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ABSTRACT: Twelve 1-liter water samples were received from the Kearny Plant for low-level PCB analyses. PCB formulations were detected above 0.1 ug/L in seven of the twelve samples. This report gives descriptions of the methods and discussion of these results.

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APPROVAL DATE: July 8, 1988

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RESEARCH REPORT

DATE: May 27, 1988

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Title: Analysis of PCBs in Kearny Plant Water Samples

ABSTRACT

Twelve 1-liter water samples were received from the Kearny Plant for low-level PCB analyses. PCB formulations were detected above 0.1 ug/L in seven of the twelve samples. This report gives descriptions of the methods and discussion of these results.

INTRODUCTION

Twelve water samples were received from the Kearny Plant for low-level PCB analyses. Four individual 1-liter samples labelled #110 were used for two duplicate analyses and two native spiking analysis studies. The remaining samples were labelled #101 - #109 and #111 and #112. Sample volumes were 1-liter.

In order to obtain highly reliable data for PCB formulation concentrations between 0.1 and 100 ug/L, the 1-liter samples were extracted with methylene chloride and the extract volume was reduced to 1.0 mL. The final extracts were analyzed using a capillary GC/MS analysis method. This method produced detailed information on the amounts of total CL_1-biphenyl through total CL_{10}-biphenyl congeners. (For this report, CL_1-biphenyl is abbreviated "CL1-BP", CL_2-biphenyl is abbreviated "CL2-BP", etc.) Estimates of the probable PCB formulations were obtained by comparing the distributions of the total chlorinated biphenyl congener concentrations with the distribution of concentrations obtained from the analysis of standards of Aroclor formulations. (Note: Aroclor is a registered trademark of the Monsanto Company.)

EXPERIMENTAL

One-liter water samples were spiked with 1 ug each of four surrogate compounds (^{13}C-labelled PCB congeners). Liquid/liquid extraction using a total of 180 mLs methylene chloride was used to isolate PCBs into the organic extract. This extract was reduced to 1.0 mL using Kudarna-Danish concentrators. This extraction method and extraction flow sheet are summarized as WSEX05 and WSFS05 in Appendix B. Appendix B also shows an example extraction sheet and summary extraction sheets for this sample set.

Once the 1-liter samples are extracted, 200 uL of the 1000 uL extract or 200 uL of all calibration solutions were combined with 200 uL anthracene-d_{10} (10 ug/mL) in autosampler vials. The extracts and standards to which the anthracene-d_{10} internal standard had been added were then analyzed on a capillary GC/MS analytical system using Standard Analysis Method MSAN25. Brief descriptions of this method and the flow sheet (MSFS25) are also shown in Appendix B. These methods also describe calculations which are used to obtain individual PCB congener concentrations, total PCB congener concentrations for CL_1-biphenyl through CL_{10}-biphenyl congeners, and the total PCB concentrations.

RESULTS AND DISCUSSION

Representative chromatograms and tables resulting from Lotus/1-2-3 calculations are located in Appendix C. The Lotus calculation results are shown in five sets of tables shown in Appendix C. Tables 1. and 2. summarize: 1) total CL_1-BP through total CL_{10}-BP congener extract concentrations (in units of ug/mL), 2) total PCB extract concentrations (in units of ug/mL), and 3) total PCBs detected in each sample (in units of ug/L). Since there is a one thousand-fold decrease in volume from the water sample volume to the extract volume, there is exactly a one thousand-fold decrease in concentration from the measured extract concentration to the water sample concentration. Tables 1. and 2. in Appendix C also show recoveries of the four ^{13}C-labelled surrogates which were added to each water sample before extraction began, and pertinent information relating to file names used for extract analysis, dilution factors, and internal standard areas measured.

Tables 1.a and 2.a summarize Average Relative Response Factors which were determined from the triplicate analysis of a standard solution containing 24 monochlorobiphenyl through decachlorobiphenyl congeners. Tables 1.b and 2.b summarize the ratios of characteristic/confirming ion responses for standards and extracts. Tables 1.c and 2.c summarize the average surrogate recoveries, standard deviations, minimum recovery, and maximum recovery measured for the set of data shown in Tables 1. and 2., respectively.

Table 3. summarizes recoveries of 24 native PCB congeners which were added to sample #110 for quality control purposes and Tables 4., 4.a, 4.b., 5., 5.a, and 5.b summarize total PCB congener concentrations measured for Aroclor formulation standard solutions obtained from the Supelco Company.

All of these data which are shown in Appendix C are further condensed and shown in Table 1 and Figures 1 - 10 of the present report. Figures 1 - 3 summarize the total PCB concentrations and total PCB congener concentrations for CL_1-BP through CL_{10}-BP congeners measured for samples #101 through #112. (Note that 0.1 ug/L was detected for the CL_3-biphenyl congener in #103 which is at our method detection limit. This extract was analyzed immediately after the extract for sample #102 which contained almost 100 ug/L total PCBs. We have assumed that this trace quantity of CL_3-biphenyl congeners is due to carry-over from the analysis of #102.)

In order to determine the nature of the PCBs detected in sample numbers 101, 102, 104, 105, 106, 107, and 109, the total PCB congener distributions were compared to distributions measured from the analysis of various PCB formulation standards. These standards were obtained from the Supelco Company as 100 ug/mL Aroclor formulations in methanol. Figure 4 shows that the distribution of PCB congeners for Aroclor 1248 is unaffected from 0.1 to 10 ug/mL. (This corresponds to a concentration range of 0.1 to 10 ug/L in a 1-liter water sample.) Therefore comparisons of distributions of total PCB congeners in water extracts with distributions from Aroclor formulation analyses should provide valuable information on the formulation identity.

Figure 5 shows that the distribution for sample numbers 105, 106, 107, and 109 appear to be nearly identical to that shown in Figure 4 for Aroclor 1248. Furthermore, although #102 and #104 are very similar to Aroclor 1248, this PCB congener distribution probably arises from a lower formulation such as Aroclor 1242. Figures 6 and 7 show the PCB congener distributions for eight formulations and Figure 8 shows that the distributions for #102 and #104 are very similar to the Aroclor 1242 distribution.

The extract of sample #101 was analyzed in duplicate. The distributions obtained from the duplicate analysis of this extract is shown in Figure 9. Along with these two distributions are shown distributions for Aroclors 1254, 1260 and 1262. It appears that sample #101 can best be described as a mixture of these three formulations.

Absolute concentrations of total PCBs were also determined from the analysis of all formulations shown in Figures 4, 6 and 7. The results of analysis of Aroclor formulation standards from Supelco Company are shown in Tables 4., 4.a, 4.b, 5., 5.a, and 5.b in Appendix C. Portions of these data are also summarized in Table 1 of this report. Figure 10 summarizes the results of these analyses. For all standard Aroclor formulations analyzed, the total PCB concentration determined was within 47% of the concentration reported by Supelco. If one excludes the 0.1 ug/mL Aroclor 1248 standard which was at our instrument level of detection, only Aroclors 1262 and 1268 show deviations as large as 47%. All other standard solutions were within 15% of the correct value.

CONCLUSIONS

Sample numbers 103, 108, 110, 111, and 112 did not contain any detectable PCB congeners above the estimated level of detection of 0.1 ug/L per PCB congener. Comparison of PCB congener distributions indicate that the remaining positive samples consisted of PCB formulations similar to Aroclors 1242, 1248, and a mixture of 1254, 1260, and 1262.

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 is to determine the validity of the reported results so that the analysis requestor 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. Tables 1. and 2. in Appendix C report the results of a duplicate analysis for #101 in which compounds were present above the level of detection. (Note that this is not a true duplicate analysis since duplicate 1-liter water samples were not provided for this sample.) These tables also report the percent recovery of surrogate compounds which were added to each sample before analysis. These data show that the analytical precision is better than 30% for this set of samples. Analyses of all Aroclor formulation standards ranging in concentration from 0.1 to 10 ug/mL resulted in total PCB concentrations within 47% of the reported Aroclor concentrations. If one excludes the 0.1 ug/mL Aroclor 1248 standard which was at our instrument level of detection, only Aroclors 1262 and 1268 show deviations as large as 47%. All other standard solutions were within 15% of the correct value. NOTE!!! It is the responsibility of the analysis requestor to review 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. Tables 1. and 2. in Appendix C show the recoveries measured when all samples were spiked with four ^{13}C-labelled surrogate compounds (4-CL-BP-$^{13}C_6$, 3,3',4,4',CL_4-BP-$^{13}C_{12}$, 2,2',3,3',5,5',6,6',CL_8-BP-$^{13}C_{12}$, and CL_{10}-$^{13}C_{12}$). Table 3. in Appendix C shows the recoveries measured when 24 PCB congeners were spiked into sample 110 at 0.1 and 1.0 ug/L per component.

For the 40 surrogate recoveries reported in Table 1. in Appendix C:

Recovery Mean	= 98 %
Recovery Standard Deviation	= 27 %
Recovery Range	= 157 %
Minimum Recovery	= 50 %
Maximum Recovery	= 207 %

The frequency distribution for these recoveries are also shown in Table 1.c. This distribution shows that 1 of the 40 recoveries is above 180% and 1 is below 50%. Thirty-eight of the 40 values fall between 50 and 150% recovery.

For the 40 surrogate recoveries reported in Table 2. in Appendix C:

Recovery Mean	= 100 %
Recovery Standard Deviation	= 31 %
Recovery Range	= 170 %
Minimum Recovery	= 58 %
Maximum Recovery	= 228 %

The frequency distribution for these recoveries are also shown in Table 2.c. This distribution shows that 1 of 40 recoveries is above 180% and that 37 of 40 values fall between 50 and 150%.

For the 56 native and surrogate recoveries reported in Table 3. of Appendix C for native spiking concentrations of 0.1 and 1.0 ug/L and surrogate spiking concentrations of 1.0 ug/L:

Recovery Mean	= 86.38 %
Recovery Median	= 86.50 %
Recovery Standard Deviation	= 14.11 %
Recovery Range	= 67.00 %
Minimum Recovery	= 55.00 %
Maximum Recovery	= 122.00 %

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

Bias. The Recovery Means for surrogate recoveries in Tables 1. and 2. of Appendix C and shown above, are within 2% of 100% and therefore there are no significant biases introduced into these analyses. The Recovery Means for native and surrogate recoveries in Table 3. of Appendix C and shown above, are within 14% of 100% which further substantiates that 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 requestor to apply appropriate bias corrections if he/she feels that such corrections are warranted.

False Positives and Negatives. The analyses of Organic Free Water shown in Table 1. of Appendix C shows that the analytical system introduces no false positive values. The results of analysis of sample #110 which was spiked with 0.1 ug/L shown in Table 3. demonstrate that no false negative values have been reported. The reported levels of detection in this study are approximately equal to the concentration of the lowest native spiking study reported in Table 3. of Appendix C (0.1 ug/L).

REFERENCES

A more complete description of this method may be obtained from a Research Report published in Monsanto's Reports Library, MSL-7410, ESC-UAG-87-174, issued December 18, 1987 entitled *"Summary of Improvements in Incidental PCB Analysis Approaches"*, by D. E. McKenzie, L. R. Minor and B. M. Hughes.

LIST OF APPENDICES

Appendix A. Summary of project details for this report. (This section contains data processing information on the names of files, names of samples, names of methods, etc.)

Appendix B. Summary of Extraction and Analysis Methods used for this project.

Appendix C. Summary of GC/MS Raw Data and Detailed Lotus/1-2-3 Outputs.

Appendix D. Important analytical request and chain-of-custody information recorded at sample login.

Appendix E. Descriptions of important terminology used in Quality Assurance/Quality Control, Analyte Extraction, and Instrumental Analysis.

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

ANALYST APPROVAL: B. Mason Hughes

Table 1. Summary of total PCB congeners and total PCBs detected in water samples from the Kearny Plant and Aroclor standards.

