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TITLE: SUMMARY OF IMPROVEMENTS IN INCIDENTAL PCB ANALYSIS APPROACHES

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

ABSTRACT: Significant improvements were made in incidental PCB analyses using capillary GC/MS analysis methods. These improvements do not significantly lower the detection limits for these compounds, but greatly improve the documentation and efficiency of these analyses. An automated method was developed which transfers raw analyte area and characteristic/confirming ion ratio information from the HP-1000 GC/MS data system to a LOTUS 1-2-3 template file on a personal computer. This template file calculates relative response factors, surrogate recoveries, native recoveries, and produces graphs which display ion ratios. A PCB congener mixture was purchased which is routinely used to show the analysis windows for the CL1-biphenyl through CL9-biphenyl isomers. A second capillary column was evaluated for the analysis of complex PCB formulations, and several different cleanup methods were used for low-level PCB analyses.

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APPROVAL DATE: December 29, 1987

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RESEARCH REPORT

DATE: December 18, 1987

PROJECT NO: 760.24-710

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Title: Summary of Improvements in Incidental PCB Analysis Approaches.

ABSTRACT

Significant improvements were made in incidental PCB analyses using capillary GC/MS analysis methods. These improvements do not significantly lower the detection limits for these compounds, but greatly improve the documentation and efficiency of these analyses. An automated method was developed which transfers raw analyte area and characteristic/confirming ion ratio information from the HP-1000 GC/MS data system to a LOTUS 1-2-3 template file on a personal computer. This template file calculates relative response factors, surrogate recoveries, native recoveries, and produces graphs which display ion ratios. A PCB congener mixture was purchased which is routinely used to show the analysis windows for the CL1-biphenyl through CL9-biphenyl isomers. A second capillary column was evaluated for the analysis of complex PCB formulations, and several different cleanup methods were used for low-level PCB analyses.

INTRODUCTION

During 1987, significant improvements were made to ESC's Incidental PCB Analysis method. These improvements were funded through Core Research Funding of Project Number 760.24-710. The present report is a Research Report which briefly summarizes how 0.1 person-years of core funding were expended. Figure 1 summarizes the various projects which were indirectly affected by this support. Although not specifically shown, approximately 20% of this support was devoted to training of new employees who were involved in PCB analyses. It is estimated that the remaining 80% of support was devoted to research studies described in the following sections of this report.

(continued)

Appendix A summarizes the mass spectrometric instrumental analysis method which has evolved over several years of incidental PCB analysis research. In addition, this appendix describes three specialized extraction methods which were developed this year. Appendix B gives a brief description of the Incidental PCB regulation, from the perspective of the analyst who must conduct these studies. The following paragraphs discuss the contents of the remaining appendices in this research report.

MASS SPECTROMETER TUNING AND CHROMATOGRAPHIC DOCUMENTATION

An overall approach was developed to routinely evaluate how the mass spectrometer was electronically tuned, and the chemical condition of the capillary column used to separate the various PCB congeners. A system performance standard is routinely analyzed and a procedure file named SYSPRF is used to automatically evaluate data from analysis of this standard solution. Appendix C gives a listing of this procedure file and an example of output generated from its use. This standard contains 2,6-dimethylphenol, 2,6-dimethylaniline, decanol, pentachlorophenol, anthracene-d10, 1-octadecene, n-octadecane, decafluorotriphenylphosphine (DFTPP), eicosane, heneicosane, pyrene, methyl stearate, and chrysene. These components have widely varying basicities and acidities and DFTPP and pentachlorophenol have mass spectral and chromatographic properties which EPA has defined in detail in their 625 and 1625 priority pollutant methods.

The mass spectrometer tuning is evaluated by comparing the obtained spectrum for DFTPP with EPA's definition of what criteria are acceptable for this compound. The first output shown in Appendix C shows how ESC's DFTPP spectrum compares with EPA's Criterion for this compound.

Chromatographic performance is evaluated by observing the relative heights of the first two components which are 2,6-dimethylphenol and 2,6-dimethylaniline. Significant differences in the heights of these two peaks indicates that there is significant tailing for either or both of these compounds and that corrective measures should be taken. Tailing of decanol, pentachlorophenol, or chrysene are also indications that column activity is high and should be corrected. Finally, the resolution between anthracene-d10, 1-octadecene and n-ocadecane, and between pyrene, heneicosane, and methyl stearate demonstrate the reactivity of the column. Normally, these two triplets should be baseline resolved or corrective action should be taken.

(continued)

MASS SPECTROMETER CALIBRATION AND DATA ANALYSIS

Once the mass spectrometer and capillary column have been shown to be functioning properly, a standard containing PCB congeners from CL1-biphenyl through CL10-biphenyl are injected while scanning the mass spectrometer using the selected ion monitoring mode of scanning. The instrumental analysis method MSAN25, which is summarized in Appendix A, describes the masses and approximate mass windows which are monitored for Incidental PCB analysis. The instrumental scanning method, PCSC25, is also shown in this appendix. One important advancement in the analysis of PCB congeners is shown in Appendix D. Table D-1 shows Internal Standard, and Surrogate and Native Spiking Compounds which are used in the analysis of incidental PCBs. In addition, Appendix D shows a listing of the procedure file named PCCALC which is used to automatically produce high quality graphic outputs which can be used to easily evaluate whether various responses are likely due to PCBs. This output is shown following the listing and a typical ASCII file is also shown which can be transferred to a personal computer LOTUS template file. This LOTUS file was designed to calculate relative response factors, and other important quantitative formation relating to data analysis. Tables 1, 1a, 1b, and 2 are examples of tables which are automatically generated from this LOTUS template file.

Appendix E shows another type of procedure file which is used to analyze for all PCB congeners with a certain number of chlorine atoms. This proceedure file output reports total PCB isomers within a certain window. In order for responses to be summed in this total, retention times of the characteristic and confirming mass chromatographic peaks must be within 10 seconds of one another.

