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TITLE: Analysis of Two St. Charles Fire Testing Facility Soil Samples for PCBs

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

ABSTRACT: Two soil samples from the St. Charles Fire Testing Facility were analyzed for PCB formulations. Both samples were analyzed in duplicate. Both samples contained Aroclor 1248 in detectable quantities. Results of Therminol Loop sample analyses were 76 and 17 ug/g. Results of Burn Lab Area sample analyses were 24 and 10 ug/g. During sample preparation it was noted that samples were not homogeneous, which explains the differences in duplicate analysis values. These results were generated in order to assist Research Safety in the determination of proper waste disposal methodology.

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APPROVAL DATE: March 4, 1988

MONS 024922

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AUTHORS: B.M. Hughes, D.E. McKenzie, L.R. Minor
TITLE: Analysis of Two St. Charles Fire Testing Facility Soil Samples for PCBs

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ANALYSIS REPORT

DATE: February 19, 1988

LOG NUMBER: U-88-02-17-01 PROJECT NO: 760.28-8603

TO: Randy Parsons
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Title: Analysis of Two St. Charles Fire Testing Facility Soil Samples for PCBs.

ABSTRACT

Two soil samples from the St. Charles Fire Testing Facility were analyzed for PCB formulations. Both samples were analyzed in duplicate. Both samples contained Aroclor 1248 in detectable quantities. Results of Therminol Loop sample analyses were 76 and 17 ug/g. Results of Burn Lab Area sample analyses were 24 and 10 ug/g. During sample preparation it was noted that samples were not homogeneous, which explains the differences in duplicate analysis values. These results were generated in order to assist Research Safety in the determination of proper waste disposal methodology.

INTRODUCTION

Two St. Charles Fire Testing Facility soil samples were analyzed for PCBs. The samples were analyzed using standard extraction method PCEX02, which is included in a later section of this report.

The method involves weighing the samples (1g) into a 14 mL Pierce vial and spiking with surrogate spiking compounds. Benzene (10 mL) was added to each sample, and the samples were placed in an ultrasonic bath for 1/2 hour. Extracts from these samples were analyzed using internal standard methodology which is described in Standard Analysis Method ECAN06. Standard Analysis Method ECAN06 is located in a later section of this report.

Both samples were analyzed in duplicate. In addition, Aroclor 1254 was added to two separate aliquots of the Therminol Loop Area sample at concentrations corresponding to 2 and 10 ug/g. A method blank consisting of blank soil was also included in these analyses.

Aroclor 1248 was detected in both samples, and was quantitated using a Supelco Aroclor 1248 standard. Results are located in Table 1. All samples were

spiked with surrogate spiking compounds (pentachlorobenzene and 2,4,6-trichlorobiphenyl). Surrogate recoveries are shown in Table 1 and 2. The results of Aroclor 1254 native additions are located in Table 2. Because of the relatively high concentration of Aroclor 1248 present in the samples, recovery for the low level native spike is very high and recovery for the high level native spike is also high. This is not unusual since these two Aroclor formulations chromatographically overlap. As a result, the level of detection was determined visually to be approximately 2 ug/g. This value is approximately 20 times the signal to noise ratio.

SUMMARY

Two St. Charles Fire Testing Facility soil samples were analyzed for PCBs at the request of Research Safety. Results are to be used for the determination of proper disposal methodology.

Both samples contained detectable amounts of Aroclor 1248. Results of sample analysis are located in Table 1. Aroclor 1248 concentrations are listed in ug/g and surrogate spiking compound recoveries are listed adjacent to the respective sample in units of percent recovery. The results of quality control experiments are shown in Table 2.

Results of duplicate analysis for the Therminol Loop Area sample were 76 and 17 ug/g. Results of duplicate analysis for the Burn Lab Area sample were 24 and 10 ug/g. This variability of duplicate analyses is thought to arise from the obvious sample inhomogeneity noted during sample preparation.

Since the results of these analyses are to be used to determine proper disposal methodology, several factors should be included in the interpretation of the results. These are:

1) Since the samples are obviously not homogeneous, concentration may vary drastically from position to position at the St. Charles Test Burn Facility and therefore the present samples may not be representative of large areas of the site.

2) If additional analyses are requested the relative size of the area should be taken into account so that an appropriate number of samples are analyzed.

QC RESULTS

The present section summarizes QA/QC results for this set of samples. Quality Assurance and Quality Control studies are conducted upon each set of samples which ESC is requested to analyze. The purpose of these studies are to determine the validity of the reported results so that the analysis requester can better understand what limitations should be placed upon these results. The following paragraphs describe what experiments were conducted to estimate the precision and accuracy of the reported results, whether the data exhibit positive or negative bias, and what provisions have been made to assure that no false positive or negative values are reported.