Project Number 760.24-0222 — May 25, 1988

Sample Number	Total PCB Congener Concentrations (ug/L) CL1-BP	CL2-BP	CL3-BP	CL4-BP	CL5-BP	CL6-BP	CL7-BP	CL8-BP	CL9-BP	CL10-BP	Total PCBs	Probable Formulation
101	0	0	0	0	4.5	3.8	5.6	2.3	0.1	0	16.3	mixture
101-dup	0	0	0	0	0.2	5.5	11.1	2.8	0.3	0	25.9	mixture
102	0.3	10.7	30.0	33.2	19.4	3.0	1.2	0.3	0	0	99.5	A1242
103	0	0	(0.1)	0	0	0	0	0	0	0	0	
104	0.1	2.4	10.1	8.6	1.9	0.1	0	0	0	0	24.2	A1242
105	0	0	0.2	0.3	0.1	0	0	0	0	0	0.6	A1248
106	0	0	0.1	0.2	0.1	0	0	0	0	0	0.4	A1248
107	0	0	1	1.0	0.9	0.1	0	0	0	0	3.0	A1248
108	0	0	0	0	0	0	0	0	0	0	0	
109	0	0	1.7	3.5	1.7	0.1	0	0	0	0	7	A1248
110	0	0	0	0	0	0	0	0	0	0	0	
110-dup	0	0	0	0	0	0	0	0	0	0	0	
111	0	0	0	0	0	0	0	0	0	0	0	
112	0	0	0	0	0	0	0	0	0	0	0	
Rock Drum	0	0	0	0	0	0	0	0	0	0	0	
Spiked Sample 0110												
110-LS	0.3	0.4	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1	1.9	
110-HS	2.6	3.7	2.6	2.5	2.4	2.7	1.6	0.9	0.9	0.9	20.8	

Aroclor Standards											Total	Correct	Total/Correct (%)
A1260(.1)	0	0	0.02	0.06	0	0	0	0	0	0	0.06	0.1	60
A1260(1)	0	0.06	0.21	0.51	0.16	0	0	0	0	0	0.92	1	92
A1260(5)	0.01	0.17	1.11	2.36	1.07	0.06	0	0	0	0	4.76	5	95.2
A1260(10)	0.03	0.32	2.26	4.85	2.26	0.16	0	0	0	0	9.88	10	98.8
A1016(1)	0.01	0.2	0.5	0.24	0	0	0	0	0	0	0.95	1	95
A1221(5)	3.64	1.44	0.19	0.08	0	0	0	0	0	0	5.35	5	107
A1232(2)	0.59	1.13	0.35	0.06	0	0	0	0	0	0	2.11	2	105.5
A1242(1)	0.01	0.19	0.39	0.29	0.04	0	0	0	0	0	0.92	1	92
A1254(2)	0	0	0.02	0.29	1.26	0.53	0.06	0	0	0	2.16	2	108
A1260(2)	0	0	0	0	0.4	0.95	0.79	0.17	0	0	2.29	2	114.5
A1262(1)	0	0	0	0	0	0.25	0.35	0	0	0	0.6	1	60
A1268(1)	0	0	0	0	0	0	0.09	0.02	0.48	0.08	1.47	1	147

Figure 1. Summary of Total PCBs Measured in Kearny Water Samples

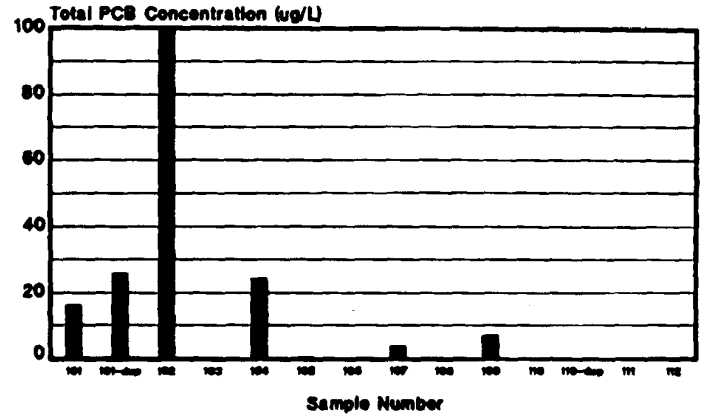


Figure 2. Summary of PCB Congeners Measured in Kearny Water Samples

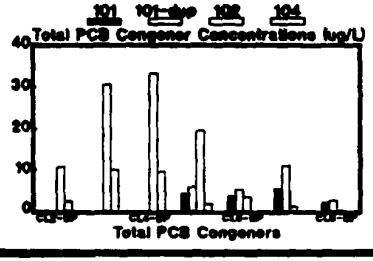
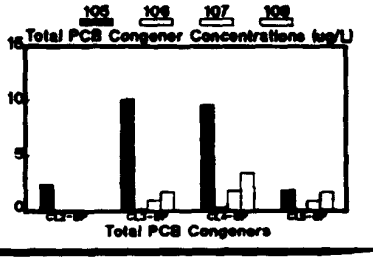
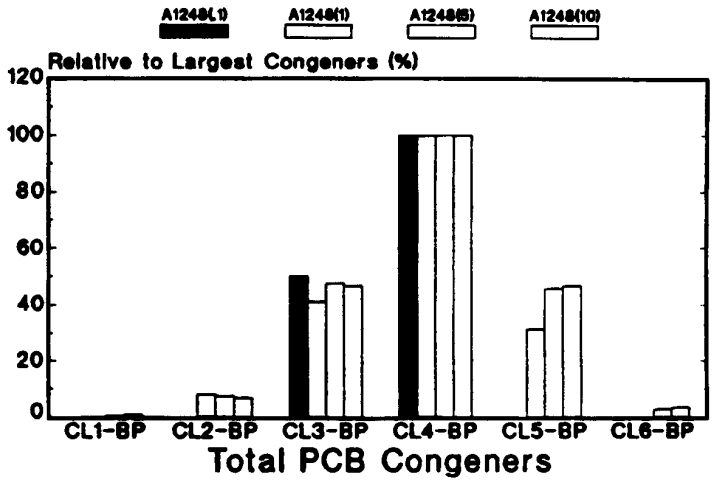


Figure 3. Summary of PCB Congeners Measured in Kearny Water Samples



MONS 024979

Figure 4. Summary of PCB Congener Distribution for Aroclor 1248 Standards



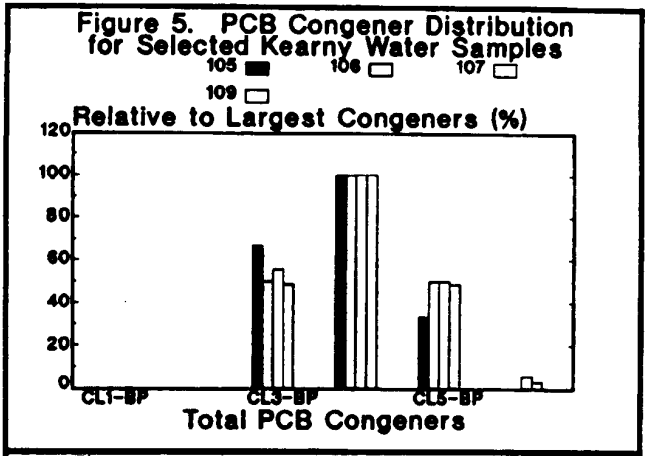


Figure 5. PCB Congener Distribution for Selected Kearny Water Samples

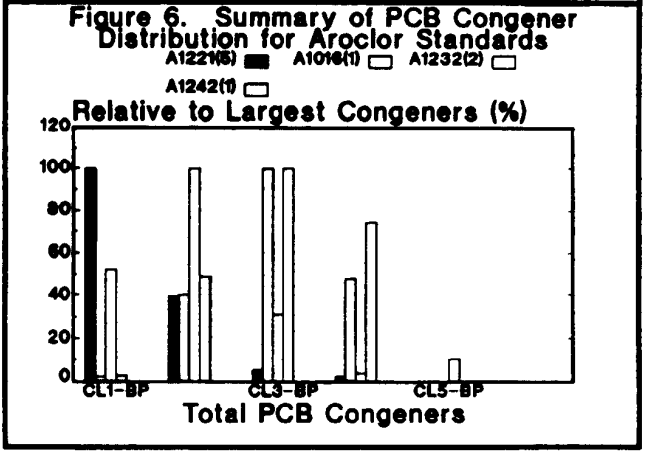


Figure 6. Summary of PCB Congener Distribution for Aroclor Standards

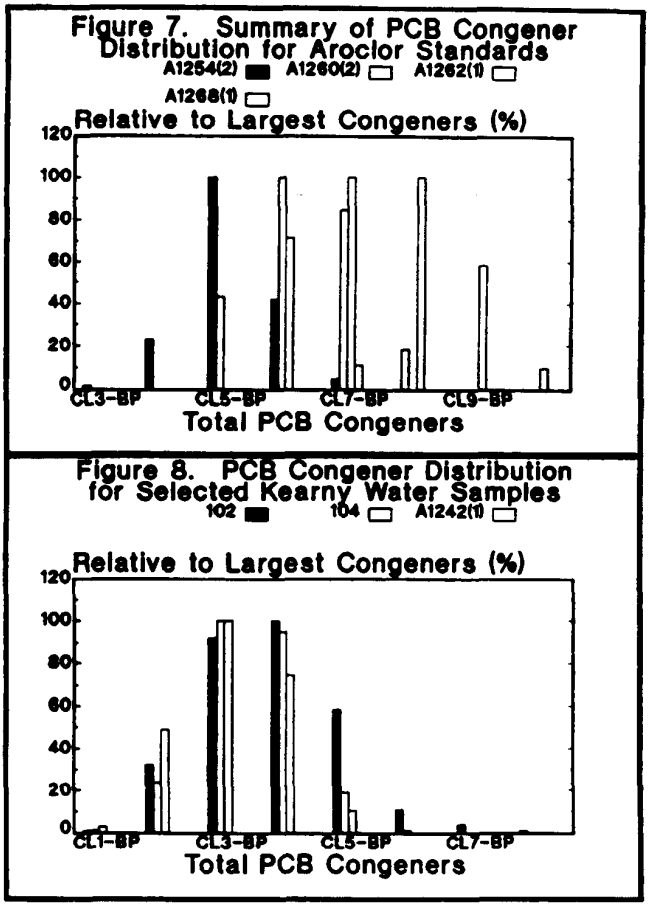


Figure 7. Summary of PCB Congener Distribution for Aroclor Standards
A1254(2)
A1260(2)
A1262(1)
A1268(1)
Relative to Largest Congeners (%)
120
100
80
60
40
20
0
CL3-BP
CL5-BP
CL7-BP
CL9-BP
Total PCB Congeners
Figure 8. PCB Congener Distribution for Selected Kearny Water Samples
102
104
A1242(1)
Relative to Largest Congeners (%)
120
100
80
60
40
20
0
CL1-BP
CL3-BP
CL5-BP
CL7-BP
Total PCB Congeners

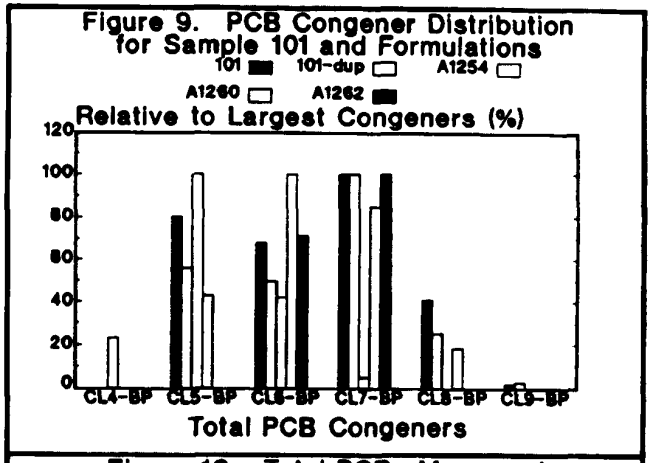


Figure 9. PCB Congener Distribution for Sample 101 and Formulations
101
101-dup
A1254
A1260
A1262
Relative to Largest Congeners (%)
120
100
80
60
40
20
0
CL4-BP
CL5-BP
CL6-BP
CL7-BP
CL8-BP
CL9-BP
Total PCB Congeners

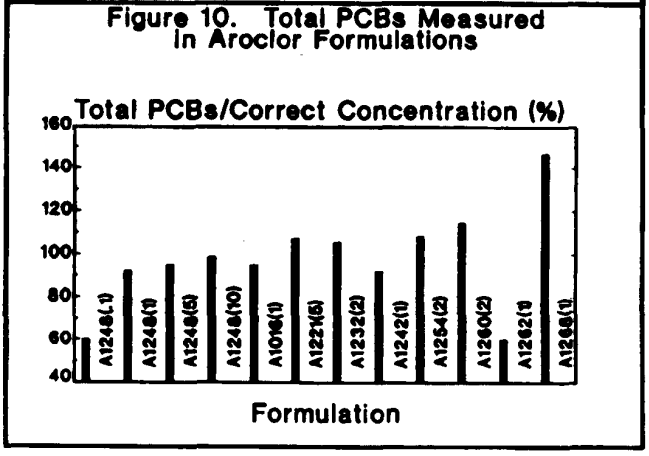


Figure 10. Total PCBs Measured In Aroclor Formulations
Total PCBs/Correct Concentration (%)
160
140
120
100
80
60
40
A1248(.1)
A1248(1)
A1248(5)
A1248(10)
A1016(1)
A1221(5)
A1232(2)
A1242(1)
A1254(2)
A1260(2)
A1262(1)
A1268(1)
Formulation

Appendix A. Summary of project details for this report. (This section contains data processing information on the names of files, samples, extraction and analysis methods, etc.)

Standard Sampling Method(s): Samples supplied by outside consultant.

Standard Extraction Method(s): WSEX05 (See WSFS05 for method flow sheet.)

Standard Instrumental Analysis Method(s): MSAN25 (See MSFS25 for method flow sheet.)

GC/MS Data Acquisition Method Name: PCSC25

Sample Identification(s): #101 - #112. (See further information on extract numbers, notebook page numbers, etc. in Appendix D.

Final Report File(s): \msword\projects\proj8222\ra8222.doc, and ...\aa8222.doc

Table File(s): \123\projects\proj8222\ta8222

Appendix B. Summary of Extraction and Analysis Methods used for this project.

This section contains brief method descriptions and flow sheets for Standard Extraction Method WSEX05, copies of extractions sheets generated during sample extraction, Standard Analysis Method MSAN25, and a copy of the GC/MS instrumental method used to acquire selected ion monitoring data for PCBs.

ESC Standard Extraction Method (SEM)
TITLE: Wide scan extraction method for water.

