Monsanto

S&EH/ESC
(Co./Div./Dept./Location)

Analysis
(Type of Report)

REPORT NO.: MSL-5755
ESC-UAG-86-48

JOB/PROJECT NO.: 6221

DATE: June 6, 1986

TITLE: ANALYSIS OF TOTAL PCB CONGENERS IN KRUMMRICH SOIL SAMPLES

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

ABSTRACT: Nine soil samples were analyzed for total PCB congener concentrations. These analyses are required as a part of the Krummrich Plant cleanup of an area where chemicals had previously been stored.

TECHNICAL APPROVAL: Paul L. Sherman
P.L. Sherman
Environmental Sciences Center
Senior Research Group Leader

APPROVED BY: William M Mees
W.M. Mees
Environmental Sciences Center
Quality Assurance Specialist

APPROVAL DATE: 6-20-86

MONS 024131

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 the Company without proper authorization.

REPT. NO.: MSL-5755
ESC-UAG-86-48 AUTHORS: B.M. Hughes, D. McKenzie, and C. Trang
TITLE: ANALYSIS OF TOTAL PCB CONGENERS IN KRUMMRICH SOIL SAMPLES
COPY NO.:

R-1000(A)
R-1000(B)

DISTRIBUTION

COPY NUMBER

1. J. Gloeckner - 1740
2. B.M. Hughes - U4E
3. D. McKenzie - U4F
4. C. Trang - U1F
5. W.M. Mees - U4D
6. P.L. Sherman - U1F

7-9 Reports Library - R2C

10. Chesterfield Information Center - AA3A

ABSTRACT ONLY

H.A. Woltermann - U4E

COMPANY CONFIDENTIAL

This report has been assigned to you. When it is no longer needed, you are responsible for returning it to:

[ ]

If you transfer it to anyone else, please let your librarian know, so the records can be changed.

Time Stamp - <860605.1001>

ANALYSIS REPORT

DATE: June 6, 1986

LOG NUMBER: U-86-04-24-01 PROJECT NO: 760.28-6221

TO: Jim Gloeckner/Krummrich Plant

FROM:

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

Phone No: (314) 694-1464

Title: Analysis of Total PCB Congeners in Krummrich Soil Samples.

ABSTRACT

Krummrich Plant soil samples were analyzed for total PCB congener concentrations using capillary GC/EC analysis techniques. All samples contained total PCB congeners at levels less than 50 ug/g.

INTRODUCTION

Nine soil samples were analyzed for total PCB congener concentrations. These analyses are required as a part of the Krummrich Plant cleanup of an area where chemicals had previously been stored.

SUMMARY

Table 1 summarizes the results of capillary GC/EC analyses of 9 soil extracts. Note that all samples contained a total PCB concentration less than 50 ug/g. Table 1 also summarizes quality control experiments conducted on one blank soil sample. Table 2 summarizes results from quality control native spiking studies for blank soil and sample B-DD 0-2". See PCEX21 and PCFS21 for details of how the samples were extracted and cleaned up prior to GC/EC analysis. Refer to Standard Analysis Method ECAN02 and ECFS02 for the details of how the cleaned up extracts were analyzed. This information is included in a later section of this report.

The patterns of PCB congeners found in these soil samples were similar to the Aroclor 1262 pattern. However they were not identical. In a previous project for the Krummrich Plant (Project Number 760.28-6204), similar results were obtained for soil samples which were analyzed using GC/EC and GC/MS extract analysis methods. In that study, the PCB congener patterns were also not identical to Aroclor 1262. Therefore we have chosen to report data based upon this less expensive, yet technically equivalent, GC/EC analysis method for this present study.

(continued)

QC RESULTS

Quality control results are shown in Tables 1 and 2. These tables show good recoveries of surrogate compounds which were added to each sample before extraction. This information indicates how well each soil sample was extracted. Duplicate analyses of B-DD 0-2" and B-FF 0-18" are also shown in Table 1. Table 2 summarizes recoveries of Aroclor 1254 which was added to Blank Soil and B-DD 0-2". These data show that good PCB congener recoveries would be expected using these methods for the analysis of soils from the Krummrich Plant.

DETAILS

Standard Sampling Method No(s): Samples supplied by the Krummrich Plant.