Precision. The precision of the data reported in the tables can be estimated from the results of analysis of duplicate samples which contain analytes above the level of detection and from the results of spiking the samples with surrogate and native compounds. Table 1 reports the results of a duplicate analysis in which compounds were present above the level of detection and both tables report the percent recovery of surrogate compounds which are added to each sample before analysis. As mentioned before the major factor effecting

the precision of these analyses was sample inhomogeneity. Both samples were analyzed in duplicate and Aroclor formulation concentrations were measured to be 76 and 17 ug/g for the Therminol Loop Area sample and 24 and 10 ug/g for the Burn Lab Area sample. These concentrations indicate between 200 and 500% variability in the analysis method. However, another estimate of the analytical precision may be obtained from the recoveries of the surrogate spiking compounds (pentachlorobenzene and 2,4,6-trichlorobiphenyl) which were added to each sample prior to extraction. All pentachlorobenzene recoveries, except for the blank soil, were high which indicates the presence of pentachlorobenzene or a chromatographic interferent in the samples. The recoveries for 2,4,6-trichlorobiphenyl were, however, on the order of 100%. These values are listed in Tables 1 and 2 and statistical summaries for all surrogate compound recoveries are tabulated in Table 1. Analytical precision using the the recoveries of 2,4,6-trichlorobiphenyl is on the order of plus or minus 7% for these analyses. However total analytical precision with respect to sample inhomogeniety is on the order of plus or minus 500%. NOTE!! It is the responsibility of the analysis requester to review these these data and draw his/her own conclusions concerning the precision of these determinations, and, more importantly, if the stated precision of the determination meets his/her needs.

Accuracy. The accuracy of the data reported in the tables can be estimated from the results of spiking the samples with surrogate and native compounds. Unfortunately, the low and high level native Aroclor 1254 spike conducted on these samples revealed very little since the amount of Aroclor 1248 present in these samples essentially covered up the Aroclor 1254 which was added. These data are listed in Table 2, primarily for completeness. Also the presence of either pentachlorobenzene or a chromatographic interference in the samples essentially nullified pentachlorobenzene as a useful surrogate compound. 2,4,6-Trichlorobiphenyl was also added to each sample as a surrogate spiking compound. Tables 1 and 2 list the recoveries of each of these surrogate. Based on the recovery of 2,4,6-trichlorobiphenyl in all the samples, the accuracy of these analyses can be judged to be better than 30% Table 1 gives a statistical summary for all surrogate recoveries. Another estimation of accuracy of analysis may be obtained setting up a linear array for each formulation analyzed by quantitating the named formulation on each peak and deriving average concentrations from the average of all of the named peaks. All samples were analyzed using a total of 19 named peaks. These outputs are included in the Raw Data Section of this report. This latter data also indicates that the accuracy of analysis is better than 30%.

NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the analysis requester to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Bias. The Recovery Means for surrogate and native recoveries, shown in Table 1 for 2,4,6-trichlorobiphenyl are within 5% of 100%, and therefore there are no significant biases introduced into these analyses. NOTE!! No data reported in this report includes any correction for this bias. It is the responsibility of the analysis requester to apply appropriate bias corrections if he/she feels that such corrections are warranted.

False Positives. The analyses of Blank Soil shown in Table 1 shows that the analytical system introduces no false positive values. The results of analysis of native spiked samples (shown in Table 2) are inconclusive since the spiking levels of 2 and 10 ug/g were overshadowed by the amount of Aroclor 1242 present in the samples.

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 Flow Sheet ECFS06)

Sample Identification(s): Therminol Loop Area and Burn Lab Area

Final Report File No(s): RB8603

Table File No(s): TC8603 and TD8603

Also included in this report are:

1) Brief Descriptions of Standard Extraction and Analysis Methods used in this study.
2) Raw Data outputs including chromatograms of samples, standards, and GC/EC analysis outputs for samples.
3) Chain-of-Custody 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. M. Hughes, D. E. McKenzie, and L. R. Minor

ANALYST APPROVAL: B. M. Hughes

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

ESC Project No: 760.28 - 8603 Date: February 19, 1988

SAMPLE I.D.	EXTRACT #	LOD(a) (ug/g)	AROCLOR 1248 DETECTED (ug/g)	SURROGATE COMPOUNDS PERCENT RECOVERY CL5 (c)	CL3 (d
Therminol Loop Area	PCB-2278	2	76	216	116
Therminol Loop Area (duplicate)	PCB-2279	2	17	162	103
Burn Lab Area	PCB-2282	2	24	231	99
Burn Lab Area (duplicate)	PCB-2283	2	10	129	102
Blank Soil	PCB-2277	2	ND(d)	98	100

Surrogate Recovery Tabulation (e)

Average of Surrogate Spiking Compound Recovery	222	105
Standard Deviation of Surrogate Spiking Compound Recovery	161	7
Minimum of Surrogate Spiking Compound Recovery	98	99
Maximum of Surrogate Spiking Compound Recovery	570	116

Footnotes:

a. LOD = limit of detection
b. CL5 = Pentachlorobenzene used as a surrogate spiking compound.
c. CL3 = 2,4,6,-trichlorobiphenyl used as a surrogate spiking compound.
d. ND= No Aroclor formulations detected (< 2 ug/mL). Formulations include: Aroclors; 1232, 1242, 1248, 1254, 1260, and 1262.
e. Statistics also include surrogate spiking compound recoveries listed in Table 2.

TABLE FILE NO: TC8603:mh

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

ANALYST APPROVAL: David McKenzie

MONS 024928

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

ESC Project No: 760.28 - 8603 Date: February 19, 1988

Sample I.D.	Extract #	Aroclor 1254 Amount Added (ug/g)	Aroclor 1254 Recovery (%)	Surrogate Compounds Percent Recovery CL5 (a)	Surrogate Compounds Percent Recovery CL3 (b)
Therminol Loop Area + LS	PCB-2280	2	2351	570	113
Therminol Loop Area + HS	PCB-2281	10	171	146	103

TABLE FILE NO: TD8603:mh

Footnotes:

a. CL5 = Pentachlorobenzene used as a surrogate spiking compound.
b. CL3 = 2,4,6-trichlorobiphenyl used as a surrogate spiking compoud.