ESC Standard Extraction Method No: WSEX05

MATRIX:	TYPICAL SAMPLE SIZE (Liters)	TYPICAL EXTRACT VOLUME (ml)
water	0.01 - 4	1 - 40

TYPICAL ANALYTES: Aroclors, PCBs, priority pollutants

ESC Standard Analysis Method Nos: ECANxx and MSANxx

INTERFERENCES: Various; See individual interferences for individual analytes.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
phenol-d5	20-80
CL-phenol-d4	20-80
CL2-benzene-d4	50-120
naphthalene-d8	50-120
pyrene-d10	50-120
chrysene-d12	50-120
pentachlorobenzene	50-120
2,4,6-trichloro-biphenyl	50-120

APPARATUS AND MATERIALS:
Burdick and Jackson methylene chloride
Separatory funnels
Sodium sulfate for extract drying
Sodium hydroxide and sulfuric acid for pH adjustments
Nitrogen for extract volume reduction
Water bath and K-D apparatus for extract volume reduction

LIMITATIONS: Sufficient sample amount to provide GC-MS LOD.

SAFETY PRECAUTIONS: See Safe Laboratory Practices.

DATES OF METHOD CREATION AND UPDATES: 5/16/84, 10/5/84, 12/7/84

FLOW SHEET WSFS05
FOR ESC EXTRACTION METHOD WSEX05

SAMPLE ID: Water samples #101-112 MRC EXTRACT # PCB 2295-2310

LOG NO: U-88-04-25-02

EXTRACTION DATE and TIME: APRIL 29, 1988 / 10 am

RECORD NATIVE pH ---7--- WAS pH ADJSTED YES (NO) IF YES TO WHAT pH----------

PLACE SAMPLE INTO AN APPROPRIATELY SIZED SEPERATORY FUNNEL (1/2 HEAD SPACE SUGGESTED FOR ADEQUATE MIXING) ⋈ SAMPLE VOLUME ----1L----

SURROGATE SPIKE
SPK = 100 µL 1,3 PCB-2 ng/mL
AS = 100 µL 1NPB10 ng/mL
HS = 100 µL 4NPB10 (Stock) ng/mL

IF pH ADJUSTED ( ) ADJUST WITH, 50% NaOH OR H_2SO_4

ADD METHYLENE CHLORIDE IN THE APPROPRIATE AMOUNT (1 L SAMPLE 30 mL SOLVENT, 100 mL SAMPLE 15 mL SOLVENT) ⋈ RECORD AMOUNT METHYLENE CHLORIDE ADDED FOR EACH EXTRACTION
1) 60mL
2) 60mL
3) 60mL

SHAKE SAMPLE ONE TIME AND VENT THROUGH STOPCOCK REPEAT DOUBLING THE NUMBER OF SHAKES BETWEEN VENTING SAMPLES UNTIL THE NUMBER OF SHAKES EXCEED 80 ⋈

OBSERVATIONS

ALLOW PHASES TO SEPARATE ⋈

REPEAT EXTRACTION PROCEDURE TWO MORE TIMES ⋈ [ ]

IF ACID OR BASE WASHED RECORD TYPE AND AMOUNT. [ ]

ACID/BASE WASH COMENTS

DRY EXTRACT WITH 4 INCH SODIUM SULFATE COLUMN (COLUMN PRERINSED WITH 30 mL METHYLENE CHLORIDE) ⋈

[ ] EXTRACT DRYING NOT REQUIRED

RINSE SODIUM SULFATE COLUMN WITH 2 X 30 mL ⋈

REDUCE EXTRACT VOLUME TO 1 mL BY K-D TECHNIQUE AND OR NITROGEN SOLVENT VOLUME REDUCTION ⋈

⋈ TRANSFER EXTRACT INTO VIAL FOR ANALYSIS

EXTRACT NO: PCB 2295-2314 DATE/TIME COMPLETED: 4/30/88 /

ANALYST: [signature] ESC JOB NO: 760-3[illegible]

MONS 024987

PROJECT NAME/NUMBER: Koerny 18222 DATE: 4/28/88

EXT. LOGBOOK PAGE NUMBER: 15646 EXTRACTION METHOD: WSEX05

LOG NUMBER FOR GROUP: U-88-04-25-02 ANALYST: Donner/McKenzie

EXTRACT NUMBER	SAMPLE IDENTIFICATION	WEIGHT/ VOLUME	COMMENTS:
PCB2295	BLANK	1L	PH = 7 COLOR
PCB2296	110		PH = 7 ORG
PCB2297	110 (DUP)		PH = 7 ORG
PCB2298	110 + LS		PH = 7 ORG
PCB2299	110 + HS		PH = 7 ORG
PCB2300	109		PH = 7 ORG
PCB2301	111		PH = 7 YEL
			pH is not adjusted
			1 x 60 mL chromatography
			ORG = ORANGE
			YEL = YELLOW

Calibration notes: class S weight - Balance Reading

SOLVENT USED: CH_2Cl_2 NUMBER OF TIMES EXTRACTED X AMOUNT: 3 x 60

SPIKING INFORMATION

(ALL SAMPLES RECEIVE SPK AND ONLY THOSE INDICATED RECEIVE LS OR HS IN ADDITION TO SPK)
(FILE SLSAVE:mh)

FINAL VOLUME EXTRACT: 1mL

SPK- C_{13} PCB-2 100µl

LS- INPC10 1:10 1ug/mL 100µl

HS- INPC10 STOCK 100µl

MONS 024988

PROJECT NAME/NUMBER: Kearney / 8222 DATE: 4/29/88

EXT. LOGBOOK PAGE NUMBER: 15864 EXTRACTION METHOD: WSEK05

LOG NUMBER FOR GROUP: U-88-04-25-02 ANALYST: Muncer / McKenzie

EXTRACT NUMBER	SAMPLE IDENTIFICATION	WEIGHT/ VOLUME	COMMENTS:
			Color
PCB2302	101	1L	PH = 7 gray muddy — lost ≈200ml on first ext.
PCB2303	102	↓	PH = 7 contains muc-gray
PCB2304	103		PH = 7 yellow orange
PCB2305	104		PH = 7 yellow
PCB2306	105		PH = 7 yellow-orange
PCB2307	106		PH = 7 yellow orange
PCB2308	107	↓	PH = 7 yellow orange
	PCB 2302 & 2303 white large emulsions 1st extract glass wool plug was attached to the drying column (prior) emulsion was run directly through them	400ml each	pH wasn't adjusted. 1X 60ml column rinse

Calibration notes: class S weight - Balance Reading

SOLVENT USED: CH_2Cl_2 NUMBER OF TIMES EXTRACTED X AMOUNT: 3 X 60

SPIKING INFORMATION FINAL VOLUME EXTRACT: 1ml

SPK- 100ul O;SPCB-2

(ALL SAMPLES RECEIVE SPK AND ONLY THOSE INDICATED RECEIVE LS OR MS IN ADDITION TO SPK)
(FILE BLBANU:mh)

LS- 100ul INPC10 1ug/ml

MS- 100ul INPC10 STOCK

MONS 024989

PROJECT NAME/NUMBER: Kearny / 8822 DATE: 4/29/88

EXT. LOGBOOK PAGE NUMBER: 15/616 EXTRACTION METHOD: WSEL05

LOG NUMBER FOR GROUP: 4-88-04-25-02 ANALYST: Thomas McTemple

EXTRACT NUMBER	SAMPLE IDENTIFICATION	WEIGHT/ VOLUME	COMMENTS:
PCB2309	108	1L	pH = 7 Cloudy yellow
PCB2310	112	1L	pH = 7 clear

Calibration notes: class S weight - Balance Reading

SOLVENT USED: CH_2Cl_2 NUMBER OF TIMES EXTRACTED X AMOUNT: 3 x 60

SPIKING INFORMATION

(ALL SAMPLES RECEIVE SPK AND ONLY THOSE INDICATED RECEIVE LS OR BS IN ADDITION TO SPK)
(FILE BLEAVU:mh)

FINAL VOLUME EXTRACT: 1ml

SPK- 100µl C13 PCB-2

LS- 100µl WP010 1ug/ml

BS- 100µl WP010 STOCK

MONS 024990

ESC Standard Analysis Method (SAM) **ESC Standard Analysis Method No:**
TITLE: Polychlorinated Compounds in Solvents **MSAM25**

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5985/5987 Capillary GC/MS-Selected Ion Monitoring with RTE-VI operating system.

Typical Run Time:	15 - 40 min.
Typical Multiplier Voltage:	1800 - 2800 eV
Typical Dwell Times:	100 - 500 msec/mass
Maximum # of Masses:	20 masses x 5 ranges = 100 masses
Electron Energy:	70 eV
Mass Spectrometer Tuning File:	MT8602
Mass Spectrometer Data Acquisition Method:	PCSC25

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Organic Compounds eluting between toluene and n-C34-ane	0.01 - 0.1	30	30

INTERFERENCES AND LIMITATIONS: Analyte characteristic mass must be chromatographically resolved from other eluting compounds which produce the same mass.

CHROMATOGRAPHIC COLUMN: 30-meter fused silica J&W DB-5 (0.25 uM) with wide bore (0.32 mm)

COLUMN TEMPERATURE PROGRAM: 50(4)/8/300 **INJECTION TYPE:** Splitless

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CALIBRATION AND STANDARDIZATION:

Quantitative: Use internal standard quantitation technique and calibrate instrument response factors from standard solutions of analytes of interest.

Qualitative: When multiple characteristic masses are monitored for a given analyte, the ratio of characteristic mass responses must be within 20% of those for the authentic standard. For PCB analyses, the characteristic masses which are monitored are the molecular ion containing all 35-CL isotopes and the molecular ion containing one 37-CL isotope. The following table summarizes the masses and approximate retention time regions monitored for the analysis of internal standard, surrogate standards, and chlorobiphenyl (CL-BP) through decachlorobiphenyl (CL10-BP) PCB congeners.

(continued)

DATA ACQUISITION INFORMATION

Analyte	Masses	Retention Times (minutes)
CL-BP isomers, anthracene-d10	188,190	15 - 22
4-CL-BP-6Cl3 surrogate	194,196	15 - 22
CL2-BP isomers	222,224	15 - 22
CL3-BP isomers	255.9,257.9	15 - 30
CL4-BP isomers	289.8,291.8	22 - 30
3,4,3',4'-CL4-BP-12Cl3 surrogate	301.8,303.8	22 - 30
CL5-BP isomers	323.9,325.9	22 - 30
CL6-BP isomers	359.8,361.8	22 - 35
CL7-BP isomers	393.8,395.8	22 - 35
CL8-BP isomers	427.7,429.7	30 - 35
2,2',3,3',5,5',6,6-CL8-BP-12Cl3 surr.	439.7,441.7	30 - 35
CL9-BP isomers	461.6,463.6	30 - 35
CL10-BP	497.6,499.6	30 - 35
CL10-BP-12Cl3 surrogate	509.6,511.6	30 - 35

CALCULATIONS: Analyte concentration = (Analyte area)/[(Analyte RRF)(IS area)]

where: Analyte area is the characteristic mass area of the analyte.
RRF is the relative response factor for the analyte.
IS is the internal standard and is usually anthracene-d10.

RRF = (analyte area in standard)/[(analyte conc.)(IS area)]

PCB concentrations are calculated in one of two ways:

1) If an Aroclor formulation can be identified from the patterns of PCB isomers, selected PCB isomers or classes of isomers (i. e. all CL4-biphenyl isomers) are used to quantify the Aroclor concentration. Relative Response Factors are calculated using the concentration of the total Aroclor formulation in the above RRF equation.

2) If no unique Aroclor formulation is present, then the total PCB concentration is calculated by using standard solutions containing known amounts of chlorobiphenyl through decachlorobiphenyl congeners to calculate Relative Response Factors for each chlorine class in the above RRF equation. The RRFs for the corresponding PCB isomer classes are used to calculate total PCB isomer concentrations of each class in unknown samples.
(For more information regarding standards, see Supplement to MSAN25 on the following page.)