Standard Extraction Method No(s): PCEX21 (See flow sheet PCFS21)

Standard Analysis Method No(s): ECAN02 (See flow sheet ECFS02)

Sample Identification(s): B-M 0-2", B-W 0-2", B-DD 0-2", B-EE 0-2", B-GG 0-2", B-P 0-18", B-W 18-36", B-DD 30-36", B-FF 0-18".

Final Report File No(s): A22118

Table File No(s): B22118, C22118

Also included in this report are:

1) Summaries of Standard Extraction and Analysis Methods used for this project.

2) Chain-of-custody information.

3) Analysis Request Information recorded at sample login.

4) Copies of sample extraction flow sheets for three samples.

5) A summary of important QA/QC, sample extraction and instrumental analysis terminology used in this project.

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

ANALYST APPROVAL: B. Mason Hughes

Table 1. Results of capillery GC/EC analyses of Soil samples using Standard Analysis Method ECAN02

ESC Project No: 760.28 - 6221 Date: May 28, 1986

Sample I.D.	Surrogate Recoveries(%Recovery)		PCB Concentration (ug/g)				
	CL5-benzene	2,4,6-CL3-biphenyl	A1248	A1254	A1260	A1262	Total
Method Blank	67	89	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
Soil Blank	52	72	"	"	"	"	"
B-M 0-2"	45	93	"	"	"	"	"
B-W 0-2"	150	94	"	"	"	1.42	1.42
B-DD 0-2"	78	86	"	"	"	ND(a)	ND(a)
B-DD 0-2" (DUP)	77	127	"	"	"	"	"
B-EE 0-2"	53	75	"	"	"	"	"
B-GG 0-2"	102	116	"	"	"	.32	.32
B-P 0-18"	48	82	"	"	"	1.00	1.00
B-W 18-36"	213	98	"	"	"	1.00	1.00
B-DD 30-36"	68	95	"	"	"	ND(a)	ND(a)
B-FF 0-18"	55	62	"	"	"	.30	.30
B-FF 0-18" (DUP)	43	63	"	"	"	.30	.30

Footnotes:

a. ND= None Detected (<1.0 ug/g)

TABLE FILE NO: B22118:MH

Table 2. Summary of Aroclor 1254 Spiking Concentrations and Recoveries in Quality Control Experiments for Soil Samples.

ESC Project No: 760.28 - 6221 Date: May 23, 1986

Sample I.D.	Surrogate Recoveries (%Recovery)		Aroclor 1254	
	CL5-benzene	2,4,6-CL3-Biphenyl	Amount Added (ug/g)	Recovery (%)
Soil Blank + L. SP	31	37	1.00	87
Soil Blank + H. SP	81	104	10.00	111
B-DD 0-2" + L. SP	59	98	1.00	107
B-DD 0-2" + H. SP	77	101	10.00	93

TABLE FILE NO: C22118:MH

MONS 024136

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

ESC Standard Extraction Method (SEM) | **ESC Standard Extraction Method No:**
TITLE: PCBs in Non-EC Active Matrices | **PCEX21**

MATRI(X),(CES):

Soils and other non-EC active solids and solvents

TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
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 that is non acid hydrolyzable

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.
Pierce vials.
Sulfuric acid at varying concentrations.
Ultrasonic bath.
Disposable glassware.
(See flow sheet for this extraction method in PCFS21.)

LIMITATIONS: Method applies only to matrices which do not contain interfering electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region.

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

DATES OF METHOD CREATION AND UPDATES: 12/17/84

FLOW SHEET PCFS21

FOR ESC EXTRACTION METHOD PCEX21

SAMPLE ID: ---------- MRC EXTRACT # ----------

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

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

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

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

ADD 10.0 mL BENZENE TO SAMPLE.	[ ]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[ ]	
PLACE 2 mL OF THE EXTRACT INTO A 7 mL VIAL	[ ]	
ADD 2 mL ------ SULFURIC ACID AND SHAKE	[ ]	
ALLOW PHASES TO SEPARATE REMOVE 1 mL FOR ANALYSIS.	[ ]	FINAL VOLUME ---------- 10.0 mL

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

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

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

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

MONS 024139

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

---

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: 10/25/83, 2/3/84 Time Stamp - <860605.0809>

FLOW SHEET ECFS02

FOR ESC ANALYSIS METHOD ECAN02

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 .
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. .
.
[ ]

CALCULATE RRFs FOR 10 - 30 PCB CONGENERS IN THE APPROPRIATE AROCLOR FORMULATION, USING THE AROCLOR CONCENTRATION AS THE ANALYTE CONCENTRATION FOR EACH PCB CONGENER.

