instrumental analysis Instrumental Analysis Terminologies are given in the following section of this report.

Sample - Terminology used to reference the material which you submit for analysis. Samples may be solid, liquid, and in some cases, gases In general, when a sample is received for analysis, it is logged into the ESC sample login book and proper chain-of-custody documents are maintained in the event this information may be required for litigation purposes.

Analyte - Terminology used to reference specific compounds or formulations of compounds. When an analysis is requested for a certain analyte within a certain sample matrix, normally some type of extraction process is required.

Extraction - This terminology refers to the step which isolates the analyte of interest into an appropriate solvent. Extraction is required for several reasons. In many cases, the concentration of the analyte is below the level of detection of the instrument which will be used for analysis. Therefore, extraction involves removing the analyte of interest from a relatively large volume of sample and placing it into a relatively small volume of solvent. This places the analyte in the solvent at a concentration which can easily be detected by the analytical instrument.

A second reason that extraction is required, is that the sample may not be appropriate for direct instrumental analysis of the analyte of interest. This can be easily understood for analytes in soils. Very few analytical instruments allow soils to be directly inserted into the instrument. Therefore some type of isolation step (i. e. extraction) is required in order to place the analyte in an appropriate matrix for instrumental analysis.

When the analyte of interest is present at sufficient concentration, and the sample matrix can be directly inserted into the analytical instrument, the sample, or the diluted sample, can be directly analyzed. However, this case represents a minority of sample analyses which ESC is asked to perform.

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Matrix - Terminology used to reference the major components of a sample In most cases, major components are not of direct interest except as they affect the types of extraction steps and other isolation procedures which must be used to isolate the analyte from the sample matrix. For the majority of environmental samples which ESC is asked to analyze, the matrix is normally water or soil. However, many environmental matrices contain relatively high levels of organic compounds which may interfer with the instrumental analysis of the analyte of interest Therefore, depending upon the analyte of interest, the required level of detection, and the amounts and types of other organic materials which may interfer with the instrumental analysis, extraction methods must be developed which are very specific for a given set of samples. In order to deal with almost a limitless set of matrices, analytes, extraction methods and instrumental methods, an organized approach to sample extraction, followed by instrumental analysis, has been developed at ESC

Extract - Terminology used to identify the analyte-containing solvent which was obtained from the extraction of a certain matrix.

Extract Clean-up - Terminology used to identify chemical steps which are required in order to reduce the concentration of organic compounds which may be extracted with the analyte of interest. These co-extracted compounds may be at such high levels and/or produce responses so that they interfer with the instrumental analysis of the analyte in the extract.

Extractable Compounds - Terminology which refers to the ability of a compound to be isolated from the sample matrix and placed into the extract solvent. When the sample matrix is primarily soil which contains little organic matter, most organic compounds are extractable compounds. However, when the sample matrix is primarily water, only those compounds which are hydrophobic are extractable compounds

Standard Extraction Method - Terminology used to reference an organized description of the steps involved in isolating an analyte from a given matrix. These methods are assigned short names which can also be used as computer file names for these methods. The names of specific Standard Extraction Methods which are used in this study are listed in the final report in the section entitled "Details" In addition, copies of these Standard Extraction Methods are shown in a separate section of this report. Standard Extraction Methods also include details of sample clean-up, if required.

Extraction Flow Sheet - Terminology used to reference a graphic description of a Standard Extraction Method. This flow sheet is filled out by the analyst in the laboratory and serves as a detailed record of what occurred in the sample extraction step. Flow sheets of individual sample extractions may be included in a section of this report.

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Surrogate Standards (SS) - One important element in the extraction of an analyte from a matrix, is the determination of what percent of the analyte was actually extracted from the matrix. For methods which involve liquid/liquid extraction of water with an organic solvent, or liquid/solid extraction of soils with an organic solvent, it is imparative that data be obtained which gives the analysis requestor information concerning what percentage of ths analyte was isolated in the extract during the extraction step. Surrogate Standards serve this important purpose.

The philosophy on the use of Surrogate Standards is as diverse as the analyets who use them. If cost were no object, analyte recovery information could be obtained by the use of standards addition analysis methods. Howsver, since many samples require extensive labor to isolate an analyte into an extract, standards addition analysis methods are too costly for most studies. (A standards addition analysis requires each sample to be extracted and analyzed at leest three times.) Surrogata Standards give the same type of information, with only one analysis being required.

A "Surrogate" Standard is a compound which is chosen to behava exactly as the analyte. It is a surrogate for that analyte and thus information obtained for this surrogate can be assumed to also apply to the analyte of interest. For ths analysis of regulated analytes, it is important that when data is generated that shows that the analyte is not present, that we can also show that if ths regulated analyte were present in the sample matrix, it would have been detected. This is especially true when complex extraction and instrumental analysis steps are involved in this determination Therefore, when an extraction is performed, a known amount of a Surrogate Standard is added to the sample before any extraction steps take place. When the extract is then analyzed for the analyte of interest, the amount of the Surrogate Standard is also determined From this information one can document, for each sample, what recoveries were measured for this Surrogate Compound. From this, one can describe some level of confidance on the data generated for the analyte of interest.