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

DATE OF METHOD CREATION: 12/9/83 Time Stamp - <880408.1524>

(continued)

Supplemental Information to Standard Analysis Method MSAN25

The following describes details of internal standards, surrogate standards, and individual PCB congeners used for instrument calibration:

Internal Standard

Anthracene-d10

Surrogate Spiking Compounds C-13 labeled

4-chlorobiphenyl 13C6
3,3',4,4'-tetrachlorobiphenyl 13C12
2,2',3,3',5,5'6,6',-octachlorobiphenyl
decachlorobiphenyl 13C12

Native Spiking Compounds (used for native spikes and instrumental calibration)

2-monochlorobiphenyl
3-monochlorobiphenyl
4-monochlorobiphenyl
2,2'-dichlorobiphenyl
2,3-dichlorobiphenyl
2,4'-dichlorobiphenyl
4,4'-dichlorobiphenyl
2,4,6-trichlorobiphenyl
2,3,4-trichlorobiphenyl
2,4,4'-trichlorobiphenyl
2,3',4,6,-tetrachlorobiphenyl
2,3,5,6,-tetrachlorobiphenyl
2,2',4,4',-tetrachlorobiphenyl
2,2',3,4,5-pentachlorobiphenyl
2,2',3,4,5'-pentachlorobiphenyl
2,2',4,5,5'-pentachlorobiphenyl
2,2',3,3',4,4'-hexachlorobiphenyl
2,2',3,4,4',5'-hexachlorobiphenyl
2,2',3,3',6,6'-hexachlorobiphenyl
2,2'3,3',4,4',6-heptachlorobiphenyl
2,2'3,4,4',5',6-heptachlorobiphenyl
2,2',3,3',4,4',5,5'-octachlorobipyhenyl
2,2',3,3',4,5,5,',6,6'-nonachlorobiphenyl
decachlorobiphenyl

(continued)

PCB Windowing Compounds (used to document proper data acquisition windows)

3-CL1- and 4-CL1-biphenyl
2,6-CL2 and 4,4'CL2-biphenyl
2,4,6-CL3- and 3,4,4'-CL3-biphenyl
2,2',6,6'-CL4- and 3,3',4,4'-CL4-biphenyl
2,2',4,6,6'-CL5- and 3,3',4,4',5-CL5-biphenyl
2,2',4,4',6,6'-CL6- and 3,3',4,4',5,5'-CL6-biphenyl
2,2',3,4',5,6,6'-CL7- and 2,3,3',4,4',5,5'-CL7-biphenyl
2,2',3,3',5,5',6,6'-CL8- and 2,3,3',4,4',5,5',6-CL8-biphenyl
2,2',3,3',4,5,5',6,6'-CL9- and 2,2',3,3',4,4',5,5',6-CL9-biphenyl
CL10-biphenyl

Characteristic and Confirming Masses Monitored

	Characteristic (AMU)	Confirming (AMU)
anthracene-d10	188	190
4-chlorobiphenyl-6C13	194	196
3,3'4,4'-tetrachlorobiphenyl-12C13	301.8	303.8
2,2'3,3',5,5',6,6'-octachlorobiphenyl-12C13	439.7	441.7
decachlorobiphenyl-12C13	509.6	511.6
monochlorobiphenyls	188	190
dichlorobiphenyls	222	224
trichlorobiphenyls	255.9	257.9
tetrachlorobiphenyls	289.8	291.8
pentachlorobiphenyls	323.9	325.9
hexachlorobiphenyls	359.8	361.8
heptachlorobiphenyls	393.8	395.8
octachlorobiphenyls	427.7	429.7
nonachlorobiphenyls	461.6	463.6
decachlorobiphenyl	497.6	499.6

FLOW SHEET MSFS25

FOR ESC ANALYSIS METHOD MSAN25

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

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

MS LOGBOOK PAGE NUMBER: .....

EXTRACT COLOR AND DESCRIPTION .....

CALIBRATE MASS SPECTROMETER WITH PFTBA AND DEMONSTRATE CAPILLARY CHROMATOGRAPHIC RESOLUTION WITH SYSTEM PERFORMANCE STANDARD [ ]

PLACE IN A 1.6 mL AUTOSAMPLER VIAL THE SAMPLE EXTRACT OR STANDARD SOLUTION CONTAINING PCB CONGENERS WITH 150 uL INTERNAL STANDARD SOLUTION. [ ]

THE INTERNAL STANDARD SOLUTION USUALLY CONTAINS 10 UG/mL ANTHRACENE-D10 IN CH2CL2 OR BENZENE.

PLACE VIAL IN AUTOSAMPLER TRAY OF HP-5985/87. [ ]

CALCULATE CHARACTERISTIC ION AREAS OF ANALYTES OF INTEREST AND ANTHRACENE-D10 INTERNAL STANDARD.

RECORD MASS SPECTROMETRIC AND GAS CHROMATOGRAPHIC OPERATING PARAMETERS IN INSTRUMENT LOGBOOK. [ ] . . . . . [ ]

[ ] CALCULATE CONCENTRATION OF ANALYTES OF INTEREST USING THE FOLLOWING EQUATIONS:

$$\text{PCB Chlorine Class Concentration} = \frac{\text{(Chlorine Class Ion Area)}}{\text{(Chlorine Class RRF) (Internal Standard Area)}}$$

$$\text{Chlorine Class RRF} = \frac{\text{(Characteristic Ion Area for Chlorine Class)}}{\text{(Chlorine Class Concentration)(Internal Standard Area)}}$$

Total PCB Concentration = CL1-BP Concentration + CL2-BP Concentration + CL3-BP Concentration + ... + CL10-BP Concentration

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

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

MONS 024995

M E T H O D   F I L E   L I S T

Method file: PCSC25   GC type: 5840   Run Type: SIM, GC, EI
Column: Cap   Splitless: Yes

Temperature:	Source	Isothermal	Post Run Time
	200.0	N	0.0

Run time: 40.00
Scan Start time: 15.00

	ON	OFF	ON	OFF
Relay #1:	327.0	327.0	327.0	327.0
Relay #2:	327.0	327.0	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

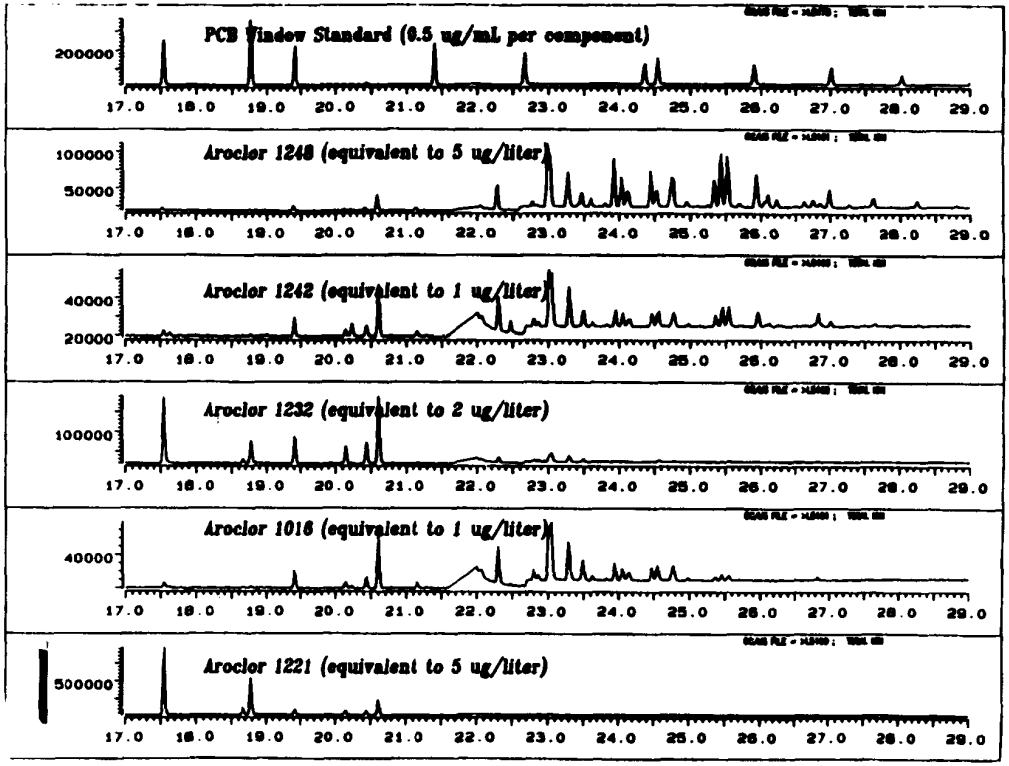
Sim Parameters: Number of groups: 3

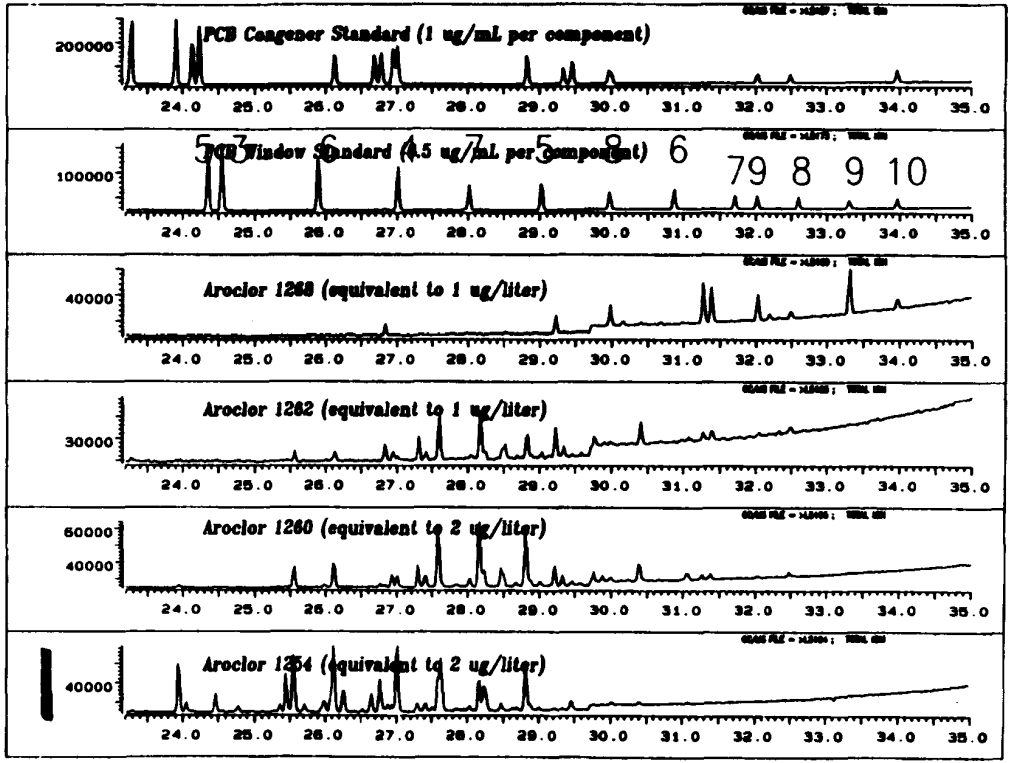
Multiplier voltage: 2600
Emission current: 300 uA

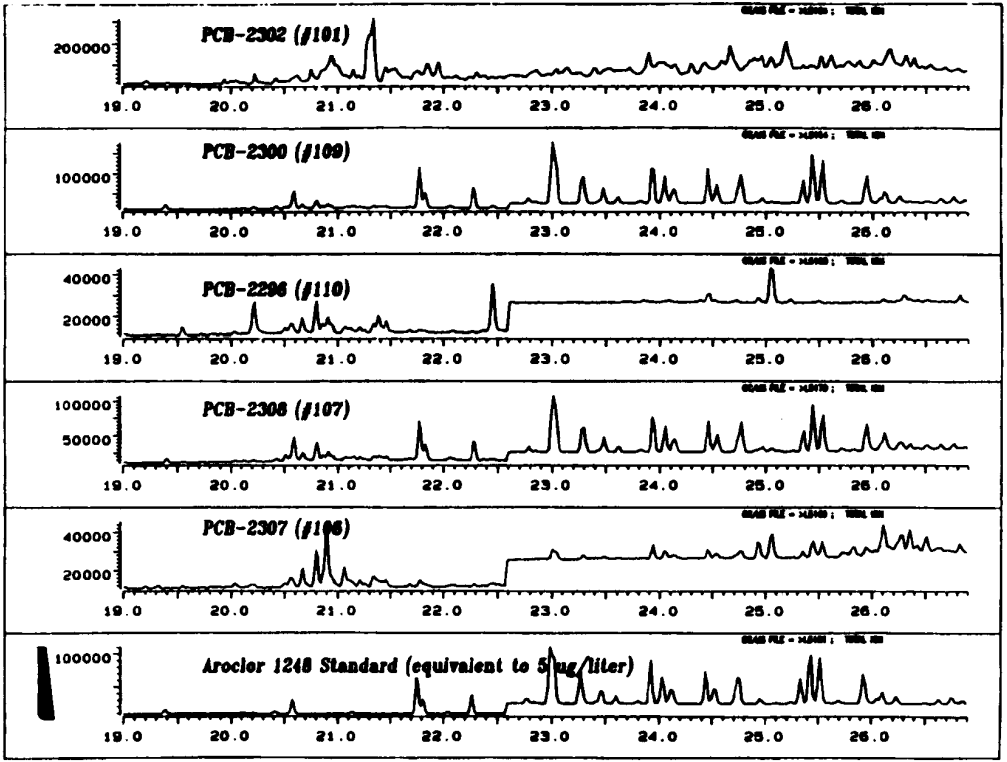
Group 1		Group 2		Group 3		Group 4		Group 5	
Start/Stop		Start/Stop		Start/Stop		Start/Stop		Start/Stop	
15.00 22.50		22.50 29.50		29.50 40.00		0.00 0.00		0.00 0.00	
M/Z	Dwell	M/Z	Dwell	M/Z	Dwell	M/Z	Dwell	M/Z	Dwell
-188.0	100	-255.9	100	-359.8	100	0.0	0	0.0	0
-190.0	100	-257.9	100	-361.8	100	0.0	0	0.0	0
-194.0	100	-289.8	100	-393.8	100	0.0	0	0.0	0
-196.0	100	-291.8	100	-395.8	100	0.0	0	0.0	0
-222.0	100	-301.8	100	-427.7	100	0.0	0	0.0	0
-224.0	100	-303.8	100	-429.7	100	0.0	0	0.0	0
-255.9	100	-323.9	100	-439.7	100	0.0	0	0.0	0
-257.9	100	-325.9	100	-441.7	100	0.0	0	0.0	0
0.0	0	-359.8	100	-461.6	100	0.0	0	0.0	0
0.0	0	-361.8	100	-463.6	100	0.0	0	0.0	0
0.0	0	-393.8	100	-497.6	100	0.0	0	0.0	0
0.0	0	-395.8	100	-499.6	100	0.0	0	0.0	0
0.0	0	0.0	0	-509.6	100	0.0	0	0.0	0
0.0	0	0.0	0	-511.6	100	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0

Appendix C. Summary of GC/MS Raw Data and Detailed Lotus/1-2-3 Outputs.