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

ANALYST APPROVAL: David McKenzie

MONS 024929

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 024931

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

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

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

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 congeners 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 024933

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

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TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Aroclor formulations 1232, 1242, 1248, 1254, 1260, and 1262	0.1	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl through octachlorobiphenyl 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: 140(1)/5/300 INJECTION TYPE: Splitless

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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 - <880217.1346>

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

CONCENTRATIONS OF NAMED COMPOUNDS IN SAMPLES

SAMPLE: PCB-2279
UNITS: UG/G
FILE: BP733
P FILE: DUMRH :RH

	AMOUNT	DEV. FROM AVG	PCT DEV FROM AVG
ACL6 BENZENE	(2.07102)	8.75347	52.0281
PK1	10.6525	5.17202	36.6848
PK2	(33.7826)	-18.9582	-100.794
PK3	11.3784	5.47107	32.5185
PK4	(2.93698)	13.8975	82.6028
PK5	25.3845	-8.54004	-50.7596
PK6	14.6933	2.13116	12.667
PK7	15.9167	.907833	5.3959
PK8	17.2737	-.449413	-2.67119
PK9	18.5026	-1.6781	-9.97416
PK10	18.4255	-1.60104	-9.51615
PK11	22.7122	-5.8877	-34.9938
PK12	14.7384	2.08613	12.3994
PK13	(7.89746)	9.72703	57.8147
PK14	19.0765	-2.25197	-13.3851
PK15	16.5555	.268948	1.59885
PK16	17.4808	-.656265	-3.90055
PK17	21.6668	-4.84228	-28.7811
PK18	17.4628	-.638264	-3.79366
PK19	15.5647	1.25981	7.48793
ODCN	8.57634	8.24815	49.0247

Not include in calculation

NUMBER OF NAMED PEAKS: 21
TOTAL NUMBER OF PEAKS: -53
PCT NAMED VS. TOTAL: -39.6226

AVG. OF VALID VALUES: 16.8245 2.44833E-06 WITH A FACTOR OF 2
RANGE OF VALID VALUES: 17.2935
STD. DEV OF VALID VALUES: 4.25714
% REL STD DEV OF VALID VALUES: 25.3032
NUMBER OF VALID VALUES: 17

MONS 024936

CONCENTRATIONS OF NAMED COMPOUNDS IN SAMPLES

SAMPLE: PCB-2270
UNITS: UG/G
FILE: BP732
M FILE: DUMRH :RH

*** 28.5714 % DATA REJECTION ***

	AMOUNT	DEV. FROM AVG	PCT DEV FROM AVG
SCL6 BENZENE	(9.4697)	66.4836	87.5322
PK1	(15.8159)	60.1374	79.1768
PK2	116.335	-40.3814	-53.1661
PK3	(22.2582)	53.6951	70.6949
PK4	(12.0517)	63.9016	84.1328
PK5	66.1163	9.83696	12.9513
PK6	58.901	17.0523	22.451
PK7	68.0805	7.87276	10.3653
PK8	69.347	6.60628	8.69781
PK9	78.5831	-2.62981	-3.4624
PK10	78.1176	-2.16434	-2.84956
PK11	85.2457	-9.29239	-12.2343
PK12	68.4236	7.52974	9.91364
PK13	(21.3696)	54.5837	71.8648
PK14	77.438	-1.48473	-1.95479
PK15	72.9984	2.95493	3.89045
PK16	83.0257	-7.07242	-9.31154
PK17	83.4864	-7.53311	-9.91809
PK18	71.8755	4.07774	5.36875
PK19	61.3253	14.628	19.2592
#OCN	(10.4539)	65.4994	86.2364

Not include in calculation

NUMBER OF NAMED PEAKS: 21
TOTAL NUMBER OF PEAKS: -91
PCT NAMED VS. TOTAL: -23.0769

AVG. OF VALID VALUES: 75.9533 3.00090E-05 WITH A FACTOR OF 2
RANGE OF VALID VALUES: 106.865
STD. DEV OF VALID VALUES: 13.6977
% REL STD DEV OF VALID VALUES: 18.0344
NUMBER OF VALID VALUES: 15

MONS 024937

CONCENTRATIONS OF NAMED COMPOUNDS IN SAMPLES

SAMPLE: PCB-2282
UNITS: UG/G
FILE: BP736
M FILE: DUMRH :RH

*** 23.809% % DATA REJECTION ***

	AMOUNT	DEV. FROM AVG	PCT DEV FROM AVG
ACL6 BENZENE	(9.74659)	13.8799	58.7472
PK1	(5.96747)	17.659	74.7425
PK2	38.6968	-15.0703	-63.7855
PK3	(6.6724)	16.9541	71.7588
PK4	(2.24796)	21.3786	90.4855
PK5	19.5605	4.06603	17.2096
PK6	19.1071	4.51942	19.1286
PK7	20.2934	3.33307	14.1073
PK8	18.9598	4.66671	19.752
PK9	22.7073	.919167	3.8904
PK10	23.0515	.57502	2.43379
PK11	23.7117	-8.51822E-02	-.360536
PK12	21.9356	1.69094	7.15695
PK13	28.9869	-5.36039	-22.688
PK14	25.2862	-1.65969	-7.02468
PK15	21.3453	2.28125	9.65545
PK16	23.2239	.402637	1.70418
PK17	28.6497	-5.02317	-21.2607
PK18	22.8696	.756893	3.20358
PK19	19.6388	3.98767	16.8779
ΦOCN	(9.8134)	13.8131	58.4644