PCB WINDOW STANDARD

The final instrumental improvement in ESC's Incidental PCB Analysis Protocol includes the use of a PCB Window Standard from Wellington Laboratories. Table F-1 in Appendix F shows specific isomers which are present in this standard and Figures F-1a and F-1b show ESC's chromatogram obtained from the analysis of this standard. This appendix also shows the output generated from the proceedure file named PCWNDW. Following this output are documentation from Wellington Laboratories and Figure F-2 which shows the chromatogram obtained by Wellington Laboratories from GC/MS analysis of this PCB Window Standard. The analysis of this standard gives good documentation that the proper retention time windows were scanned so that if any of the 209 PCB congeners had been present in a sample extract above the level of detection, it would have been identified.

(continued)

EVALUATION OF BIPHENYL CAPILLARY COLUMN

Recently capillary columns containing a polysiloxane/biphenyl stationary phase have become available commercially from Lee Scientific (Salt Lake City, UT). ESC has traditionally used J&W (Folsom, CA) DB-5 polysiloxane/phenyl capillary columns for PCB analysis. The use of a biphenyl SB-30 column for PCB analysis was investigated because the stationary phase (biphenyl) and PCBs (polychlorinated biphenyls) have similar polarities, which could possibly impart greater chromatographic selectivity in analyses. This hypothesis was tested by installing a biphenyl capillary column (25 m, SB-30, 0.32 mm diameter, 0.25 um film thickness) into the same Hewlett-Packard 5880 gas chromatograph that is used for PCB analysis equipped with a J&W (30 m, DB-5, 0.32 mm diameter, 0.25 um film thickness) capillary column.

The normal operating conditions for DB-5 column PCB analysis use a 5 psi head pressure, an oven temperature profile of 130(1)/4/300, and splitless injection. The oven temperature profile was changed to 130(1)/5/300 for the SB-30 column PCB analysis because of the longer retention times observed using this column.

Appendix G contains Figures G-1 through G-3 which show chromatograms obtained on the DB-5 and the SB-30 column. Figure G-1 shows four Aroclor formulation chromatograms obtained on a DB-5 capillary column using an electron capture detector (ECD). Figure G-2 shows five Aroclor formulation chromatograms obtained on an SB-30 capillary column. Figure G-3 shows (from bottom to top) Aroclor 1248, a sample which was known to contain chromatographic interferents, Aroclor 1248, and the same sample known to contain interferents. The bottom two chromatograms were obtained on the SB-30 column, and the top two chromatograms were obtained on the DB-5 column. Both columns obviously show that the sample contains Aroclor 1248. However the beginning of the Aroclor formulation is more discernable on the SB-30 column.

Adequate resolution was obtained on both columns, although a comparison of Figures G-1 and G-2 show the formulation patterns are different. These two figures also show that the PCBs have longer retention times on the SB-30 column even though we are using a faster temperature ramp than is normally used in the DB-5 column PCB analysis.

We can conclude that the SB-30 capillary column is usable and in some cases perhaps desirable for PCB analysis. However, no better separation of PCB congeners was obtained on the SB-30 column. Since the majority of PCB analyses performed by the Environmental Sciences Center is done using pattern comparison approaches (identifying the Aroclor which is present and calculating concentrations from that Aroclor formulation), the day to day use of an SB-30 column would be required for adequate comparison of the two columns. At present there is no distinct evidence that the use of one column would offer an advantage over the other column. Analysts have experience with patterns on the DB-5 column and there are no present plans to change columns at this time.

(continued)

SUMMARY OF EXTRACTION METHODS USED

Several projects over the past year required the use of modified extraction and particularly cleanup methods for PCB analysis. ESC has been using a mixed bed impregnated silica gel column over the past several years for the removal of interferents from sample matrices. The column consists of permanganate, base, and acid impregnated silica gels through which the extract is passed. This approach has been used in conjunction with many of the instrumental improvements described earlier in this report. A brief description of this cleanup approach can be seen in Standard Extraction Method PCEX30 and flow sheet PCFS30 which are shown in Appendix A.

One set of chloronitrobenzene samples was analyzed by reduction of the nitro-group to an NH2- group by the direct addition of HCL and mossy tin to the sample matrix prior to the use of our standard silica gel mixed bed cleanup column. This approach was completely succesful and resulted in an accurate analysis with a lower limit of detection than would have been possible without this reduction step. Sample size was approximately 1 g and final extract volume was 1 mL. A brief description of Standard Extraction Method PCEX06 and flow sheet PCFS06 are shown in Appendix A.

An even more useful technique was developed for sample cleanup as a part of Monsanto's Fermentia due diligence effort. Two compounds were analyzed which were very stable and could not be hydrolized except under very harsh conditions such as concentrated sulfuric acid. The tin/HCL approach was attempted but without success. A method was finally developed which involved: 1) the use of dimethyl formamide (DMF) to dissolve the samples; 2) addition of water to the solution; and 3) extraction of the resulting DMF/water solution with hexane. Two percent water was added to decrease the PCB solubility in the DMF/water phase. It was found that without the addition of water to DMF, the distribution coefficient of the lower chlorine class PCBs (monchloro- through tetrachloro-biphenyls) was less than 0.3. Decachorobiphenyl had a distribution coefficient close to 10. (Note: The distribution coefficient is defined as {concentration of analyte in extract}/{concentration of analyte in sample/water/DMF}.) Normally, for efficient extractions, distribution coefficients are on the order of 10,000. These low distribution coefficients were unsatisfactory for efficient extractions. When water was added to the sample/DMF solution, the distribution coefficient was raised to a point where extraction efficiencies were measured between 50 and 80% for all PCB congeners. A brief description of this cleanup approach can be seen in Standard Extraction Method PCEX04 and flow sheet PCFS04 which are shown in Appendix A.

This methodology was unique since no harsh conditions were used to hydrolize the matrix, and the amount of selectivity gained was high enough to allow a 1 g sample to be placed in 1 mL of solvent. Further work should be done in the area of terniary solvent systems in order to exploit solubility differences as a means of isolating PCB congeners.