This section contains chromatograms and Lotus outputs generated for this set of samples. All chromatograms are the sums of the intensities of the selected masses monitored for PCBs.


PCB Window Standard (0.5 ug/mL per component)
200000
17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0
Aroclor 1248 (equivalent to 5 ug/liter)
100000
50000
17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0
Aroclor 1242 (equivalent to 1 ug/liter)
40000
20000
17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0
Aroclor 1232 (equivalent to 2 ug/liter)
100000
17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0
Aroclor 1016 (equivalent to 1 ug/liter)
40000
17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0
Aroclor 1221 (equivalent to 5 ug/liter)
500000
17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0


PCB Congener Standard (1 ug/mL per component)
PCB Window Standard (4.5 ug/mL per component)
Aroclor 1268 (equivalent to 1 ug/liter)
Aroclor 1262 (equivalent to 1 ug/liter)
Aroclor 1260 (equivalent to 2 ug/liter)
Aroclor 1254 (equivalent to 2 ug/liter)
200000
100000
40000
30000
60000
24.0
25.0
26.0
27.0
28.0
29.0
30.0
31.0
32.0
33.0
34.0
35.0


PCB-2302 (#101)
200000
19.0
20.0
21.0
22.0
23.0
24.0
25.0
26.0
PCB-2300 (#109)
100000
PCB-2296 (#110)
40000
20000
PCB-2306 (#107)
100000
50000
PCB-2307 (#108)
40000
20000
Arocior 1248 Standard (equivalent to 5 ng/liter)
100000

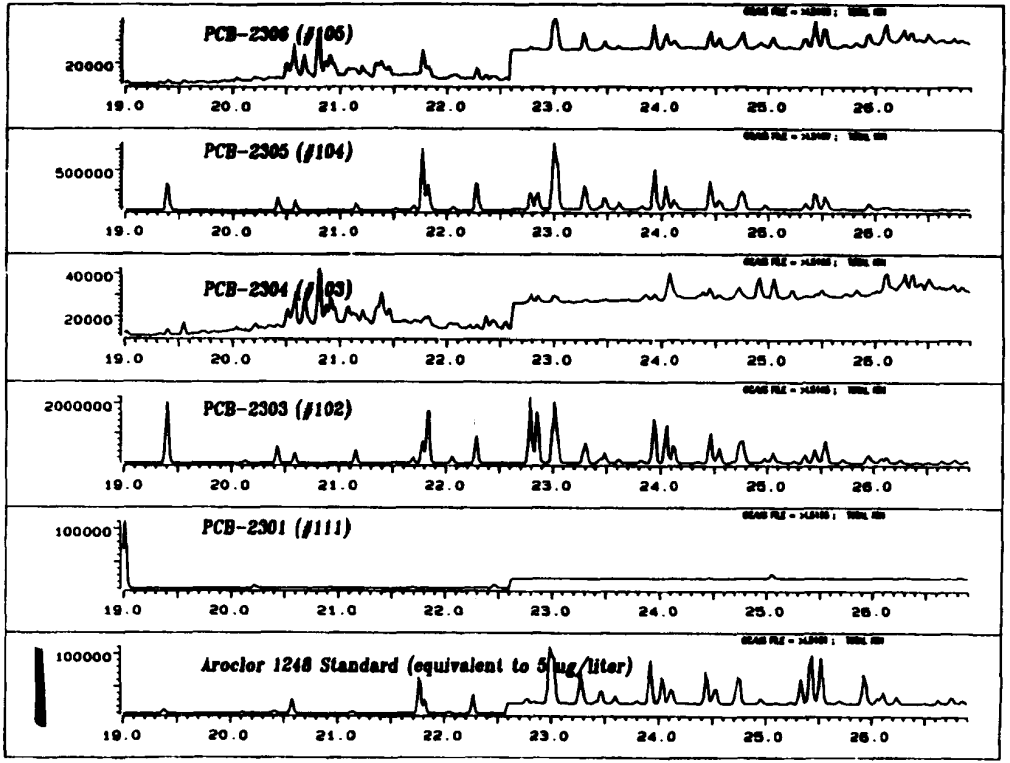

PCB-2306 (#106)
20000
19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0
PCB-2305 (#104)
500000
19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0
PCB-2304 (#103)
40000
20000
19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0
PCB-2303 (#102)
2000000
19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0
PCB-2301 (#111)
100000
19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0
Aroclor 1248 Standard (equivalent to 5 ug/liter)
100000
19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0

Table 1. Summary of Bourny PCB Analyses of Water for Project 48222

FRVS USED FOR CALIBRATION: HL5156 HL5157 HL5162

SAMPLE ID. ---	Meth Blank	#110	#110 DUP	#109	#111	#101	#102	#103	LS SPK	MS SPK
EXTRACT NUMBER ---	PCB 2295	PCB 2296	PCB 2297	PCB 2300	PCB 2301	PCB 2302	PCB 2303	PCB 2304	LS SPK	SPK
CONGENER CLASS	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Monochlorobiphenyls	0	0	0	0	0	0	0.3	0	0.3	3
Dichlorobiphenyls	0	0	0	0	0	0	10.7	0	0.4	4
Trichlorobiphenyls	0	0	0	1.7	0	0	30.6	0.1	0.3	3
Tetrachlorobiphenyls	0	0	0	3.5	0	0	53.2	0	0.3	3
Pentachlorobiphenyls	0	0	0	1.7	0	4.5	19.4	0	0.5	3
Hexachlorobiphenyls	0	0	0	0.1	0	3.8	3.6	0	0.5	2.9
Heptachlorobiphenyls	0	0	0	0	0	5.6	1.2	0	0.2	1.9
Octachlorobiphenyls	0	0	0	0	0	2.3	0.3	0	0.1	1
Nonachlorobiphenyls	0	0	0	0	0	0.1	0	0	0.1	1
Decachlorobiphenyl	0	0	0	0	0	0	0	0	0.1	1
TOTAL PCBS IN EXTRACT(ug/mL)	0	0	0	7	0	16.5	99.3	0.1	2.4	25.8
TOTAL PCBS IN SAMPLE (ug/L)	0	0	0	7	0	16.5	99.3	0.1	2.4	25.8

SURROGATE COMPOUND	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)
Monochlorobiphenyl-3C13	71	75	75	82	95	87	48	95	111	97
Tetrachlorobiphenyl-12C13	95	61	100	94	115	110	89	98	94	98
Octachlorobiphenyl-13C13	81	50	104	99	116	125	90	103	88	96
Decachlorobiphenyl-12C13	83	53	135	117	126	207	137	124	86	98

HP RUN FILE -	HL5149	HL5150	HL5151	HL5154	HL5155	HL5164	HL5165	HL5166	HL5162	HL5157
DILUTION FACTOR -	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
SAMPLE AMOUNT (mL) -	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
EXTRACT FINAL VOLUME (mL) -	1	1	1	1	1	1	1	1	1	1
ANTHRACENE D10 (1990) AREA -	12000000	12000000	11000000	12000000	11000000	7427305	13000000	15000000	13000000	12000000

Notes: Concentrations reported are rounded to 1 decimal place. Values of 0 should be interpreted as < 0.1 ug/mL.

Table Generation Comments: ALL FILES TEST OK

Figure 1-Sample Designation: MB #110 #110Dup #109 #111 #101 #102 #103 LS MS

MONS 025002

Table 1. a Summary of Kearny PCB Analyses
of Water for Project #8222

Table of Average Relative Response Factors
Generated for Each Congener Class

	Characteristic Relative Response Factors			Average Response Factor	Average Congener Response	
	Response from standard1	Response from standard2	Response from standard3			
Cl1 biphenyl #2	0.404	0.419	0.449	0.424		
Cl1 biphenyl #3	0.495	0.491	0.51	0.498	0.561777	Cl1
Cl1 biphenyl #4	0.746	0.768	0.796	0.763333		
Cl4biphenyl 6C1305	0.688	0.642	0.78	0.703333	0.703333	Cl1-C13
Cl2biphenyl #6	0.241	0.265	0.26	0.248666		
Cl2biphenyl #7	0.447	0.459	0.476	0.460666	0.301583	Cl2
Cl2biphenyl #8	0.339	0.362	0.388	0.363		
Cl2biphenyl #9	0.45	0.451	0.461	0.454		
Cl3biphenyl #10	0.288	0.293	0.312	0.297666		
Cl3biphenyl #11	0.297	0.297	0.302	0.298666	0.294222	Cl3
Cl3biphenyl #12	0.287	0.281	0.291	0.286333		
Cl4biphenyl #13	0.215	0.218	0.219	0.217333		
Cl4biphenyl #14	0.182	0.176	0.174	0.177333	0.205888	Cl4
Cl4biphenyl #15	0.219	0.225	0.225	0.223		
Cl4biph 12C13816	0.178	0.139	0.144	0.153666	0.153666	Cl4-C13
Cl5biphenyl #17	0.0687	0.0663	0.0603	0.0651		
Cl5biphenyl #18	0.0611	0.0541	0.0551	0.056766	0.0598	Cl5
Cl5biphenyl #19	0.0627	0.0564	0.0535	0.057533		
Cl6biphenyl #20	0.133	0.118	0.111	0.120666		
Cl6biphenyl #21	0.1	0.0861	0.0757	0.087266	0.092155	Cl6
Cl6biphenyl #22	0.08	0.0673	0.0583	0.068533		
Cl7biphenyl #23	0.0473	0.0384	0.0347	0.040133	0.037616	Cl7
Cl7biphenyl #24	0.0413	0.0343	0.0297	0.0351		
Cl8biphenyl #25	0.0251	0.0212	0.018	0.021433	0.021433	Cl8
Cl8bigh 12C13826	0.0677	0.0541	0.0502	0.057333	0.057333	Cl8-C13
Cl9biphenyl #27	0.0349	0.0285	0.0259	0.029766	0.029766	Cl9
Cl10biphenyl #28	0.0392	0.03	0.0249	0.031366	0.031366	Cl10
Cl10bigh 12C13829	0.0186	0.0138	0.0126	0.015	0.015	Cl10-C13

MONS 025003

Table 1. b Summary of Enormy PCB Analyses
of Water for Project 88222

COMPOUND ID	<---------- Characteristic Ion Response / Confirming Ion Response x 100 for PCBs Detected ---------->													<----------		
	STANDARD USED IN CALCULATIONS			FILE NAMES												
	S1	S2	S3	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10			
	>L5156	>L5157	>L5162	>L5149	>L5150	>L5151	>L5154	>L5155	>L5164	>L5165	>L5166	>L5162	>L5157			
001 = anthracene-d10	9523	9217	9231	9253	9009	8309	7878	8046	3078	8830	6307	9251	9217	9217	9523	9257
002 = Dich-Cl-biphenyl	306	298	283	0	0	0	0	0	0	276	0	283	298	283	306	298
003 = ICAL USED	299	300	282	0	0	0	0	0	0	0	0	282	300	282	300	296
004 = "	299	301	289	0	0	0	0	0	0	0	0	289	301	289	301	296
005 = 4-Cl-biphenyl-13C13	314	311	315	308	310	288	279	318	290	320	260	315	311	311	315	313
006 = Dich-Cl2-biphenyl	157	157	149	0	0	0	0	0	0	156	0	149	157	149	157	154
007 = ICAL USED	156	156	157	0	0	0	0	0	0	0	0	157	156	156	157	156
008 = "	144	155	161	0	0	0	0	0	0	0	0	161	155	144	161	153
009 = "	157	155	154	0	0	0	0	0	0	0	0	154	155	154	157	155
010 = Dich-Cl3-biphenyl	96	96	96	0	0	0	95	0	0	96	133	96	96	96	96	96
011 = ICAL USED	92	94	96	0	0	0	0	0	0	0	0	96	94	92	96	94
012 = "	97	95	97	0	0	0	0	0	0	0	0	97	95	95	97	96
013 = Dich-Cl4-biphenyl	78	81	80	0	0	0	81	0	0	81	0	80	81	78	81	80
014 = ICAL USED	81	82	81	0	0	0	0	0	0	0	0	81	82	81	82	81
015 = "	80	83	85	0	0	0	0	0	0	0	0	85	83	80	83	83
016 = Cl4-DP-12C13 Surr.	85	81	83	83	82	84	82	83	78	82	83	83	81	81	83	82
017 = Dich-Cl5-biphenyl	63	67	63	0	0	0	65	0	62	65	0	63	67	63	67	64
018 = ICAL USED	67	64	69	0	0	0	0	0	0	0	0	69	64	64	69	67
019 = "	62	66	66	0	0	0	0	0	0	0	0	66	66	62	66	66
020 = Dich-Cl6-biphenyl	133	129	130	0	0	0	148	0	156	130	0	130	129	129	133	131
021 = ICAL USED	120	131	133	0	0	0	0	0	0	0	0	133	131	120	133	131
022 = "	130	128	121	0	0	0	0	0	0	0	0	121	128	121	130	129
023 = Dich-Cl7-biphenyl	108	104	104	0	0	0	0	0	137	111	0	104	104	104	108	105
024 = ICAL USED	101	105	98	0	0	0	0	0	0	0	0	98	105	98	105	101
025 = Dich-Cl8-biphenyl	83	92	86	0	0	0	0	0	87	72	0	86	92	83	92	87
026 = Cl8-DP-12C13 Surr.	96	96	95	95	94	96	96	95	96	97	90	95	96	95	96	96
027 = Dich-Cl9-biphenyl	81	75	81	0	0	0	0	0	94	0	0	81	75	75	81	79
028 = Cl10-biphenyl	121	118	132	0	0	0	0	0	0	0	0	132	118	118	132	124
029 = Cl10-DP-12C13 Surr	118	118	126	118	118	116	120	130	122	117	118	126	118	118	126	121