Not include in calculation

NUMBER OF NAMED PEAKS: 21
TOTAL NUMBER OF PEAKS: -56
PCT NAMED VS. TOTAL: -37.5

AVG. OF VALID VALUES: 23.6265 4.52995E-06 WITH A FACTOR OF 2
RANGE OF VALID VALUES: 28.9502
STD. DEV OF VALID VALUES: 5.03033
% REL STD DEV OF VALID VALUES: 21.291
NUMBER OF VALID VALUES: 16

MONS 024938

CONCENTRATIONS OF NAMED COMPOUNDS IN SAMPLES

SAMPLE: PCB-2283
UNITS: UG/G
FILE: BP737
M FILE: DUMRH :RH

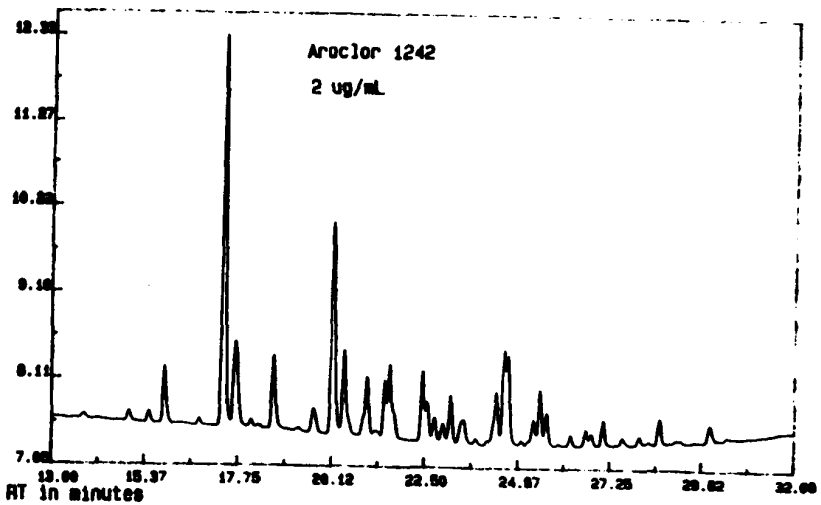
	AMOUNT	DEV. FROM AVG	PCT DEV FROM AVG
SCL6 BENZENE	(8.83392)	.849806	8.77561
[illegible]	(.933386)	8.75034	90.3613
[illegible]	11.763	-2.0793	-21.4721
[illegible]	(3.02829)	6.65544	68.7281
[illegible]	(1.90298)	7.78075	80.3487
[illegible]	7.34028	2.34345	24.1999
[illegible]	7.[illegible]	1.93915	20.0248
[illegible]	8.[illegible]	1.25795	12.9901
[illegible]	9.[illegible]	.663514	6.87043
PK10	8.[illegible]	1.46277	15.1022
PK11	7.82[illegible]	1.8592	19.1992
PK12	7.[illegible]	1.87027	19.3135
PK13	12.1362	-2.4565	-25.3673
PK14	11.6162	-2.13442	-22.0415
PK15	9.51396	.169765	1.7531
PK16	8.69826	.985466	10.1765
PK17	13.7762	-4.09247	-42.2613
PK18	11.[illegible]4	-1.55769	-16.0857
PK19	10.8976	-.414024	-4.27546
#OCN	9.49[illegible]	.191071	1.97311

Not included in calculation

NUMBER OF NAMED PEAKS: 20
TOTAL NUMBER OF PEAKS: -42
PCT NAMED VS. TOTAL: -47.619

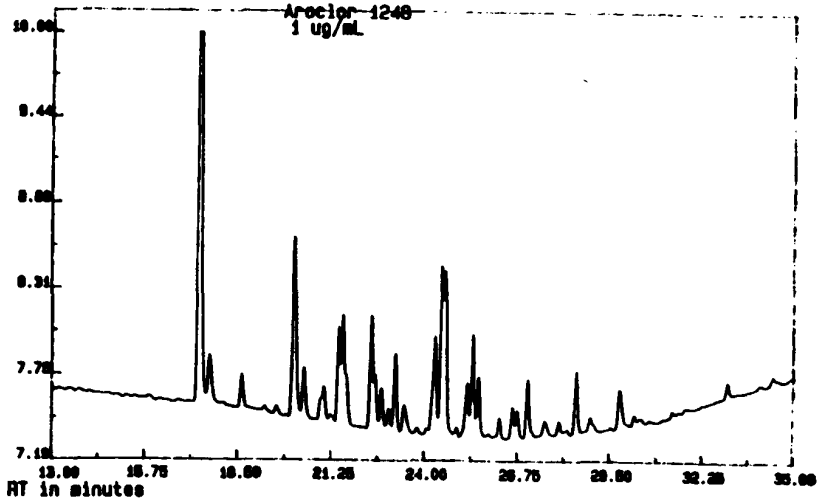
AVG. OF VALID VALUES: 9.68373 -1.668935-06 WITH A FACTOR OF 2
RANGE OF VALID VALUES: 6.43592
STD. DEV OF VALID VALUES: 1.92594
% REL STD DEV OF VALID VALUES: 19.8884
NUMBER OF VALID VALUES: 16