(continued)

DETAILS

Standard Extraction Method(s): PCEX04 (Also see PCFS04 for method flow sheet.)
PCEX06 (Also see PCFS06 for method flow sheet.)
PCEX30 (Also see PCFS30 for method flow sheet.)

Standard Analysis Method: MSAN25

Mass Spectrometer Data Acquisition Method: PCSC25

Mass Spectometric Tuning File: MT3602

Final Report File No(s): RA7710

Table File No(s): TA7710, TB7710, FA7710, FB7710, RB7710

APPENDICES

Appendix A. Summary of Extraction and Analysis Methods Developed.

Appendix B. Brief Description of Analysis Approaches Used in the Analysis of Incidental PCBs.

Appendix C. Listing of Procedure Files and Examples of Computer outputs Produced from the Analysis of the System Performance Standard. (SYSPRF Transfer File.)

Appendix D. Listing of PCB Congeners Used for Instrument Calibration and Procedure Files and Examples of Computer Outputs Produced in Incidental PCB Analyses. (PCCALC and PCOUTP Transfer Files.)

Appendix E. Listing of Procedure Files and Examples of Computer Outputs Produced in Incidental PCB Analyses. (PTCALC and PTOUTP Files.)

Appendix F. Listing of PCB Congers Present in PCB Window Standard and Procedure Files and Examples of Computer Outputs Produced in Incidental PCB Analyses. (PTWNDW and PTOUTP Transfer Files.)

Appendix G. Chromatograms Obtained from the Comparison of DB-5 and SB-30 Capillary Columns.

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

ANALYST APPROVAL: _____

Figure 1 – PCB Activities (Adjunct)

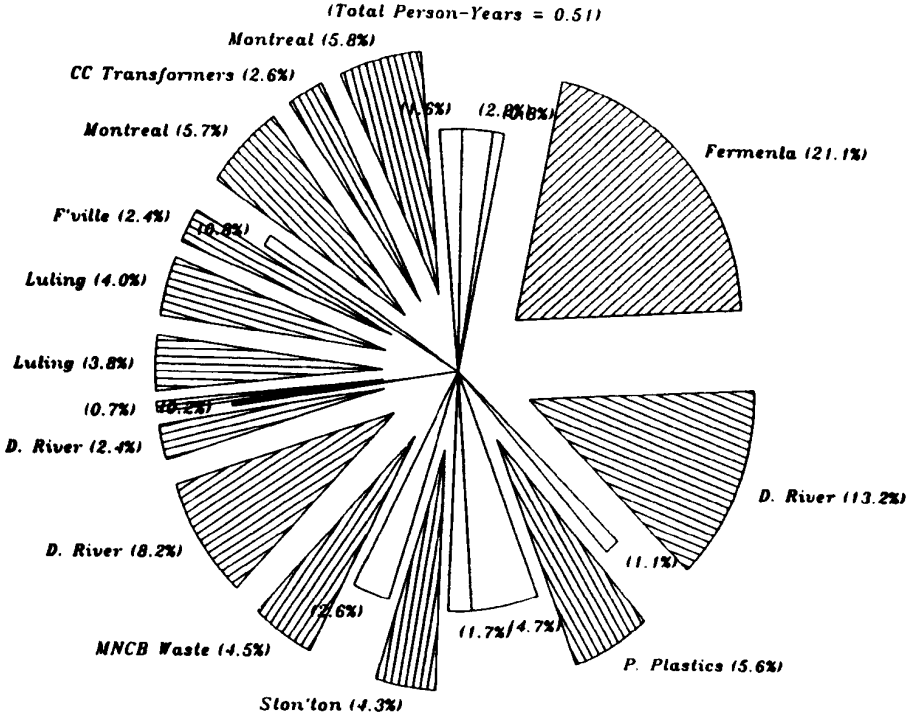

Table 1. Example of Incidental PCB Concentration Results Automatically Generated from LOTUS 1-2-3.

Table 1. Results of Enrmarich Incidental PCB Study in Chloronitrobenzenes Using Standard Extraction and Analysis Methods PCEM06 and MBAN25. (Project: 7251)

FRMS USED FOR CALIBRATION: >L4302 >L4305 >L4309

SAMPLE ID. ··· / EXTRACT NUMBER ··· / DESCRIPTION	Meta-CNB PCB-2132 (ug/mL)	Meta-CNB DUP PCB-2133 (ug/mL)	Crude-CNB PCB-2136 (ug/mL)	Crude-CNB DUP PCB-2137 (ug/mL)	Meth Blank PCB-2140 (ug/mL)	Meta-CNB+MS PCB-2135 (ug/mL)	Crude-CNB+MS PCB-2139 (ug/mL)	Meta-CNB+LS PCB-2134 (ug/mL)	Crude-CNB+LS PCB-2138 (ug/mL)	MS SPK MS SP DUP (ug/mL)
Monochlorobiphenyls	0	0	0	0	0	9	7.7	2.5	2.3	8.9
Dichlorobiphenyls	0.5	0.4	1	1	0	11.5	11.2	3.2	3.7	11.2
Trichlorobiphenyls	0.1	0	0	0	0	8.1	8	2.1	2.4	8.2
Tetrachlorobiphenyls	0	0	0	0	0	7.5	7.1	1.9	2.7	7.9
Pentachlorobiphenyls	0	0	0	0	0	8.3	7.8	1.7	2.4	8.2
Hexachlorobiphenyls	0	0	0	0	0	7.0	6.7	1.9	2.8	7
Heptachlorobiphenyls	0	0	0	0	0	5.4	5.2	1.1	1.8	5.6
Octachlorobiphenyls	0	0	0	0	0	2.4	1.9	0.7	1.1	1.8
Nonachlorobiphenyls	0	0	0	0	0	2.4	2.1	0.6	1.2	1.8
Decachlorobiphenyl	0	0	0	0	0	1.5	1.1	0.9	1.5	0.8
TOTAL PCBS IN EXTRACT(ug/mL)	0.6	0.4	1	1	0	63.7	58.8	16.6	21.9	61.2
TOTAL PCBS IN SAMPLE (ug/g)	0.4	0.3	0.8	0.7	0	51.7	57.2	13.8	22.1	60.2
SURROGATE COMPOUND	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)
Monochlorobiphenyl-DC13	100	92	85	78	40	100	95	83	78	90
Tetrachlorobiphenyl-12C13	92	58	110	87	113	88	65	80	105	71
Octachlorobiphenyl-12C13	90	40	140	102	148	43	33	65	100	33
Decachlorobiphenyl-12C13	276	57	230	200	295	20	10	100	133	16
MS RUN FILE ·	: >L4297	: >L4298	: >L4299	: >L4300	: >L4301	: >L4306	: >L4307	: >L4308	: >L4304	: >L4305
DILUTION FACTOR ·	0.600	0.831	0.771	0.675	1	0.812	0.973	0.832	1.01	1
SAMPLE AMOUNT (g) ·	1.433	1.204	1.297	1.485	1	1.232	1.028	1.202	0.99	1
EXTRACT FINAL VOLUME (mL) ·	1	1	1	1	1	1	1	1	1	1
AUTOMASSIGNS 0100 (1600) AREA ·	266075	1044699	954998	1223708	1349165	961563	1142561	1207997	1209922	1254388