CALCULATE CONCENTRATION OF AROCLOR FORMULATION BY AVERAGING THE VALUES DETERMINED FROM THE 10 - 30 PCB CONGENERS.

Where:

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

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

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

The above calculations are performed for 10 - 30 PCB congeners and the values averaged to yield an average Aroclor formulation concentration.

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

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

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

Monsanto

MONSANTO INDUSTRIAL CHEMICALS CO.
Seuget, Illinois 62201
Phone: (618) 271-5635

CHAIN OF CUSTODY

SAMPLING RECORD

Sampling Identification: B-MM 0"-2" B-P 0'-18"
B-NN 0"-2" B-NN 18"-36"
Log Number: B-OO 0"-2" B-OO 36"-56"
Date: B-FF 0"-2" B-FF 0"-18"
B-GG 0"-2"

Analyses Required: PCB

Sample Taken By: PENNY CHOU Date: 4-14/15/18-86

LABORATORY RECORD

Received By: Ken McEntire Date: 4-18-86

Relinquished To	Date:	Comments
[signature]	____	_____
_____	____	_____
_____	____	_____

REPORT OF ANALYSES

Date	Report No.	Author
_____	____	_____
_____	____	_____
_____	____	_____

INSTRUCTIONS

The person taking the sample completes the information in the Sampling Record area.

Each person to whom the sample is relinquished should sign the form and forward as necessary. Analyses performed should be listed with comments.

When all analyses are complete, the original should return to the "Received By" signature.

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

FLOW SHEET PCFS21

FOR ESC EXTRACTION METHOD PCEX21

SAMPLE ID: Method Blank ---------- MRC EXTRACT # PCB-1606

LOG NO: N.A.

EXTRACTION DATE and TIME: 4-29-86

SAMPLE COLOR AND DESCRIPTION -----

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[x]	SAMPLE WEIGHT 0.0000 g
SURROGATE SPIKE OF SAMPLE	[x]	100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)
ADD 10.0 mL BENZENE TO SAMPLE.	[x]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[x]	
PLACE 2 mL OF THE EXTRACT INTO A 7 mL VIAL	[x]	
ADD 2 mL 75 % SULFURIC ACID AND SHAKE	[x]	
ALLOW PHASES TO SEPARATE REMOVE 1 mL FOR ANALYSIS.	[ ]	FINAL VOLUME 10.0 mL

Note - 1 mL of the extract was subjected to the multiphase column on PCFS03.

EXTRACT NO: PCB-1606

DATE/TIME COMPLETED 4-30-86 / 3:00

ANALYST: McKenzie

ESC JOB NO: 760.28-6221

FLOW SHEET PCFS21

FOR ESC EXTRACTION METHOD PCEX21

SAMPLE ID: Blank Soil ---------- MRC EXTRACT # PCB-1607

LOG NO: N.A.

EXTRACTION DATE and TIME: 4-29-86

SAMPLE COLOR AND DESCRIPTION: Brown Soil

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[x]	SAMPLE WEIGHT: 1.1580 g
SURROGATE SPIKE OF SAMPLE	[x]	100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)
ADD 10.0 mL BENZENE TO SAMPLE.	[x]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[x]	
PLACE 2 mL OF THE EXTRACT INTO A 7 mL VIAL	[x]	
ADD 2 mL 75 % SULFURIC ACID AND SHAKE	[x]	
ALLOW PHASES TO SEPARATE REMOVE 1 mL FOR ANALYSIS.	[ ]	FINAL VOLUME: 10.0 mL

Note - 1 mL of the extract was subjected to the multiphase column on PCFS03.