MONS 024939



SAMPLE: A1242(2) INJECTED AT 15:45:27 ON FEB 17, 1988
Meth: PCBSSW Raw: BR726 Proc: BP726

MONS 024940



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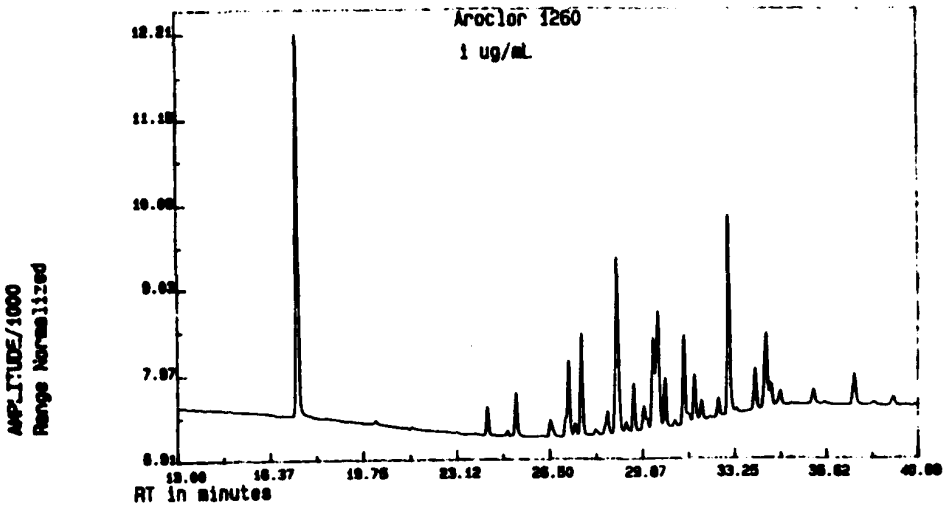
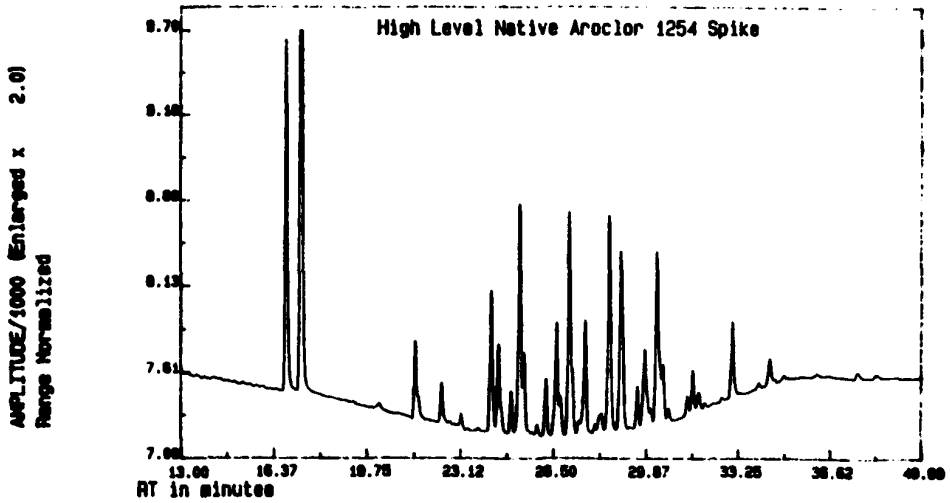
Meth: PCBSSW Raw: BR727 Proc: BP727