MONS 025004

Table 1. c STATISTICAL EVALUATION OF SURROGATE SPIKING COMPOUNDS
Summary of Kearny PCB Analyses
of Water for Project #0222

SURROGATE COMPOUNDS	AVERAGE SURROGATE RECOVERIES (% REC)	STANDARD DEVIATION (% REC)	MINIMUM RECOVERY (% REC)	MAXIMUM RECOVERY (% REC)	NUMBER OF DETERMINATIONS
Monochlorobiphenyl-6C13	85	13	68	111	10
Tetrachlorobiphenyl-12C13	95	14	61	118	10
Octachlorobiphenyl-12C13	96	20	50	125	10
Decachlorobiphenyl-12C13	116	42	53	207	10
VALUES FOR ALL SURROGATES	98	27	50	207	40

FREQUENCY DISTRIBUTION OF RECOVERIES FOR : ~~32~~ 40 VALUES 7/11/88 BMA

	0-30 (% REC)	30-50 (% REC)	50-70 (% REC)	70-90 (% REC)	90-110 (% REC)	110-130 (% REC)	130-150 (% REC)	150-180 (% REC)	>180 (% REC)
Monochlorobiphenyl-6C13	0	0	4	2	3	1	0	0	0
Tetrachlorobiphenyl-12C13	0	0	1	2	5	2	0	0	0
Octachlorobiphenyl-12C13	0	1	0	2	5	2	0	0	0
Decachlorobiphenyl-12C13	0	0	1	2	1	3	2	0	1
FOR ALL SURROGATE VALUES	0	1	6	8	14	8	2	0	1

Table 2. Summary of Enormy PCB Analyses of Water for Project 08222

FANS USED FOR CALIBRATION: HLS163 HLS176 HLS179

SAMPLE ID. ---	0110+LS	0110+MS	0104	0105	0106	0107	0108	0112	0101 dil 1:10	0101
EXTRACT NUMBER ---	PCB 2298	PCB 2299	PCB 2305	PCB 2306	PCB 2307	PCB 2308	PCB 2309	PCB 2310	PCB 2302	PCB 2302
CONGENER CLASS	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Monochlorobiphenyls	0.3	2.6	0.1	0	0	0	0	0	0	0
Dichlorobiphenyls	0.4	3.7	2.4	0	0	0	0	0	0	0
Trichlorobiphenyls	0.2	2.6	10.1	0.2	0.1	1	0	0	0	0
Tetrachlorobiphenyls	0.2	2.5	9.6	0.3	0.2	1.0	0	0	0.2	0
Pentachlorobiphenyls	0.2	2.4	1.9	0.1	0.1	0.9	0	0	0.3	6.2
Hexachlorobiphenyls	0.2	2.7	0.1	0	0	0.1	0	0	0.2	5.5
Heptachlorobiphenyls	0.1	1.6	0	0	0	0	0	0	0.2	19.1
Octachlorobiphenyls	0.1	0.9	0	0	0	0	0	0	0	2.8
Nonachlorobiphenyls	0.1	0.9	0	0	0	0	0	0	0	0.3
Decachlorobiphenyl	0.1	0.9	0	0	0	0	0	0	0	0
TOTAL PCBS IN EXTRACT(ug/mL)	1.9	20.8	26.2	0.6	0.4	3.0	0	0	0.9	33.9
TOTAL PCBS IN SAMPLE (ug/L)	1.9	20.8	26.2	0.6	0.4	3.0	0	0	9	33.9
SURROGATE COMPOUND	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)
Monochlorobiphenyl-4C13	95	90	117	109	104	100	91	87	96	76
Tetrachlorobiphenyl-12C13	83	83	99	76	90	95	82	79	107	165
Octachlorobiphenyl-12C13	63	90	110	85	105	100	66	75	82	165
Decachlorobiphenyl-12C13	58	96	124	96	116	121	73	75	137	200
HS RAW FILE -	: HLS152	: HLS153	: HLS167	: HLS168	: HLS169	: HLS170	: HLS171	: HLS172	: HLS186	: HLS189
DILUTION FACTOR -	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.01	0.001
SAMPLE AMOUNT (mL) -	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
EXTRACT FINAL VOLUME (mL) -	1	1	1	1	1	1	1	1	10	1
AUTOINJECTED D10 (ISTD) AREA -	11000000	12000000	12000000	11000000	11000000	12000000	12000000	12000000	9000000	13000000

Note: Concentrations reported are rounded to 1 decimal place. Values of 0 should be interpreted as < 0.1 ug/mL.

Table Generation Comments: ALL FILES TEST OK

Figure 2-Sample Designation 0110+LS 0110+MS 0104 0105 0106 0107 0108 0112 0101 1:10 0101

Table 2. a Summary of Kearny PCB Analyses of Water for Project #8222

Table of Average Relative Response Factors Generated for Each Congener Class

	Characteristic Relative Response Factors			Average Response Factor	
	Response from standard1	Response from standard2	Response from standard5		Average Congener Response
Cl1 biphenyl #2	0.404	0.485	0.438	0.415666	
Cl1 biphenyl #3	0.481	0.472	0.516	0.489666	0.549866 Cl1
Cl1 biphenyl #4	0.724	0.723	0.784	0.743666	
Cl4biphenyl 6C13d5	0.649	0.648	0.652	0.649666	0.649866 Cl1-C13
Cl2biphenyl #6	0.251	0.222	0.235	0.229333	
Cl2biphenyl #7	0.448	0.399	0.441	0.429333	0.357833 Cl2
Cl2biphenyl #8	0.351	0.333	0.345	0.343	
Cl2biphenyl #9	0.462	0.387	0.44	0.429666	
Cl3biphenyl #10	0.295	0.288	0.304	0.295666	
Cl3biphenyl #11	0.325	0.281	0.319	0.308333	0.297666 Cl3
Cl3biphenyl #12	0.301	0.267	0.297	0.288333	
Cl4biphenyl #13	0.238	0.215	0.227	0.226666	
Cl4biphenyl #14	0.193	0.173	0.189	0.185	0.212333 Cl4
Cl4biphenyl #15	0.239	0.206	0.231	0.225333	
Cl4biph 12C13d16	0.184	0.17	0.171	0.175	0.175 Cl4-C13
Cl5biphenyl #17	0.0786	0.0647	0.0699	0.071066	
Cl5biphenyl #18	0.0697	0.0565	0.0619	0.0627	0.066833 Cl5
Cl5biphenyl #19	0.0708	0.059	0.0632	0.064333	
Cl6biphenyl #20	0.14	0.0995	0.113	0.117433	
Cl6biphenyl #21	0.11	0.0793	0.0913	0.093533	0.095266 Cl6
Cl6biphenyl #22	0.0886	0.0659	0.07	0.074833	
Cl7biphenyl #23	0.0522	0.0475	0.0463	0.048666	0.045416 Cl7
Cl7biphenyl #24	0.0472	0.0378	0.0415	0.042166	
Cl8biphenyl #25	0.0289	0.0252	0.0261	0.026733	0.026733 Cl8
Cl8biph 12C13d26	0.0728	0.0498	0.0572	0.059933	0.059933 Cl8-C13
Cl9biphenyl #27	0.0371	0.0345	0.0328	0.0348	0.0348 Cl9
Cl10biphenyl #28	0.0409	0.0355	0.0349	0.0371	0.0371 Cl10
Cl10biph 12C13d29	0.0193	0.0161	0.0175	0.017633	0.017633 Cl10-C13

Table 2. b Summary of Kearny PCB Analyses
of Water for Project 88222

COMPOUND ID	<---------- Characteristic Ion Response / Confirming Ion Response x 100 for PCBs Detected ---------->														MINIMUM RATIO	MAXIMUM RATIO	AVERAGE RATIO
	STANDARD USED IN CALCULATIONS			FILE NAMES													
	S1	S2	S3	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10				
	>L5163	>L5176	>L5175	>L5152	>L5153	>L5167	>L5168	>L5169	>L5170	>L5171	>L5172	>L5196	>L5197				
001 = anthracene-d10	9292	9520	9571	8295	8510	9164	8108	8761	7671	9082	9222	7650	2502		9092	9520	9004
002 = CL-biphenyl #1	301	285	305	235	284	256	0	0	0	0	0	0	0		285	305	297
003 = " #2	300	294	306	274	295	0	0	0	0	0	0	0	0		294	306	300
004 = " #3	296	291	304	305	296	0	0	0	0	0	0	0	0		291	304	297
005 = 4-CL-biphenyl-dc13	315	311	306	290	295	314	270	311	263	300	310	311	290		306	315	301
006 = CL2-biphenyl #1	155	159	155	158	157	159	0	0	0	0	0	0	0		155	159	156
007 = " #2	154	153	157	162	156	0	0	0	0	0	0	0	0		153	157	155
008 = " #3	154	158	155	128	145	0	0	0	0	0	0	0	0		154	158	156
009 = " #4	155	159	158	178	157	0	0	0	0	0	0	0	0		150	158	154
010 = CL3-biphenyl #1	95	100	98	90	98	96	95	107	99	0	0	0	0		95	100	98
011 = " #2	98	94	95	98	94	0	0	0	0	0	0	0	0		94	98	95
012 = " #3	98	97	95	94	95	0	0	0	0	0	0	0	0		95	97	96
013 = CL4-biphenyl #1	81	84	80	71	80	81	79	138	78	0	0	81	0		80	84	82
014 = " #2	79	83	81	78	80	0	0	0	0	0	0	0	0		79	83	80
015 = " #3	79	79	81	83	82	0	0	0	0	0	0	0	0		79	83	80
016 = CL4-DP-12C13 Surr.	81	80	81	81	83	82	82	82	83	81	81	71	61		80	81	81
017 = CL5-biphenyl #1	68	66	66	68	65	67	67	64	69	0	0	92	66		66	68	67
018 = " #2	68	67	67	59	66	0	0	0	0	0	0	0	0		67	68	67
019 = " #3	65	67	65	65	66	0	0	0	0	0	0	0	0		65	67	66
020 = CL6-biphenyl #1	127	114	129	127	129	179	0	0	139	0	0	177	160		114	129	135
021 = " #2	128	134	128	120	130	0	0	0	0	0	0	0	0		128	134	130
022 = " #3	128	131	123	134	129	0	0	0	0	0	0	0	0		123	131	127
023 = CL7-biphenyl #1	103	100	100	101	102	0	0	0	0	0	0	146	100		100	103	101
024 = " #2	105	95	105	84	104	0	0	0	0	0	0	0	0		95	105	102
025 = CL8-biphenyl	97	120	92	81	91	0	0	0	0	0	0	0	77		92	120	105
026 = CL8-DP-12C13 Surr.	97	92	97	94	96	96	96	95	94	93	94	88	94		92	97	95
027 = CL9-biphenyl	76	80	77	81	76	0	0	0	0	0	0	0	134		76	80	84
028 = CL10-biphenyl	117	125	113	144	118	0	0	0	0	0	0	0	122		113	125	118
029 = CL10-DP-12C13 Surr	117	108	110	127	116	110	122	117	121	110	110	113	114		108	110	114

MONS 025008

Table 2. c STATISTICAL EVALUATION OF SURROGATE SPIKING COMPOUNDS
Summary of Kearny PCB Analyses
of Water for Project #8222

SURROGATE COMPOUNDS	AVERAGE SURROGATE RECOVERIES (% REC)	STANDARD DEVIATION (% REC)	MINIMUM RECOVERY (% REC)	MAXIMUM RECOVERY (% REC)	NUMBER OF DETERMINATIONS
Monochlorobiphenyl-6C13	97	12	76	117	10
Tetrachlorobiphenyl-12C13	96	26	76	165	10
Octachlorobiphenyl-12C13	94	30	63	165	10
Decachlorobiphenyl-12C13	112	48	58	228	10
VALUES FOR ALL SURROGATES	100	31	58	228	40

FREQUENCY DISTRIBUTION OF RECOVERIES FOR : ~~40~~ VALUES 7/11/88

	0-30 (% REC)	30-50 (% REC)	50-70 (% REC)	70-90 (% REC)	90-110 (% REC)	110-130 (% REC)	130-150 (% REC)	150-180 (% REC)	>180 (% REC)
Monochlorobiphenyl-6C13	0	0	1	2	6	1	0	0	0
Tetrachlorobiphenyl-12C13	0	0	2	4	3	0	0	1	0
Octachlorobiphenyl-12C13	0	0	3	3	3	0	0	1	0
Decachlorobiphenyl-12C13	0	0	3	0	2	3	1	0	1
FOR ALL SURROGATE VALUES	0	0	9	9	14	4	1	2	1