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

Table Generation Commentor: ALL FILES REST OK

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Table 1a. Example of Incidental PCB Relative Response Factor Results Automatically Generated from LOTUS 1-2-3.

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	0.33	0.363333	0.42	0.371111	0.371111
Cl1 biphenyl #3	0.386	0.436666	0.51	0.443888	0.515222
Cl1 biphenyl #4	0.642	0.726666	0.823333	0.730666	
Cl4Biphenyl 6C1305	0.966	1.14	1.36	1.148	1.148
Cl2Biphenyl #6	0.266	0.291666	0.37	0.309222	
Cl2Biphenyl #7	0.517	0.563333	0.696666	0.592333	0.504305
Cl2Biphenyl #8	0.413	0.436666	0.576666	0.475444	
Cl2Biphenyl #9	0.554	0.59	0.776666	0.640222	
Cl3Biphenyl #10	0.224	0.232	0.302666	0.252888	
Cl3Biphenyl #11	0.248	0.246	0.323666	0.272555	0.260111
Cl3Biphenyl #12	0.225	0.231	0.308666	0.254888	
Cl4Biphenyl #13	0.141	0.128	0.173333	0.147444	
Cl4Biphenyl #14	0.116	0.108666	0.148333	0.124333	0.141074
Cl4Biphenyl #15	0.142	0.133333	0.179	0.151444	
Cl4Biph 12C13016	0.527	0.329	0.533	0.463	0.463
Cl5Biphenyl #17	0.0751	0.077	0.102333	0.084811	
Cl5Biphenyl #18	0.0616	0.069466	0.093	0.074755	0.078559
Cl5Biphenyl #19	0.064	0.067	0.097333	0.076111	
Cl6Biphenyl #20	0.0517	0.043333	0.072333	0.055788	
Cl6Biphenyl #21	0.0398	0.035333	0.059666	0.044933	0.0455
Cl6Biphenyl #22	0.0322	0.0278	0.047333	0.035777	
Cl7Biphenyl #23	0.0461	0.048666	0.066666	0.053811	0.054611
Cl7Biphenyl #24	0.0469	0.049666	0.069666	0.055411	
Cl8Biphenyl #25	0.0299	0.014433	0.039333	0.024955	0.024955
Cl8Biph 12C13026	0.243	0.0703	0.327	0.213433	0.213433
Cl9Biphenyl #27	0.0232	0.016166	0.041333	0.0269	0.0269
Cl10Biphenyl #28	0.0161	0.003766	0.022733	0.0142	0.0142
Cl10Biph 12C13429	0.0822	0.00936	0.0875	0.059686	0.059686

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Table 1b. Example of Incidental PCB Characteristic Ion/Confirming Ion Ratio Results Automatically Generated from LOTUS 1-2-3.

TABLE OF CHARACTERISTIC ION RESPONSES TO CONFIRMING IONS 100 FOR SAMPLES 1-10 FOR SAMPLES IN CONCENTRATION TABLE:
Table 1. Results of Grumman Lab Incidental PCB Study in Chloronitrobenzenes Using
Standard Extraction and Analysis Methods PCE#06 and #SA#25. (Project: 7251)