EXTRACT NO: PCB-1607

DATE/TIME COMPLETED: 4-30-86 / 3:00

ANALYST: McKenzie

ESC JOB NO: 760.28-6221

MONS 024146

FLOW SHEET PCFS21

FOR ESC EXTRACTION METHOD PCEX21

SAMPLE ID: B-M, 0-2" ---------- MRC EXTRACT # PCB-1610

LOG NO: U-86-04-24-01

EXTRACTION DATE and TIME: 4-29-86

SAMPLE COLOR AND DESCRIPTION grey wet sandy rocks

WEIGH OUT SAMPLE INTO A 14 OR 40 mL PIERCE VIAL	[x]	SAMPLE WEIGHT 1.1506 g
SURROGATE SPIKE OF SAMPLE	[x]	100 uL DEM-050885-01 (10 ug/mL + 20 ug/mL)
ADD 10.0 mL BENZENE TO SAMPLE.	[x]	
ULTRASONICATE SAMPLE FOR 1/2 HOUR IN A SOUND BATH.	[x]	
PLACE 2 mL OF THE EXTRACT INTO A 7 mL VIAL	[x]	
ADD 2 mL 75 % SULFURIC ACID AND SHAKE	[x]	
ALLOW PHASES TO SEPARATE REMOVE 1 mL FOR ANALYSIS.	[ ]	FINAL VOLUME 10.0 mL

Note - 1 mL of the extract was subjected to the multiphase column on PCFS03.

EXTRACT NO: PCB-1610

DATE/TIME COMPLETED 4-30-86 / 3:00

ANALYST: McKenzie

ESC JOB NO: 760.28-6221

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

Time Stamp - <860605.1359>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 4-23-86 LOG NO: U-86-04-24-01

REQUESTER: Krummrich/Gloekner PROJECT NO: 760.28-6221

LOGIN NOTEBOOK PAGE: 20

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

ANALYSIS REQUESTS SENT TO: FILES, Mees, Hughes, Sherman, Hughes

SAMPLE PICKUP BY: McKenzie

SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 9 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
Method Blank		PCB-1606
Blank Soil		PCB-1607
Blank soil + Low level native spike		PCB-1608-LS
Blank soil + high level native spike		PCB-1609-HS
B-M, 0-2"	1 X 4 oz	PCB-1610 PCB-1624-NS
B-W, 0-2"	1 X 4 oz	PCB-1611, PCB-1626-NS
B-DD, 0-2"	1 X 4 oz	PCB-1612, PCB-1613-DUP PCB-1614-LS, PCB-1615-HS
B-EE, 0-2"	1 X 4 oz	PCB-1616
B-GG, 0-2"	1 X 4 oz	PCB-1617, PCB-1625-NS\
B-P, 0-18"	1 X 4 oz	PCB-1618, PCB-1623-NS
B-W, 18-36"	1 X 4 oz	PCB-1619
B-DD, 30-36"	1 X 4 oz	PCB-1620
B-FF, 0-18"	1 X 4 oz	PCB-1621, PCB-1622-DUP

Note: DUP refers to duplicate analyses, LS refers to low level native spike, HS refers to high level native spike, and NS refers to no surrogate spike. The samples which were worked up with no surrogate spike were done in order to asses if the surrogate spiking compounds used (pentachlorobenzene and 2,4,6-trichlorobiphenyl) were present in the matrices.

RECORDED BY: McKenzie

REQUIRED ANALYSES: PCBs

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158608

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
x GC/EC (18)	6/6/86	ECAN02	No. 157258

FINAL REPORT NOTEBOOK PAGE: 3,434,403

QUALITATIVE: x SEMIQUANTITATIVE: QUANTITATIVE: x

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: -- HOLD FOR 60 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA221:LI

MONS 024150

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 Bill Mees (U4E) METS 544-1499.

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 unduely 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 proceedures 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.

(continued)

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.

(continued)

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.

(continued)

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.

(continued)

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 imperative that data be obtained which gives the analysis requestor information concerning what percentage of the 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 analysts who use them. If cost were no object, analyte recovery information could be obtained by the use of standards addition analysis methods. However, 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 least three times.) Surrogate Standards give the same type of information, with only one analysis being required.

A "Surrogate" Standard is a compound which is chosen to behave 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 the 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 the 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 confidence 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 surrogates 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.

(continued)

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 chemically identical 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 analysis what analytes may be present, surrogates must be chosen that could not possibly be present 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 as a surrogate. Normally, phenol-d5, 2-CL-phenol-d4, CL2-benzene-d4, pyrene-d10 and 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 are 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 as the polychlorinated biphenyls (PCBs) in the Aroclor formulations. Any halogenated compound can be used which acts exactly as PCBs in the extraction and clean-up step. ESC normally uses CL5-benzene and 2,4,6-CL3-biphenyl for this purpose.

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.

(continued)

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.

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.

Statement of Work for the Contract Laboratory Program 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

ANEXTE:MH

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:

(continued)

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

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:

(continued)

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.

(continued)

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.

INANTE:MH

MONS 024163