A detailed discussion on how Surrogate Standards are chosen is beyond the scope of this section on Analyte Extraction Terminology. ESC normally uses two types of Surrogate Standards. The first is when the surrogate is exactly the same as the analyte of interest, except that the naturally occurring carbon, hydrogen, or halogen atoms in the analyte have been replaced with non-naturally occurring isotopes. These surrogatee can be expected to act exactly as the native analytes. One good example of this type of surrogate is the 2,3,7,8-TCDD surrogate in which all carbon atoms are carbon-13 or all chlorine atoms are chlorine-37.

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The second type of Surrogate Standard is one which does not have the same chemical structure as the analyte of interest, but is known or assumed to act chemicelly identicel to the analyte of interest during the extraction and clean-up steps. Examples of this type of surrogates are deuterated compounds which may be added to a matrix in which any chromatographable materials are to be identified This approach can only be applied to GC/MS analyses, since the surrogates can be differentiated from the native compounds from their different mass spectra. Since it is not known before sample extraction and extract anelysis whet analytes may be present, surrogates must be chosen that could not possibly be preeant in the sample In addition, since only liquid/liquid or liquid/solid extraction would be involved, with no clean-up (since clean-up would remove compounds in which the requester is interested) any hydrophobic deuterated compound could serve es a surrogate. Normally, phenol-d5, 2-CL-phenol-d4, CL2-benzene-d4, pyrene-d10 end chrysene-d12 are used as Surrogate Standards for the analysis of a wide variety of hydrophobic materials in environmental samples.

A sub-set of this second type of Surrogate Standards are the surrogates which ere used in the capillary GC/EC analysis of Aroclor formulations. Since mass spectrometric detection is not used, surrogates which contain unique isotopes cannot be distinguished from their native counterparts Therefore, other compounds are used as Surrogate Standards since they have been observed to act exactly es the polychlorineted biphenyls (PCBs) in the Aroclor formulations. Any halogenated compound can be used which acts exactly as PCBs in the extraction and cleen-up step. ESC normally uses CL5-benzene and 2,4,6-CL3-biphenyl for this purpoee.

Native Analytes - Terminology which references the analytes of interest which contain naturally-occurring isotopes of carbon, hydrogen, halogens, etc.

Native Spike - Terminology which references the addition of a known amount of native analytes to the sample matrix. Normally, at least one sample will be spiked with the native analyte at the reported level of detection (low native spike) and at 10 times the level of detection (high native spike). Recoveries of the native analytes provide further documentation on the quality of the analyses performed. For sample sets which contain more than 10 samples, the frequency of low and high native spikes is 10% of the number of samples.

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Method Blank - Terminology which refers to a sample which is known to contain none of the analytes of interest. For water analyses, this is normally Burdick and Jackson Brand Water, de-ionized, or distilled water. For soil, it may be soil which has been previously analyzed and known to contain none of the analytes of interest. All extraction steps, using randomly selected glassware, solvents, and all surrogate spiking solutions must be used. This provides documentation that the analytes of interest were not inadvertantly present in extraction glassware and solvents used in the analysis. A minimum of one method blank must be generated with each set of samples For sample sets containing more than 10 samples, method blanks are generated at a frequency of 10% of the number of samples

Duplicate Analyses - Terminology used to indicate the duplicate extraction and duplicate extract analysis of a sample. This provides documention on the precision of an analysis. Normally the precision of environmental analyses of homogeneous samples is on the order of 30%. A minimum of one duplicate analysis is required for each sample set For sample sets containing more than 10 samples, duplicate analyses are generated at a frequency of 10% of the number of samples.

Further Information

The general area of Analyte Extraction, along with much of the terminology described above, is very complex. The following references may provide further information which may be useful in understanding extraction fundamentals and terminology.

<u>40 CFR Part 136</u> entitled "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule". This document includes details of EPA's 600-series analysis methods. A current copy of these methods can be found in the October 26, 1984 Federal Register.

<u>Statement of Work for the Contract Laboratory Program</u> which is used in conjunction with EPA's Superfund Site studies. A current copy may be obtained by contacting:

Joan F. Fisk, Project Officer
Analytical Support Branch
Hazardous Response Support Division
USEPA
Washington, D. C. 20460

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Instrumental Analysis Terminology

This section describes commonly used terminology and phrases which are used in conjunction with extract analysis. This information may be helpful in understanding some details of how instrumental analyses of sample extracts were performed. The previous section of this report describes terminology associated with analyte extraction.

Sample Extract - Terminology used to describe the solvent containing the analyte of interest which is obtained from some type of sample extraction step. Detailed information on how this extract is prepared can be found in the Standard Extraction Methods and flow sheets associated with the methods. Summaries of these methods which were used for this project can be found in a separate section of this report.