	STANDARD USED IN CALCULATIONS			FILE NAMES										MINIMUM RATIO	MAXIMUM RATIO	AVERAGE RATIO
	S1	S2	S3	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10			
COMPOUND ID	>L4302	>L4305	>L4309	>L4297	>L4298	>L4299	>L4300	>L4301	>L4306	>L4307	>L4303	>L4304	>L4305			
001 = anthracene-d10	8720	8669	8520	9172	8821	8816	8744	8743	8699	8396	8452	8700	8669	8520	8720	8630
002 = Cl-biphenyl #1	290	294	288	323	274	238	305	160	285	295	284	286	294	288	294	291
003 = " #2	288	296	291	190	58	154	225	217	292	297	294	289	296	288	296	292
004 = " #3	292	294	285	97	241	264	261	213	305	287	292	281	294	285	294	290
005 = 4-Cl-biphenyl-DC13	291	298	306	300	295	301	289	297	295	299	297	299	298	291	306	298
006 = Cl2-biphenyl #1	160	160	159	49	224	178	158	100	164	159	161	163	160	159	160	160
007 = " #2	159	162	157	164	166	162	165	0	160	161	162	159	162	157	162	159
008 = " #3	162	160	157	162	164	159	162	0	161	166	160	160	160	157	162	160
009 = " #4	160	162	157	152	164	156	160	0	160	161	162	161	162	157	162	160
010 = Cl3-biphenyl #1	88	82	86	85	90	97	87	259	88	80	86	88	82	82	88	85
011 = " #2	91	82	85	80	78	81	107	78	82	80	87	86	82	82	91	86
012 = " #3	86	83	83	116	83	96	96	429	82	80	86	87	80	83	86	86
013 = Cl4-biphenyl #1	93	96	85	220	242	743	129	126	97	96	92	90	96	85	96	91
014 = " #2	90	97	86	105	417	462	132	200	96	98	96	90	97	86	97	94
015 = " #3	90	96	86	150	121	1450	276	294	97	100	92	91	96	80	96	90
016 = Cl4-DP-13C15 Surr.	92	117	91	97	96	93	97	96	112	129	96	96	117	91	117	100
017 = Cl5-biphenyl #1	60	59	59	120	200	575	104	229	60	60	60	58	59	59	60	59
018 = " #2	58	61	61	105	109	161	206	588	59	61	60	60	61	58	61	60
019 = " #3	60	60	62	2100	293	103	543	350	60	59	60	62	60	60	62	61
020 = Cl6-biphenyl #1	57	64	58	95	167	229	667	316	65	64	57	57	64	57	64	60
021 = " #2	56	62	56	233	250	240	310	1575	63	65	58	57	62	56	62	59
022 = " #3	56	64	55	293	132	371	240	257	62	64	58	57	64	55	64	59
023 = Cl7-biphenyl #1	147	150	151	200	371	73	285	55	149	153	148	149	150	147	151	149
024 = " #2	150	156	153	444	415	106	123	176	150	148	148	148	156	150	156	153
025 = Cl8-biphenyl	80	71	84	205	322	208	114	149	72	70	80	85	71	71	84	78
026 = Cl8-DP-13C13 Surr.	81	56	83	76	77	82	80	80	65	56	75	77	56	56	83	73
027 = Cl9-biphenyl	93	106	86	247	483	108	210	750	108	105	100	97	106	86	106	99
028 = Cl10-biphenyl	142	255	133	231	167	109	121	118	279	208	147	146	255	133	255	177
029 = Cl10-DP-13C13 Surr	163	133	152	166	157	156	155	154	137	132	173	343	133	133	163	149

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Table 2. Example of Incidental PCB Native Spiking Recovery Results Automatically Generated from LOTUS 1-2-3.

Table 2. Native Spiking Recoveries in Krummrich Chloronitrobenzene Samples
Using Standard Extraction and Analysis Methods PCE806 and MSAN25. (Project 7231)

MS CALIB FILE: >L4385
LS CALIB FILE: >L4382

COMPOUND ID	Mate-CNB+MS PCB-2135 (% REC)	Crude-CNB+MS PCB-2139 (% REC)	MS SPK MS SP STD (% REC)	Mate-CNB+LS PCB-2134 (% REC)	Crude-CNB+LS PCB-2138 (% REC)	LS SPK LS SP STD (% REC)
002 = Cl-biphenyl #1	105	89	100	95	89	100
003 = " #2	102	88	100	93	87	100
004 = " #3	100	86	100	95	88	100
006 = Cl2-biphenyl #1	104	92	100	90	95	100
007 = " #2	98	92	100	85	89	100
008 = " #3	107	117	100	103	140	100
009 = " #4	103	101	100	96	99	100
010 = Cl3-biphenyl #1	96	97	100	79	89	100
011 = " #2	102	100	100	80	85	100
012 = " #3	99	98	100	81	93	100
013 = Cl4-biphenyl #1	95	93	100	67	93	100
014 = " #2	88	89	100	70	93	100
015 = " #3	94	90	100	69	95	100
017 = Cl5-biphenyl #1	105	97	100	65	98	100
018 = " #2	100	94	100	69	96	100
019 = " #3	102	96	100	70	98	100
020 = Cl6-biphenyl #1	105	92	100	58	93	100
021 = " #2	111	97	100	75	107	100
022 = " #3	121	98	100	80	112	100
023 = Cl7-biphenyl #1	100	99	100	62	106	100
024 = " #2	101	94	100	66	110	100
025 = Cl8-biphenyl	135	110	100	83	135	100
027 = Cl9-biphenyl	135	116	100	71	134	100
028 = Cl10-biphenyl	196	142	100	75	132	100
SURROGATE COMPOUNDS						
005 = 4-Cl-biphenyl-dC13	101	94	100	99	93	100
016 = Cl4-BP-12C13 Surr.	124	91	100	70	92	100
026 = Cl8-BP-12C13 Surr.	132	100	100	57	103	100
029 = Cl10-BP-12C13 Surr	120	120	100	76	97	100
MS RAW FILE =	: >L4386	: >L4387	: >L4385	: >L4383	: >L4384	: >L4382

LS = the addition of 0.1 ug per PCB congener.

MS = the addition of 1 ug per PCB congener.

Note - Recoveries are rounded to the nearest whole number

MONS 024764

Appendix A. Summary of Extraction and Analysis Methods Developed.

MONS 024765

ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No:
TITLE: Polychlorinated Compounds in Solvents — MSAN25

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-6C13 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-12C13 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-12C13 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-12C13 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 - <871123.1532>

(continued)

Supplemental Information to Standard Analysis Method MSAN35

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
2,5-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

Enter the name of the method file: pcsc25

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

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Water Soluble Matrices

ESC Standard Extraction Method No: PCEX04

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Nylon, SEF fibers, water soluble matrices etc.	0.5 - 5.0	1 - 10

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers for ECAN01 and ECAN02. Any species in the appropriate retention time region which give the same relative characteristic mass responses as PCB isomers for MSAN03.

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

APPARATUS AND MATERIALS:
Burdick and Jackson hexane for sample extraction.
Water soluble solvents such as dimethyl formamide, water, basic water, methanol, ethanol, acetic acid or others.
Sodium sulfate to dry the sample matrix.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Disposable glassware.
Nitrogen for extract volume reduction.
(See flow sheet for this extraction method in PCFS04.)

LIMITATIONS: Method applies only to matrices which contain hydrolizable electron capturing species in the trichlorobiphenyl through octachlorobiphenyl isomer retention time region for EC detection methods.