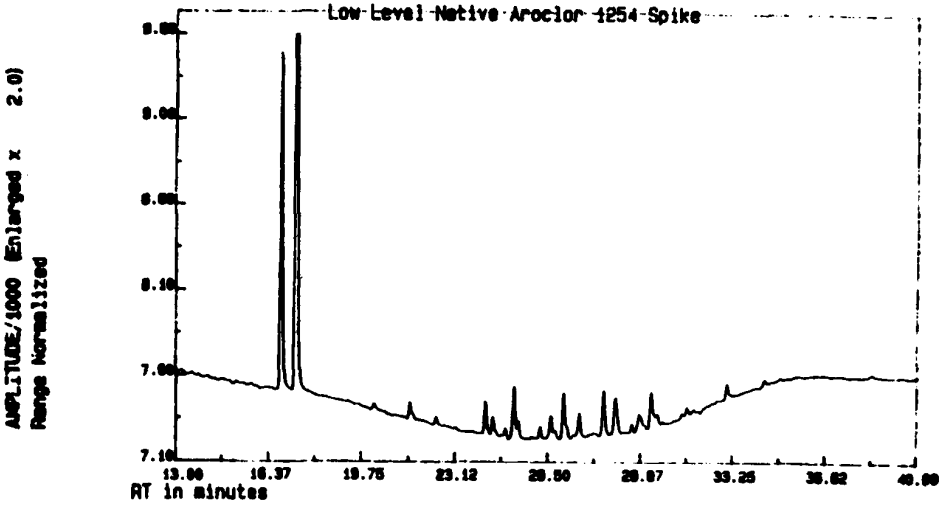


SAMPLE: A1260 (1) INJECTED AT 17: 49: 49 ON FEB 17, 1988
Meth: PCBSSM Raw: BR728 Proc: BP728

MONS 024942

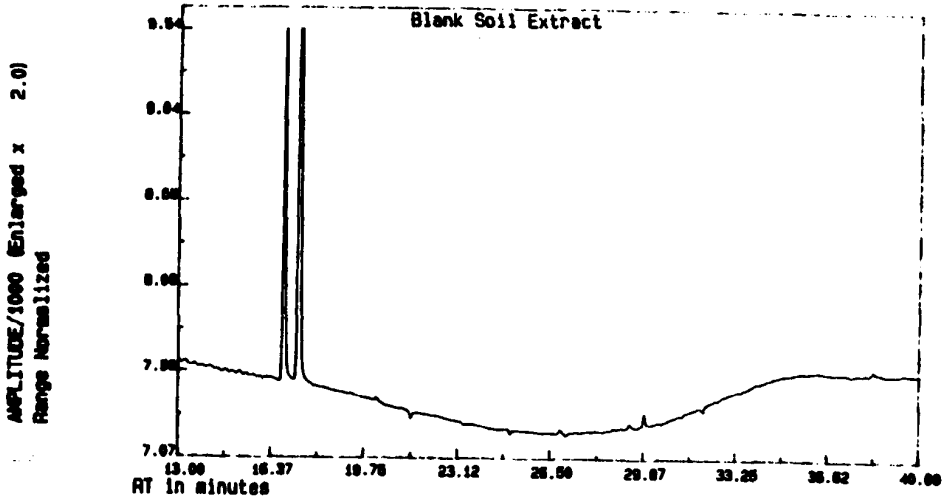


SAMPLE: MS SPK INJECTED AT 18:51:50 ON FEB 17, 1988
Meth: PCBSSM Raw: BR729 Proc: BP729

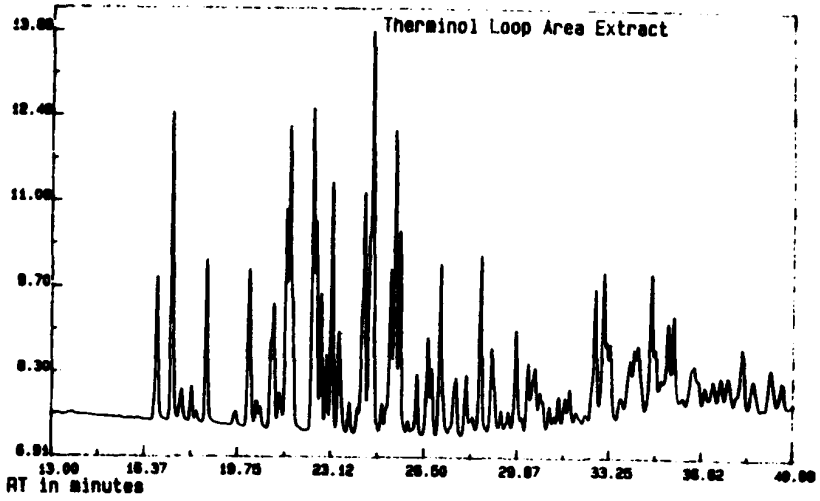


SAMPLE: LS SPK INJECTED AT 19:54:17 ON FEB 17, 1988
Meth: PCBSSM Raw: BR730 Proc: BP730

MONS 024944

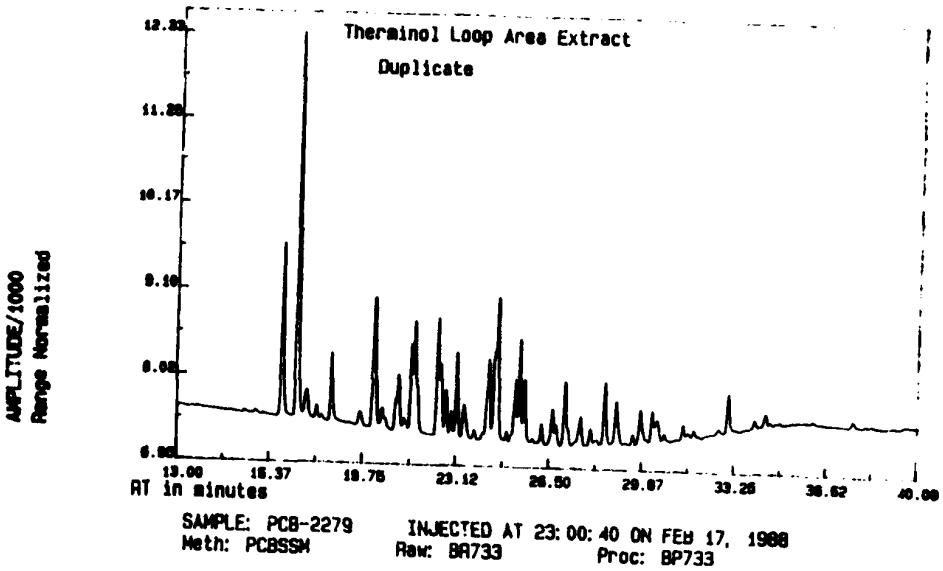


SAMPLE: PCB-2277 INJECTED AT 20:56:22 ON FEB 17, 1988
Meth: PCBSSM Raw: BR731 Proc: BP731