Table 3. Native Spiking QA/QC Results for Kearny PCB in Water Analyses

(Where MS = Addition of 26 ug and LS = Addition of 2.6 ug PCBs) Proj# 8222

MS CALIB FILE: >LS157

LS CALIB FILE: >LS156

COMPOUND ID	#110+MS PCB 2299 (% REC)	MS SPK (% REC)	MS SPK (% REC)	#110 + LS PCB 2298 (% REC)	LS SPK (% REC)	LS SPK (% REC)
#02 = CL-biphenyl #1	87	100	105	95	100	100
#03 = " #2	85	100	105	82	100	96
#04 = " #3	87	100	105	87	100	97
#06 = CL2-biphenyl #1	84	100	96	82	100	92
#07 = " #2	85	100	96	85	100	89
#08 = " #3	87	100	95	116	100	98
#09 = " #4	88	100	98	90	100	86
#10 = CL3-biphenyl #1	86	100	104	79	100	100
#11 = " #2	91	100	107	87	100	95
#12 = " #3	86	100	106	84	100	95
#13 = CL4-biphenyl #1	88	100	104	83	100	100
#14 = " #2	86	100	107	80	100	95
#15 = " #3	86	100	103	81	100	96
#17 = CL5-biphenyl #1	87	100	105	87	100	94
#18 = " #2	91	100	114	70	100	92
#19 = " #3	92	100	112	75	100	94
#20 = CL6-biphenyl #1	88	100	96	63	100	75
#21 = " #2	99	100	106	63	100	79
#22 = " #3	100	100	104	70	100	82
#23 = CL7-biphenyl #1	97	100	121	77	100	100
#24 = " #2	101	100	121	70	100	92
#25 = CL8-biphenyl	115	100	123	70	100	100
#27 = CL9-biphenyl	106	100	115	79	100	99
#28 = CL10-biphenyl	115	100	116	59	100	91
SURROGATE COMPOUNDS						
#05 = 4-CL-biphenyl-6C13	91	100	102	90	100	94
#16 = CL4-BP-12C13 Surr.	104	100	123	81	100	96
#26 = CL8-BP-12C13 Surr.	99	100	106	56	100	76
#29 = CL10-BP-12C13 Surr	122	100	127	55	100	87
MS RAW FILE -	: >LS153	: >LS157	: >LS175	: >LS152	: >LS156	: >LS174

Note - Recoveries are rounded to the nearest whole number

Table 4. Summary of Aroclor Formulations Analyzed with Congener Response Factors Used in Kearny PCB in Water Analysis Quantitation

RUNS USED FOR CALIBRATION: HLS163 HLS176 HLS175

SAMPLE ID. ···	A1248 (1ppm)		A1248 (10 ppm)		A1262 (1 ppm)		A1221 (5 ppm)		A1254 (2 ppm)
EXTRACT NUMBER ··· A1248 (0.1ppm)	A1248 (5 ppm)		A1260 (2 ppm)		A1016 (1 ppm)		A1232 (2 ppm)		
CONGENER CLASS	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Monochlorobiphenyls	0	0	0.01	0.03	0	0	0.01	3.64	0.59	0
Dichlorobiphenyls	0	0.04	0.17	0.32	0	0	0.2	1.44	1.13	0
Trichlorobiphenyls	0.02	0.21	1.11	2.26	0	0	0.5	0.19	0.35	0.02
Tetrachlorobiphenyls	0.04	0.51	2.34	4.85	0	0	0.24	0.08	0.04	0.29
Pentachlorobiphenyls	0	0.16	1.07	2.26	0.4	0	0	0	0	1.26
Hexachlorobiphenyls	0	0	0.06	0.16	0.95	0.25	0	0	0	0.58
Heptachlorobiphenyls	0	0	0	0	0.29	0.35	0	0	0	0.06
Octachlorobiphenyls	0	0	0	0	0.17	0	0	0	0	0
Nonachlorobiphenyls	0	0	0	0	0	0	0	0	0	0
Decachlorobiphenyl	0	0	0	0	0	0	0	0	0	0
TOTAL PCBS IN EXTRACT(ug/mL)	0.06	0.92	4.76	9.88	2.29	0.6	0.95	5.35	2.11	2.16
TOTAL PCBS IN SAMPLE (ug/g)	0.06	0.92	4.76	9.88	2.29	0.6	0.95	5.35	2.11	2.16
SURROGATE COMPOUND	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)
Monochlorobiphenyl-4C13	0	0	0	0	0	0	0	0	0	0
Tetrachlorobiphenyl-12C13	0	0	0	0	0	0	0	0	0	0
Octachlorobiphenyl-12C13	0	0	0	0	0	0	0	0	0	0
Decachlorobiphenyl-12C13	0	0	0	0	0	0	0	0	0	0

MS DOS FILE ·	HLS179	HLS180	HLS181	HLS182	HLS195	HLS186	HLS191	HLS190	HLS192	HLS196
DILUTION FACTOR ·	1	1	1	1	1	1	1	1	1	1
SAMPLE AMOUNT (g) ·	1	1	1	1	1	1	1	1	1	1
EXTRACT FINAL VOLUME (mL) ·	1	1	1	1	1	1	1	1	1	1
ANTHRACENE D10 (1570) AREA ·	12000000	11000000	11000000	11000000	14000000	13000000	13000000	13000000	13000000	15000000

Note: Concentrations reported are rounded to 2 decimal places. Values of 0 should be interpreted as < 0.1 ug/g.

Table Generation Comments: ALL FILES TEST OK

MONS 025011

Table 4. a Summary of Aroclor Formulations Analyzed with Congener Response Factors Used in Kearny PCB in Water Analysis Quantitation

Table of Average Relative Response Factors
Generated for Each Congener Class

	Characteristic Relative Response Factors			Average Response Factor	
	Response from standard1	Response from standard2	Response from standard5		Average Congener Response
Cl1 biphenyl #2	0.404	0.485	0.438	0.415666	
Cl1 biphenyl #3	0.481	0.472	0.516	0.489666	0.549666 Cl1
Cl1 biphenyl #4	0.726	0.723	0.706	0.743666	
Cl4biphenyl 4C13#5	0.649	0.648	0.652	0.649666	0.649666 Cl1-C13
Cl2biphenyl #6	0.231	0.222	0.235	0.229333	
Cl2biphenyl #7	0.448	0.399	0.441	0.429333	0.357833 Cl2
Cl2biphenyl #8	0.351	0.333	0.345	0.343	
Cl2biphenyl #9	0.442	0.387	0.44	0.429666	
Cl3biphenyl #10	0.295	0.288	0.304	0.295666	
Cl3biphenyl #11	0.325	0.281	0.319	0.308333	0.297444 Cl3
Cl3biphenyl #12	0.301	0.267	0.297	0.288333	
Cl4biphenyl #13	0.238	0.215	0.227	0.226666	
Cl4biphenyl #14	0.193	0.173	0.189	0.185	0.212333 Cl4
Cl4biphenyl #15	0.239	0.206	0.231	0.225333	
Cl4biph 12C13#16	0.184	0.17	0.171	0.175	0.175 Cl4-C13
Cl5biphenyl #17	0.0706	0.0647	0.0699	0.071066	
Cl5biphenyl #18	0.0607	0.0569	0.0619	0.0627	0.066033 Cl5
Cl5biphenyl #19	0.0708	0.059	0.0632	0.064333	
Cl6biphenyl #20	0.14	0.0993	0.113	0.117633	
Cl6biphenyl #21	0.11	0.0793	0.0913	0.093533	0.095266 Cl6
Cl6biphenyl #22	0.0806	0.0659	0.07	0.074833	
Cl7biphenyl #23	0.0522	0.0475	0.0463	0.048666	0.045416 Cl7
Cl7biphenyl #24	0.0472	0.0378	0.0415	0.042166	
Cl8biphenyl #25	0.0289	0.0252	0.0261	0.026733	0.026733 Cl8
Cl8biph 12C13#26	0.0728	0.0498	0.0572	0.059933	0.059933 Cl8-C13
Cl9biphenyl #27	0.0371	0.0345	0.0328	0.0348	0.0348 Cl9
Cl10biphenyl #28	0.0409	0.0355	0.0349	0.0371	0.0371 Cl10
Cl10biph 12C13#29	0.0193	0.0161	0.0175	0.017633	0.017633 Cl10-C13

Table 6. b Summary of Aroclor Formulations Analyzed with Congener Response Factors Used in Osprey PCB in Water Analysis Quantitation

COMPOUND ID	Characteristic Ion Response / Confirming Ion Response x 100 for PCBs Detected																
	STANDARD USED IN CALCULATIONS			FILE NAMES													
	S1	S2	S3	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10				
	>LS163	>LS176	>LS175	>LS179	>LS180	>LS181	>LS182	>LS195	>LS188	>LS191	>LS190	>LS192	>LS194				
#01 = anthracene-d10	9292	9520	9371	9350	9333	9278	0121	9494	9371	9755	9454	9378	9536	9098	9520	9086	
#02 = Dich-Cl-biphenyl	301	285	305	0	0	221	316	0	0	378	305	307	0	285	306	297	
#03 = HRR U009	300	294	306	0	0	0	0	0	0	0	0	0	0	294	306	300	
#04 = "	296	291	306	0	0	0	0	0	0	0	0	0	0	291	306	299	
#05 = 4-Cl-biphenyl-dc13	315	311	306	0	0	0	0	0	0	0	0	0	0	306	315	311	
#06 = Dich-Cl2-biphenyl	155	159	155	0	273	162	164	0	0	167	150	156	0	155	159	156	
#07 = HRR U008	154	153	157	0	0	0	0	0	0	0	0	0	0	153	157	155	
#08 = "	154	150	155	0	0	0	0	0	0	0	0	0	0	154	150	152	
#09 = "	155	150	150	0	0	0	0	0	0	0	0	0	0	150	150	150	
#10 = Dich-Cl3-biphenyl	95	100	98	96	96	96	96	0	0	98	94	100	116	95	100	98	
#11 = HRR U008	96	94	95	0	0	0	0	0	0	0	0	0	0	94	96	95	
#12 = "	96	97	95	0	0	0	0	0	0	0	0	0	0	95	97	96	
#13 = Dich-Cl4-biphenyl	81	86	80	89	80	80	81	0	0	81	81	85	90	80	86	83	
#14 = HRR U008	79	83	81	0	0	0	0	0	0	0	0	0	0	79	83	81	
#15 = "	79	79	81	0	0	0	0	0	0	0	0	0	0	79	79	80	
#16 = Cl4-BP-12C13 Surr.	81	80	81	0	0	0	0	0	0	0	0	0	0	80	81	81	
#17 = Dich-Cl5-biphenyl	68	66	66	0	68	65	67	80	0	0	0	0	70	66	68	67	
#18 = HRR U008	68	67	67	0	0	0	0	0	0	0	0	0	0	67	68	67	
#19 = "	65	67	65	0	0	0	0	0	0	0	0	0	0	65	67	66	
#20 = Dich-Cl6-biphenyl	127	116	129	0	0	123	103	134	129	0	0	0	127	116	129	103	
#21 = HRR U008	128	134	128	0	0	0	0	0	0	0	0	0	0	128	134	130	
#22 = "	128	131	123	0	0	0	0	0	0	0	0	0	0	123	131	127	
#23 = Dich-Cl7-biphenyl	105	108	108	0	0	0	0	109	115	0	0	0	217	108	108	108	
#24 = HRR U008	105	95	105	0	0	0	0	0	0	0	0	0	0	95	105	100	
#25 = Dich-Cl8-biphenyl	97	128	92	0	0	0	0	78	0	0	0	0	0	92	128	106	
#26 = Cl8-BP-12C13 Surr.	97	92	97	100	0	0	0	0	0	0	0	0	0	92	97	95	
#27 = Dich-Cl9-biphenyl	76	89	77	0	0	0	0	0	0	0	0	0	0	76	89	81	
#28 = Cl10-biphenyl	117	125	113	0	0	0	0	0	0	0	0	0	0	113	125	118	
#29 = Cl10-BP-12C13 Surr	117	108	118	100	0	0	0	0	0	0	0	0	0	108	118	114	

Table 5. Summary of Aroclor Formulations Analyzed with Congener Response Factors Used in Enormy PCB in Water Analysis Quantitation

FILES USED FOR CALIBRATION: >L5163 >L5176 >L5175

SAMPLE ID. ---	A1260 (1 ppm)	
EXTRACT NUMBER ---		A1262 (1 ppm)
CONGENER CLASS	(ug/mL)	(ug/mL)
Monochlorobiphenyls	0	0.01
Dichlorobiphenyls	0	0.19
Trichlorobiphenyls	0	0.30
Tetrachlorobiphenyls	0	0.29
Pentachlorobiphenyls	0	0.04
Hexachlorobiphenyls	0	0
Heptachlorobiphenyls	0.09	0
Octachlorobiphenyls	0.82	0
Nonachlorobiphenyls	0.48	0
Decachlorobiphenyl	0.08	0
TOTAL PCBS IN EXTRACT(ug/mL)	1.47	0.92
TOTAL PCBS IN SAMPLE (ug/mL)	1.47	0.92

SURROGATE COMPOUND	(% REC)	(% REC)
Monochlorobiphenyl-4C13	0	0
Tetrachlorobiphenyl-12C13	0	0
Octachlorobiphenyl-12C13	0	0
Decachlorobiphenyl-12C13	0	0
RQ DATA FILE -	: >L5180	: >L5195
DILUTION FACTOR -	1	1
SAMPLE AMOUNT (g) -	1	1
EXTRACT FINAL VOLUME (mL) -	1	1
ANTHRACENE D10 (ISTD) AREA -	14000000	14000000

Note: Concentrations reported are rounded to 2 decimal places. Values of 0 should be interpreted as < 0.1 ug/mL.