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

DATES OF METHOD CREATION AND UPDATES: 10/25/83, 9/25/84

FLOW SHEET PCFS04

FOR ESC EXTRACTION METHOD PCEX04

SAMPLE ID: ---------- MRG EXTRACT # ----------

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

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

WEIGH OUT SAMPLE INTO A 40 mL VIAL [ ]

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

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

SAMPLE WEIGHT ----------

SPIKE 100 uL ---------- [ ]

---------- ----------

ADD 15 mL WATER SOLUBLE SOLVENT (FORMIC ACID FOR NYLON, VYDYNE ECT, DIMETHYL FORMAMIDE FOR SEF FIBERS) [ ]

[ ] RINSE COLUMN WITH 2 X 20 mL HEXANE

EXTRACT 3 X 3 mL WITH HEXANE [ ] [ ] [ ]

APPLY EXTRACT TO COLUMN QUANTITATIVELY [ ]

ELUTE SAMPLE INTO 50 mL CENTRIFUGE TUBE WITH 40 mL HEXANE. [ ]

NITROGEN BLOW DOWN TO 1 mL [ ]

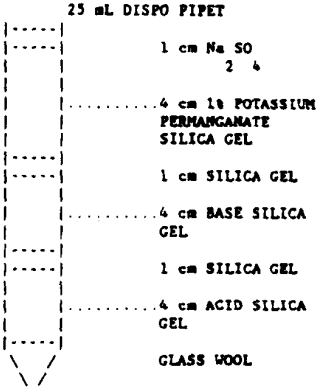
EXTRACT NO: ----------

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

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

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

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET



MONS 024772

ESC Standard Extraction Method (SEM)
TITLE: PCBs in nitro group containing matrices

ESC Standard Extraction Method No: PCEX06

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
p-nitrophenetol, aromatic/ alkyl nitro containing compounds.	0.5 - 5	1 - 10

TYPICAL ANALYTES:A1232,A1242,A1248,A1254,A1260 and A1262 and PCB isomers mono- through deca-chlorobiphenyl.

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers for ECAN01 and ECAN02. Any species in the appropriate retention time region which give the same relative characteristic mass responses as PCB isomers for MSAN03.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
pentachlorobenzene	50 - 120
2,4,6-trichloro-biphenyl	50 - 120
3,3',4,4'-CL4-biphenyl-d6	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson hexane for sample extraction
Concentrated hydrochloric acid for reduction of the nitro group.
Mossy tin for catalytic reduction of the nitro group.
Concentrated sulfuric acid for 50% extract rinse.
Distilled deionized water for dilution of hydrochloric and sulfuric acids.
Sodium hydroxide pellets for freeing the amine salt.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Disposable glassware.
Nitrogen for extract volume reduction.
(See flow sheet for this extraction method in PCFS06.)

LIMITATIONS: Method applies only to matrices which contain hydrolizable electron capturing species in the trichlorobiphenyl through octa-chlorobiphenyl isomer retention time region for EC detection methods.

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

DATES OF METHOD CREATION AND UPDATES: 4/24/84

FLOW SHEET PCFS06

FOR ESC EXTRACTION METHOD PCEX06

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

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

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

WEIGH OUT SAMPLE INTO A 40 mL VIAL [ ] WEIGHT SAMPLE ----------

SURROGATE SPIKE OF SAMPLE [ ] SPIKE ----------

ADD 1.5 g MOSSY TIN, AND 6 mL CONC. HCl TO SAMPLE [ ] ALLOW TO STAND OVERNIGHT

ADD 15 mL WATER TO SAMPLE [ ] AND BASIFY WITH NaOH (4-5g)

EXTRACT 3 X 5 mL HEXANE [ ] [ ] [ ]

RINSE EXTRACT 1 X 10 mL 50% SULFURIC ACID [ ]

REDUCE SAMPLE VOLUME TO 1-2 mL WITH NITROGEN AND APPLY TO COLUMN QUANTITATIVLY.

ELUTE WITH 30 mL HEXANE [ ]

NITROGEN BLOW DOWN TO 1 mL [ ]

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

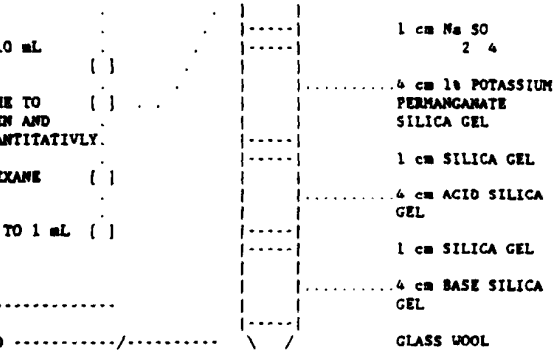
DATE/TIME COMPLETED ----------/----------

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

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

RINSE COLUMN WITH 2 X 20 mL
[ ] HEXANE

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET



MONS 024774

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Oil Matrices Requiring Cleanup

ESC Standard Extraction Method No: PCEX30

MATRI(X),(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Oils, that contain acid or base hydrolizable impurities	0.5 - 5	1 - 10

TYPICAL ANALYTES:A1232,A1242,A1248,A1254,A1260 and A1262 and PCB isomers mono- through deca-chlorobiphenyl.

INTERFERENCES:	TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl isomers for ECAN05		
Any species in the appropriate rentention time region which give the same relative characteristic mass responsee as PCB isomers for MSAN25.	pentechlorobenzene	50 - 120
	2,4,6-trichloro-biphenyl	50 - 120
	3,3',4,4'-tetrechloro-biphenyl-d6	50 - 120

APPARATUS AND MATERIALS:
Burdick and Jackson benzene for sample extraction.
Burdick and Jackson hexane for column elution.
Silica gel columns impregnated with potassium permangenate, sulfuric acid, and potassium hydroxide.
Disposable glassware.
(See flow sheet for this extraction method in PCFS07.)

LIMITATIONS: Method applies only to matrices contain hydrolizable impurities that the cleanup column can remove. EC SAMs are valid for CL3-BP through CL10BP. MS SAMs are valid for all PCBs from CL1- through CL10-BP.