Internal Standard (IS) - Terminology used to reference a compound which is added to the sample extract and standard solutions of the analytes of interest immediately before instrumental analysis. The purpose of the internal standard is to provide data which can be used to correct for variations in injected volumes of extract and standard solutions which may occur during instrumental analysis. The internal standard must not be present in the sample extract. Typical internal standards used in mass spectrometric analyses are benzene-d6, naphthalene-d8, anthracene-d10 and chrysene-d12. These deuterated compounds produce unique mass spectra and are not present in naturally-occurring samples. A commonly used internal standard for GC/EC analysis is hexachlorobenzene.

Internal Standard Quantitation Method - Terminology which refers to a method of data analysis. This method requires that a known amount of an internal standard be added to the sample extract prior to instrumental analysis. Responses of all analytes are calculated relative to the internal standard using the following relationship:

$$RR = \text{Relative Response} = \frac{\text{(Analyte Area)}}{\text{(Internal Standard Area)}}$$

If different volumes of the sample extracts and standard solutions are injected, these differences are reflected by differences in the internal standard area. In addition, if instrument sensitivity changes, these changes are also reflected in changes in the internal standard area. The calculation of the relative response corrects for these types of fluxuations.

In order to determine the analyte concentration-Relative Response relationship, analyte Relative Response Factors are calculated using the following relationship for data obtained from the analysis of standard solutions:

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$$\text{RRF - Relative Response Factor} = \frac{(\text{Analyte Area})\ (\text{Internal Standard Concentration})}{(\text{Internal Standard Area})\ (\text{Analyte Concentration})}$$

When identical amounts of the internal standard are added to both extracts and standard solutions, the internal standard concentration term in the above equation can be eliminated. When this is done and Relative Response is substituted for the ratio of analyte area to internal standard area, the RRF equation becomes:

RRF = RR/(Analyte Concentration)

Analyte concentrations in the extracts are calculated from the following equation:

Analyte Extract Concentration = RR/RRF

In order to obtain the concentration of the analyte in the sample, the following equation is used:

$$\text{Analyte Sample Concentration} = \frac{(\text{Analyte Extract Concentration})\ (\text{Extract Volume})}{(\text{Amount of Sample Extracted})}$$

The area terms in the above equations may be total ion areas, FID peak areas, EC peak areas, or extracted ion areas. The details of how the internal standard quantitation method is applied to specific methods are described in the various Instrumental Standard Analysis Methods.

The following equation shows what calculations should be made to correct for analytical bias:

$$\text{Analyte Sample Concentration (Corrected for Analytical Bias)} = \frac{(\text{Analyte Sample Concentration})}{(\text{Assumed Analyte Recovery})}$$

The Assumed Analyte Recovery can be obtained from an average of surrogate recoveries or native analyte recoveries, measured for the sample in question. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

External Standard Quantitation Method - Terminology which refers to a method of data analysis. This method requires that a known amount of the analyte standard be analyzed under the same conditions as the sample extract. In order to determine the analyte concentration-analyte response relationship, Response Factors are obtained from the following relationship:

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RF = Response Factor = (Analyte Area)/(Analyte Concentration)

Analyte concentrations in the extracts are calculated from the following equation.

Analyte Extract Concentration = (Analyte Area)/RF

In order to obtain the concentration of the analyte in the sample, the following equation is used:

$$\text{Analyte Sample Concentration} = \frac{(\text{Analyte Extract Concentration}) \quad (\text{Extract Volume})}{(\text{Amount of Sample Extracted})}$$

The area terms in the above equations may be total ion areas, FID peak areas, EC peak areas, or extracted ion areas. The details of how the external standard quantitation method is applied to specific methods are described in the various Instrumental Standard Analysis Methods.

The following equation shows what calculations should be made to correct for analytical bias:

$$\text{Analyte Sample Concentration (Corrected for Analytical Bias)} = \frac{(\text{Analyte Sample Concentration})}{(\text{Assumed Analyte Recovery})}$$

The Assumed Analyte Recovery can be obtained from an average of surrogate recoveries or native analyte recoveries, measured for the sample in question. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Standard Analysis Methods - Terminology used to reference an organized description of the steps involved in the instrumental analysis of a sample or a sample extract. These methods are assigned short names which can also be used as computer file names for these methods. The names of specific instrumental Standard Analysis Methods which are used in this study are listed in the final report in the section entitled "Details". In addition, copies of these Standard Analysis Methods are shown in a separate section of this report. Flow sheets of the Standard Analysis Methods are usually also provided in this section.

Analysis Flow Sheet - Terminology used to reference a graphic description of a Standard Analysis Method. This flow sheet gives specific information on how extracts and standards are analyzed and what types of data reduction methods are used.

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Further Information

The general area of Extract Analysis, along with much of the terminology described above, is very complex. References provided in the previous section entitled "Analyte Extraction Terminology" may provide further information which may be useful in understanding instrumental analysis fundamentals and terminology.

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