Table Generation Comments: ALL FILES TEST OK

Table 5. a Summary of Aroclor Formulations Analyzed with Congener Response Factors Used in Kearny PCB in Water Analysis Quantitation

Table of Average Relative Response Factors Generated for Each Congener Class

	Characteristic Relative Response Factors			Average Response Factor	Average Congener Response	
	Response from standard1	Response from standard2	Response from standard3			
Cl1 biphenyl #2	0.404	0.408	0.438	0.415466		
Cl1 biphenyl #3	0.481	0.472	0.516	0.489666	0.349666	Cl1
Cl1 biphenyl #4	0.724	0.723	0.784	0.743666		
Cl4biphenyl &C13#5	0.649	0.648	0.652	0.649666	0.649666	Cl1-C13
Cl2biphenyl #6	0.231	0.222	0.235	0.229333		
Cl2biphenyl #7	0.448	0.399	0.441	0.429333	0.357833	Cl2
Cl2biphenyl #8	0.351	0.333	0.345	0.343		
Cl2biphenyl #9	0.462	0.387	0.44	0.429666		
Cl3biphenyl #10	0.295	0.288	0.304	0.295666		
Cl3biphenyl #11	0.325	0.281	0.319	0.308333	0.297444	Cl3
Cl3biphenyl #12	0.301	0.267	0.297	0.288333		
Cl4biphenyl #13	0.258	0.215	0.227	0.226666		
Cl4biphenyl #14	0.193	0.173	0.189	0.185	0.212333	Cl4
Cl4biphenyl #15	0.239	0.206	0.231	0.225333		
Cl4biph 12C13#16	0.184	0.17	0.171	0.175	0.175	Cl4-C13
Cl5biphenyl #17	0.0786	0.0647	0.0699	0.071066		
Cl5biphenyl #18	0.0607	0.0565	0.0619	0.0627	0.066033	Cl5
Cl5biphenyl #19	0.0708	0.059	0.0632	0.064333		
Cl6biphenyl #20	0.14	0.0993	0.113	0.117433		
Cl6biphenyl #21	0.11	0.0793	0.0913	0.093533	0.095266	Cl6
Cl6biphenyl #22	0.0886	0.0659	0.07	0.074833		
Cl7biphenyl #23	0.0522	0.0475	0.0463	0.048666	0.045416	Cl7
Cl7biphenyl #24	0.0472	0.0378	0.0415	0.042166		
Cl8biphenyl #25	0.0289	0.0252	0.0261	0.026733	0.026733	Cl8
Cl8biph 12C13#26	0.0728	0.0498	0.0572	0.059933	0.059933	Cl8-C13
Cl9biphenyl #27	0.0371	0.0345	0.0328	0.0348	0.0348	Cl9
Cl10biphenyl #28	0.0409	0.0355	0.0349	0.0371	0.0371	Cl10
Cl10biph 12C13#29	0.0193	0.0161	0.0175	0.017633	0.017633	Cl10-C13

Table 5. b Summary of Aroclor Formulations Analyzed with Congener Response Factors Used in Saarmy PCB in Water Analysis Quantitation

COMPOUND ID	Characteristic Ion Response / Confirming Ion Response x 100 For PCBs Detected													STANDARD RATIO	MINIMUM RATIO <	MAXIMUM RATIO >	AVERAGE RATIO -
	STANDARD USED IN CALCULATIONS			FILE NAMES													
	01	02	03	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10				
	>LS168	>LS176	>LS175	>LS180	>LS195	>LS181	>LS182	>LS195	>LS188	>LS191	>LS190	>LS192	>LS194				
001 = anthracene-d10	9092	9520	9371	9486	9500	9270	9121	9494	9371	9755	9454	9570	9536	9092	9520	9804	
002 = 3SM-CL-biphenyl	301	288	305	0	312	221	316	0	0	378	305	307	0	288	305	297	
003 = 3SM UNID	300	294	306	0	0	0	0	0	0	0	0	0	0	294	306	300	
004 = "	296	291	304	0	0	0	0	0	0	0	0	0	0	291	304	297	
005 = 4-CL-biphenyl-dC13	315	311	306	0	0	0	0	0	0	0	0	0	0	306	315	311	
006 = 3SM-CL2-biphenyl	155	150	155	0	175	162	144	0	0	167	150	154	0	155	150	156	
007 = 2SM UNID	154	153	157	0	0	0	0	0	0	0	0	0	0	153	157	155	
008 = "	154	150	155	0	0	0	0	0	0	0	0	0	0	154	150	154	
009 = "	155	150	150	0	0	0	0	0	0	0	0	0	0	150	150	154	
010 = 3SM-CL3-biphenyl	95	100	98	0	97	96	96	0	0	96	94	100	116	95	100	98	
011 = 3SM UNID	96	94	95	0	0	0	0	0	0	0	0	0	0	94	96	95	
012 = "	96	97	95	0	0	0	0	0	0	0	0	0	0	95	97	96	
013 = 3SM-CL4-biphenyl	81	84	80	0	79	80	81	0	0	81	81	88	90	80	84	82	
014 = 3SM UNID	79	83	81	0	0	0	0	0	0	0	0	0	0	79	83	81	
015 = "	79	79	81	0	0	0	0	0	0	0	0	0	0	79	81	80	
016 = CL6-DP-13C13 Surr.	81	80	81	0	0	0	0	0	0	0	0	0	0	80	81	81	
017 = 3SM-CL5-biphenyl	66	64	66	0	63	65	67	89	0	0	0	70	0	64	66	67	
018 = 3SM UNID	66	67	67	0	0	0	0	0	0	0	0	0	0	67	66	67	
019 = "	65	67	65	0	0	0	0	0	0	0	0	0	0	65	67	66	
020 = 3SM-CL6-biphenyl	127	114	129	0	0	123	103	134	129	0	0	0	127	114	129	103	
021 = 3SM UNID	128	134	128	0	0	0	0	0	0	0	0	0	0	128	134	130	
022 = "	126	131	123	0	0	0	0	0	0	0	0	0	0	123	131	127	
023 = 3SM-CL7-biphenyl	103	100	100	102	0	0	0	109	115	0	0	0	217	100	100	105	
024 = 3SM UNID	105	95	105	0	0	0	0	0	0	0	0	0	0	95	105	102	
025 = 3SM-CL8-biphenyl	97	120	92	97	0	0	0	70	0	0	0	0	0	92	120	96	
026 = CL8-DP-13C13 Surr.	97	92	97	0	0	0	0	0	0	0	0	0	0	92	97	95	
027 = 3SM-CL9-biphenyl	76	89	77	80	0	0	0	0	0	0	0	0	0	76	89	84	
028 = CL10-biphenyl	117	123	113	112	0	0	0	0	0	0	0	0	0	113	123	118	
029 = CL10-DP-13C13 Surr	117	108	110	0	0	0	0	0	0	0	0	0	0	108	110	114	

MONS 025016

Appendix D. Important analytical request and chain-of-custody information recorded at sample login.

Time Stamp - <880527.0915>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: APRIL 25, 1988 LOG NO: U-88-04-25-02

REQUESTER: MARTIN PROJECT NO: 760.28-8222

LOGIN NOTEBOOK PAGE (To be filled out at login.): 3759259

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

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

SAMPLE PICKUP BY: MCKENZIE

SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 15 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
103	1 X 1 L	
103	1 X 1 L	
105	1 X 1 L	
107	1 X 1 L	
109	1 X 1 L	
101	1 X 1 L	
110	1 X 1 L	
110	1 X 1 L	
110	1 X 1 L	
110	1 X 1 L	
106	1 X 1 L	
108	1 X 1 L	
111	1 X 1 L	
102	1 X 1 L	
104	1 X 1 L	
112	1 X 1 L	

RECORDED BY: MINOR

REQUIRED ANALYSES: GC CHROMATOGRAPHABLES

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158,646

ESC STANDARD EXTRACTION METHOD(S): WSEX05

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	COMPLETED	ANALYSIS METHOD	LOGBOOK PAGE
X CCGC/MS ( )	5/27/88	MSAN25	156,603 - 156,604

FINAL REPORT NOTEBOOK PAGE: 3,434,482

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:AA8222:LI

ROUX ASSOCIATES

CHAIN OF CUSTODY RECORD - MONSANTO-1

Project No. 0660S

Project Title MONSANTO - KEARNY

Sample Source N/A

Collectors Name G. MARTIN / R. CROWELL / Gregory Martin
print signature

Field Information N/A

Method Of Shipping Fed Ex

Relinquished By:
sign Gregory Martin
for ROUX ASSOC.
Date/Time 4/21/88 @ 2030

Received By:
sign
for
Date/Time

Sample Designation	Sample Location	Date	Time	Analyte	No. Of Containers
103	N/A	4/21/88	1440	625/680 pcb *	1 - 1l glass
105	↓	↓	1440	↓	↓
107			1440		
109	↓	↓	1440	↓	↓

Comments:

* Discuss with Monsanto Lab

MONS 025020

4/25/88

ROUX ASSOCIATES

CHAIN OF CUSTODY RECORD - MONSANTO - 2

Project No. 06605

Project Title MONSANTO - KEARNY

Sample Source N/A

Collectors Name G. MARTIN / R. CROWELL / Gregory Marsh
print signature

Field Information N/A

Method Of Shipping FED EX

Relinquished By:	Received By:
sign Gregory Marsh	sign
for ROUX ASSOC.	for
Date/Time 4/21/89 @ 2030	Date/Time

Sample Designation	Sample Location	Date	Time	Analyte	No. Of Containers
101	N/A	4/21/89	1630	625/680 PCBS *	1-1L amber
~~DUP~~ 110	↓	↓	1505	↓	↓
~~DUP~~ 111			1505		
~~DUP~~ 110			1505		

Comments:

* Discuss w Monsanto Lab

4/25/89 JR

ROUX ASSOCIATES

CHAIN OF CUSTODY RECORD – MONSANTO – 3

Project No. 06605

Project Title MONSANTO KEARNY

Sample Source N/A

Collectors Name G. MARTIN / R. CERWALL / (print) (signature)

Field Information N/A

Method Of Shipping FED EX

Relinquished By:	Received By:
sign	sign
for ROUX ASSOC.	for
Date/Time 4/21/88 @ 2030	Date/Time

Sample Designation	Sample Location	Date	Time	Analyte	No. Of Containers
106	N/A	4/21/88	1115	625/680 pcbs *	1-1L glass
108	↓	↓	1125		
110			1410		
111	↓	↓	1400		

Comments:

* Discuss w Monsanto Lab

4/25/88

MONS 025022

ROUX ASSOCIATES

CHAIN OF CUSTODY RECORD - MONSANTO - 4

Project No. 0660S

Project Title MONSANTO - KEARNY

Sample Source N/A

Collectors Name G. MARTIN/R. CRAWELL / Gregory Mart
print signature

Field Information N/A

Method Of Shipping Fed Ex

Relinquished By:
sign Gregory Mart
for Roux Assoc
Date/Time 4/21/88 @ 2030

Received By:
sign
for
Date/Time

Sample Designation	Sample Location	Date	Time	Analyte	No. Of Containers
102	N/A	4/21/88	1930	625/680 pcbs *	1- 1l glass
104	↓	↓	1900	↓	↓
112	↓	↓	1710	↓	↓

Comments: * Discuss with Monsanto Lab

MONS 025023

4/25/88

Appendix E. Descriptions of important terminology used in Quality Assurance/Quality Control, Analyte Extraction, and Instrumental Analysis.

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 replicate instrumental analyses must also 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.

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 on the order of 30%. Normally, the concentrations of standard solutions which are used to calibrate analytical instrumentation are within 10% of the correct concentrations, and therefore standard solution concentrations are 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 includes 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.

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 incorrect 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 incorrect reporting of the absence of targeted 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 quantitation, a representative sample of each matrix type is spiked with the analytes at the estimated level of quantitation and at ten times the estimated level of quantitation. 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 the level of quantitation 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 quantitation. In certain instances, when the analyte being spiked is also present in the sample at concentrations greater than the level of quantitation, 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.

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 D-4210-83 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. It's 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, published by McGraw-Hill Book Company, Inc.

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.

Samples - 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.

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 interfere 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 interfere 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.

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.

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.

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:

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Hazardous Response Support Division
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Washington, D. C. 20460

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 = Relative Response = (Analyte Area)/(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 areas. In addition, if instrument sensitivity changes, these changes are also reflected in changes in the internal standard areas. The calculation of the relative response corrects for these types of fluctuation.

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:

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

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 includes 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:

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 supply 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.

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.