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

DATES OF METHOD CREATION AND UPDATES: 10/19/87

FLOW SHEET FCFS30

FOR ESC EXTRACTION METHOD FCEX30

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

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

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

PIPET 1 mL

WEIGH OUT SAMPLE INTO A 14 mL VIAL [ ] WEIGHT SAMPLE (1 g) ----------

SURROGATE SPIKE OF SAMPLE [ ] SPIKE ----------

---------- [ ] RINSE COLUMN WITH 2 X 20 mL HEXANE

ADD 10 mL BENZENE TO VIAL [ ]

SHAKE UNTIL OIL IS INTO SOLUTION [ ]

PLACE 1 mL EXTRACT ONTO HEAD OF COLUMN [ ]

ELUTE COLUME WITH 40 mL HEXANE [ ]

COLECT COLUMN ELUENT IN A 50 mL CENTRIFUGE TUBE [ ]

NITROGEN BLOW DOWN TO 1 mL [ ]

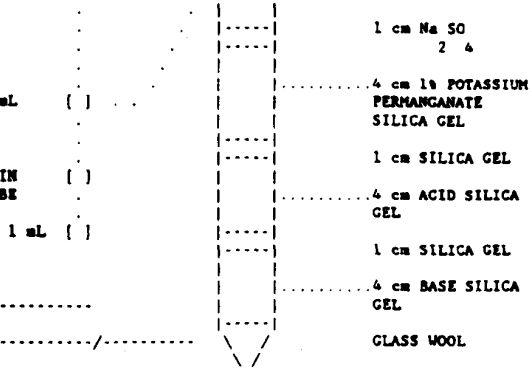
EXTRACT NO: ----------

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

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

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

CLEAN-UP COLUMN DESCRIPTION
25 mL DISPO PIPET



MONS 024776

Appendix B. Brief Description of Analysis Approaches Used in the Analysis of Incidental PCBs.

MONS 024777

Analysis of Incidental PCBs in Monsanto Products, Waste Streams, Emissions and Effluents of Closed and Controlled Waste Manufacturing Processes

B. Mason Hughes and David E. McKenzie
Environmental Sciences Center
Monsanto Company
800 N. Lindbergh Blvd.
St. Louis, Mo. 63167

The October 21, 1982 Federal Register (Vol. 47, No. 204) contains a very extensive definition of PCB-containing products, waste streams, emissions and effluents from manufacturing processes and general descriptions of methods which should be used for these analyses. The purpose of this present report is to outline the approach taken by Monsanto's Environmental Sciences Center in supplying analytical data which can be used for certification of Monsanto products, waste streams, emissions and effluents.

In order for the material to be certified as not containing PCBs, the EPA set various Levels of Quantitation (LOQ), below which a product, waste stream, emission or effluent could be certified to contain no PCBs. The LOQ is defined on the basis of the concentration per resolvable chromatographic peak. Quoting from page 46986 of the Federal Register, Vol. 47, No. 204, "This means that for a process to be eligible for exclusion under the closed and controlled waste process exclusion, no single peak on a gas chromatogram registers PCBs in excess of: ten micrograms per cubic meter in air emissions, 100 micrograms per liter in water effluents, and two micrograms per gram in products and uncontrolled waste streams." Therefore there are very specific definitions which Monsanto can apply to its products, waste streams, emissions and effluents which determine whether these materials may be eligible for exclusion. It is the goal of the Environmental Sciences Center to supply Monsanto plants with the proper data and documentation for this exclusion.

Capillary gas chromatography/electron impact mass spectometry (CGC/MS) is the instrumental technique which the Federal Register suggests should be used in the analytical method for PCB congener analysis. However for most Monsanto products, waste streams, air emissions and water effluents, this instrumental method must be used in conjunction with an extraction method in order to determine what levels of PCB congeners, if any, are present in these various materials. The remainder of this report will therefore deal with how ESC implements this instrumental method for the analysis of PCBs in various types of samples.

(continued)

In order to analyze a sample for PCBs, using CGC/MS techniques, a representative sample of interest, or a representative extract of a sample of interest must be injected into the capillary chromatographic system. The presence and quantities of PCB congeners are determined by comparing the PCB congener responses in the sample of interest with PCB congener responses in an analytical standard containing at least one PCB congener for each of the ten chlorine classes (i. e. CL1-biphenyl through CL10-biphenyl). The analysis of Monsanto products and waste streams can be divided into two types of CGC/MS analyses. The first is for products and waste streams which contain major components which can be chromatographically resolved from CL1-biphanyl through CL10-biphenyl congeners, when the product or waste stream components are present at concentrations ten million times those of the PCB congenars. This first type of analysis only requires the sample to be injected into the CGC/MS instrument and PCB congenar areas be compared to standard PCB congener areas.

The second type of analysis applies to Monsanto products and waste streams which contain major components which cannot be chromatographically resolved from PCB congeners and for all air emission and water effluent samples. This second type of analysis involves first isolation of all PCB congeners which are present in a representative sample into a suitable solvent. This extracting solvent can then be analyzed for PCB congeners as above in the first type of analysis, since the extracting solvent is chosen to be chromatographically resolved from the PCB congeners of interest. An integral part of the PCB congener analysis revolves around the validation of the extraction method used to isolate the PCB congeners and validation of the CGC/MS method used to analyze extracts. In order for Monsanto plants to understand the data that is generated for incidental PCB certification, the following sections outline how data are obtained and validated for the presence or absence of PCB congeners in various Monsanto materials.