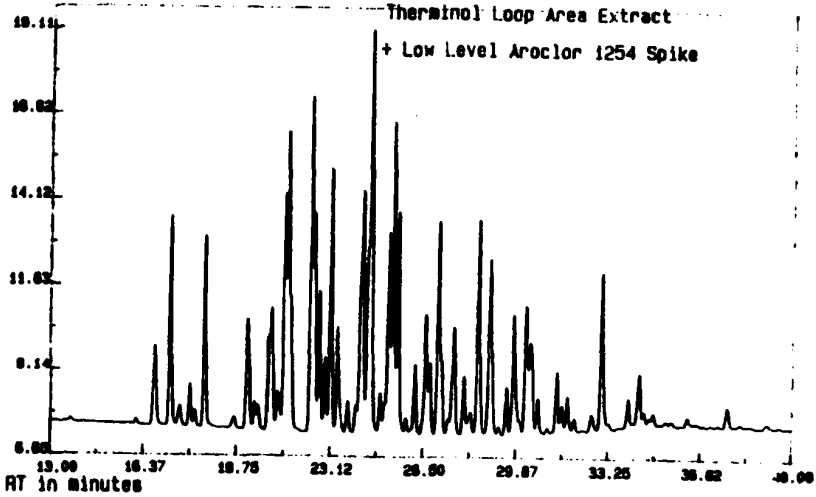


SAMPLE: PCB-2278 INJECTED AT 21:58:30 ON FEB 17, 1988
Meth: PCBSSM Raw: BR732 Proc: BP732

MONS 024946

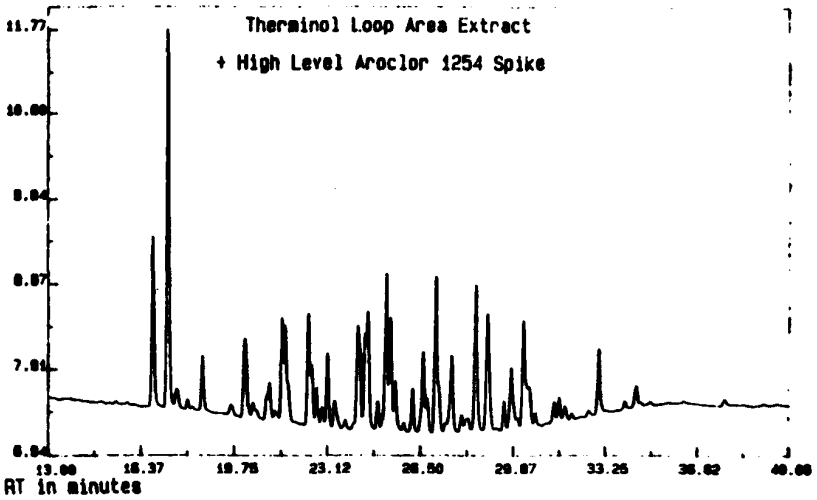

Therminol Loop Area Extract
Duplicate
AMPLITUDE/1000
Range Normalized
12.33
11.25
10.17
9.10
8.02
6.95
13.00
16.37
19.75
23.12
26.50
29.87
33.25
36.62
40.00
RT in minutes
SAMPLE: PCB-2279
Meth: PCBSSM
INJECTED AT 23:00:40 ON FEB 17, 1988
Raw: BR733
Proc: BP733

MONS 024947



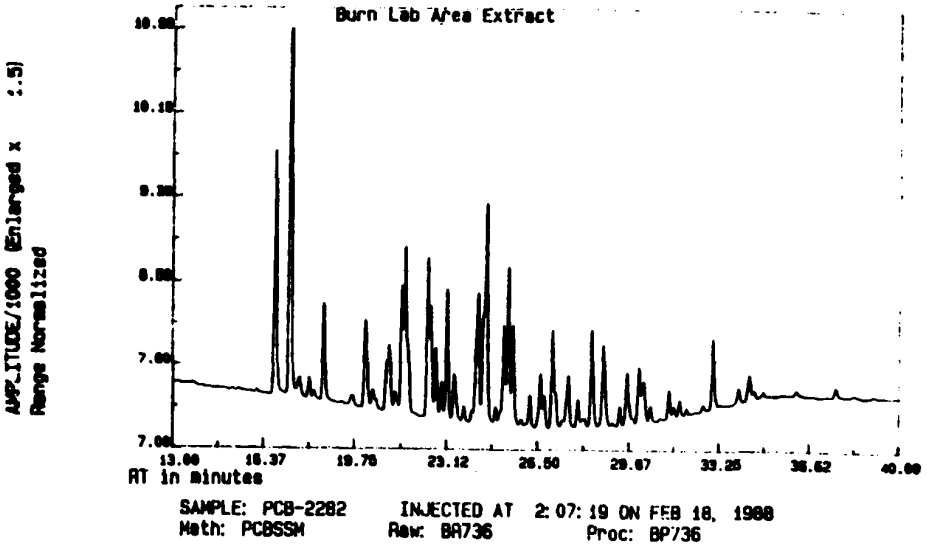
SAMPLE: PCB-2280 INJECTED AT 0:02:52 ON FEB 18, 1988
Meth: PCBSSM Raw: BR734 Proc: BP734

MONS 024948

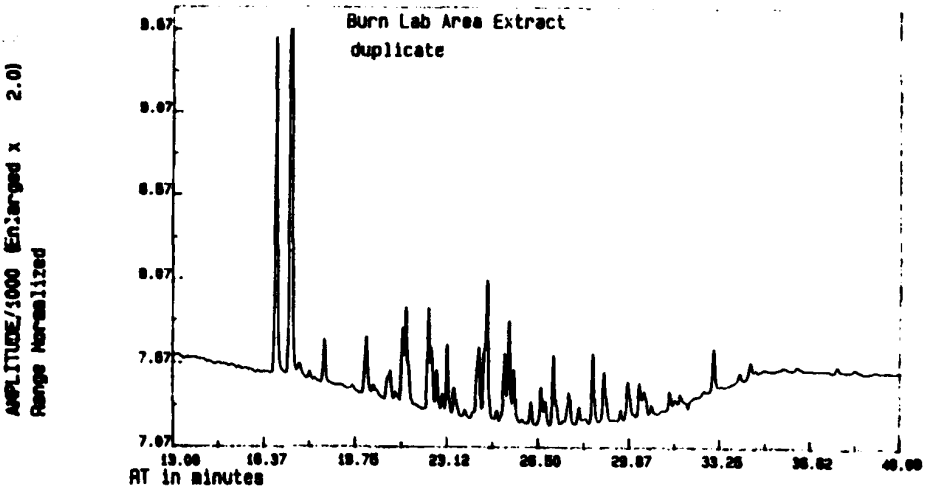


SAMPLE: PCB-2281 INJECTED AT 1:05:06 ON FEB 18, 1988
Meth: PCBSSM Raw: BR735 Proc: BP735

MONS 024949



SAMPLE: PCB-2282 INJECTED AT 2:07:19 ON FEB 18, 1988
Meth: PCBSSM Raw: BA736 Proc: BP736



SAMPLE: PCB-2283 INJECTED AT 3:09:40 ON FEB 18, 1988
Meth: PCBSSM Raw: BR737 Proc: BP737

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

MONSANTO MEMO

FROM: Randy Parson, Environmental Services, W 101, 4-1282
cc: R. J. Edwards R3B

DATE: February 16, 1988

SUBJECT: Samples from St. Charles/Fire Testing Facility

TO: Mr. Mason Huges U4E

The enclosed samples are from the soil underneath the rooms which housed the Therminol Loop & the Fire Testing Equipment at the St. Charles Fire Testing Facility. Could you please analyse the samples for PCB as soon as possible.

The charge number for this project is 83.000.760.55-265.006. If you need more information or have any questions about the samples, don't hesitate to call me at 4-1282.

Thanks again.

Randy Parson

Randy Parson

MONS 024953

Received
2-17-88

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

Time Stamp - <880219.1104>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 2-17-88 LOG NO: U-88-02-17-01

REQUESTER: RANDY PARSONS PROJECT NO: U-88-02-17-01

LOGIN NOTEBOOK PAGE: 3759243

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

ANALYSIS REQUESTS SENT TO: LEE, SHERMAN, HUGHES, MCKENZIE

SAMPLE PICKUP BY: DAVID MCKENZIE

SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 2 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
BURN LAB AREA	1 X 40 ML	PCB-2282, PCB-2283-DUP
THERMINOL LOOP AREA	1 X 40 ML	PCB-2278, PCB-2279-DUP PCB-2280+LS, PCB-2281+HS
Blank Soil		PCB-2277

NOTE: DUP refers to duplicate analysis, +LS refers to a low level addition of Aroclor 1254, and + HS refers to a high level addition of Aroclor 1254.

RECORDED BY: DAVID MCKENZIE

REQUIRED ANALYSES: PCBS

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158644

ESC STANDARD EXTRACTION METHOD(S): PCEX02

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
-x- GC/EC	2-18-88	ECAN06	157308

FINAL REPORT NOTEBOOK PAGE: 3440460

OTHER NOTEBOOK PAGES:

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: HOLD FOR 90 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AB8603

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.

MONS 024957

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.

(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 homogeneity 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 assssing 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 negative bias means that values below the correct value are normally measured. A positive 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 negative bias is introduced when the sample is not completely extracted and the extract does not contain 100% of the analyte. A positive 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.

False Positives - Terminology used to indicate the <u>incorrect</u> reporting of the presence of compounds. In many cases, the source of these false positive values can be traced to the analysis of very high level standards or samples immediately prior to the sample in which positive values are reported. In order to assure against reporting false positives, attempts are made to inject cleaner samples before dirtier samples. When high level components are detected in samples immediately prior to low level samples, these lower level samples are rerun so that it can be documented that the positive values are real. In addition, organic free water and other similar materials which are known to be organic free are used to document that there are no other unknown sources of contamination which would cause the reporting of false positives.

False Negatives - Terminology used to indicate the <u>incorrect</u> reporting of the absence of targetted compounds. The source of false negatives in many instances is due to masking of the compounds of interest by the sample matrix or from instrument malfunction. In order to determine, experimentally, that all components in all samples have been reported if they were present above a certain level of detection, a representative sample of each matrix type is spiked with the analytes at the estimated level of detection and at ten times the estimated level of detection. The recoveries of these compounds are then measured for these two native spiking studies. If recoveries between 50 and 150 % are measured in these two studies, then the lower level native spike is reported to be the level of detection for the determination. If recoveries in this range can only be reported for the higher level of spiking, then this higher level will be quoted as the level of detection. In certain instances, when the analyte being spiked is also present in the sample at concentrations greater than the level of detection, corrections will be applied in order to estimate the recovery of this compound. Instrument malfunction as a source of False Negatives is minimized with the use of internal standards. Internal standards insure no False Negatives are reported due to instrumental malfunction and surrogate standards insure no False Negatives are reported due to extraction malfunction.

(continued)

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.

Chapter 26, Statistics in Quantitative Analysis in Chemical Analysis by Herbert A. Laitinen, pulished by McGraw-Hill Book Company, Inc.

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 interfere 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 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 extrsction end 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 inadvertently 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 documentation 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:MM

MONS 024966

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 fluctuations.

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:

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

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.

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