Extraction of products, waste streams, air emissions and water effluents:

If samples of interest cannot be directly analyzed for PCBs using CGC/MS techniques, they must first be extracted with an appropriate solvent which can be analyzed for PCBs. This not only applies to the products or waste streams, but also to air samples or water samples which may contain large amounts of products and waste stream components. Two major property differences between PCBs and major compounds in various samples can be used to isolate PCB congeners into an appropriate extraction solvent. These are: 1) reactivity; and 2) water solubility. Low levels of quantitation for PCB congeners in samples which contain major components in the PCB congener elution region cannot easily be obtained unless these major components can be removed by their reactivity or water solubility. The inherent stability of PCB congeners and their high affinity for organic solvents and low water solubility are chemical and physical properties which can be exploited for this isolation. However it is important to demonstrate that, during this isolation phase, PCB congeners have not been removed during the extraction. This is done in two ways. The first way involves the use of surrogate compounds which act exactly like PCB congeners, yet are mass spectrometrically different. This is normally accomplished by using congeners which contain carbon-13 rather than carbon-12 in the biphenyl structure. These surrogate spiking compounds are added to each sample for which PCB congener extraction is required, and their recovery is reported along with any PCB congeners detected. However extraction of all PCB congener classes can be demonstrated

(continued)

only if one has isotopically labelled PCB congeners for each congener class. Since at this time there are no good sources of high purity surrogates for all classes of PCB congeners (i. e. CL1-biphenyl through CL10-biphenyl), a second type of extraction validation is normally used. This validation step involves adding at least one native PCB congener for CL1-biphenyl through CL10-biphenyl congeners and measuring the recoveries of each of these native congeners when the product, waste stream, air emission or water effluent is extracted. These native spiking studies should be performed on representative types of matrices to demonstrate the validity of the results. If no previous data is available for recovery of all PCB congener types from a specific matrix, this native spike should be performed in order to validate the data being reported.

Since it is not anticipated that incidental PCB generation will be detected for Monsanto processes, it is imperative that the above quality control steps be conducted on each sample or for each sample matrix type in order to validate the fact that if PCB congeners had been present in the Monsanto samples, they would have been detected at the levels required by this regulation.

<u>GGC/MS analysis of extracts, products and waste streams:</u>

As mentioned above, sample extracts, products and waste streams can be injected directly into the GGC/MS analytical system if the major components do not elute in the retention time regions of the CL1-biphenyl through CL10-biphenyl congeners. Data quality of this analysis step is also demonstrated by similar approaches outlined above for sample extraction. In order to verify that the instrument is functioning properly for each sample injected, an internal standard is added to each extract, product or waste stream before it is injected into the CGC/MS system. Anthracene-d10 is usually used for this purpose. The absolute areas of the internal standard in the extract, product or waste stream analysis must be within 30% of the area of the internal standard which is added to the PCB congener analytical standard.

The analytical standard, containing CL1-biphenyl through CL10-biphenyl congeners, is analyzed under the same conditions as the extract, product or waste stream samples and from the instrument responses for the ten PCB congener classes, levels of detection and quantitation are determined and the amounts of each congener class (if greater than the LOQ) is reported. Ideally, each sample should also be analyzed after spiking with at least one native PCB congener in each congener class at the level of quantitation (LOQ) in order to verify that the LOQ concentration could be detected and quantified in the presence of sample interferences. This is done in order to show that the CGC/MS instrumental analysis is valid for the sample matrix of interest. However some products and waste stream samples are not miscible with solvents in which PCB congener classes have been prepared and therefore it is not always possible to analyze a native spike of all products and waste stream samples. This is not a problem with sample extracts, since the extracting solvent has been chosen to be compatible with surrogate and native spiking studies which occur in the sample extraction step, described above.

(continued)

Quantitation and Identification of PCB Congeners:

Tables 1 and 2 show how the 209 PCB congeners are distributed amoung the CL1-biphenyl through CL10-biphenyl classes. In addition these tables show the characteristic masses and theoretical relative abundances for each of the ten congener classes. In order to routinely obtain analytical data with levels of detection on the order of 0.1 ug/ml per congener, the mass spectrometer detector is operated in the Selected Ion Monitoring mode (SIM). This mode is a screening mode and monitors at least two molecular ion characteristic masses over the elution time region of the corresponding PCB congener class for all ten congener classes. To insure that the proper retention time region is being monitored for the proper chlorine class, a PCB window standard is also analyzed under the same conditions as the samples are analyzed. This window standard contains the first and last eluting PCB congener in each of the chlorine classes. Analysis of this standard documents that all possible congeners of each chlorine class can be detected using the mass spectrometer scan parameters chosen for the analysis. Therefore the analysis conditions can detect all of the 209 PCB congeners.

PCB congeners are qualitatively identified from the ratio of the peak areas of the two molecular ion chromatograms. If the ratio of characteristic and confirming ion chromatographic peaks are within 30% of the experimental ratio obtained from the corresponding chlorine class in the PCB congener standard analysis, and the two peaks maximize within two mass spectrometer scans, then the concentrations of PCB congeners are calculated from these ion areas and appropriate dilution factors. If the peak area ratio does not agree within 30% of the experimental ratio for a particular PCB congener class, then this response is assumed not to be due to a PCB congener.

Summary:

The above appoach allows for rapid, routine analyses of extracts and samples of interest. Since few positives are expected, adequate quality control (involving the use of internal standards, native spikes and surrogate spikes) must be used to validate that the data can be used for the certification of the absence of incidental PCBs in Monsanto products, waste streams, air emissions and water effluents of closed and controlled wasts manufacturing processes.

Text File: INPC01:MH:SP
Table Files: INTB01:MH, INTB02:MH

Table 1. Characteristic masses and isotope patterns of the characteristic masses for chlorobiphenyl through pentachlorobiphenyl congeners.

		PCB Congener Class (Number of Congeners)				
Characteristic Masses	Number of Chlorines	CL1-BP (3)	CL2-BP (12)	CL3-BP (24)	CL4-BP (42)	CL5-BP (46)
152	0	x(-)	x(-)			
153	0	x(-)				
186	1	-		x(100)		
187	1		x(100)			
188	1	x(100)		x(32.4)		
189	1		x(32.4)			
190	1	x(32.4)		-		
220	2		-		x(100)	
222	2		x(100)		x(64.8)	
224	2		x(64.8)		x(10.5)	
226	2		x(10.5)		-	
253.9	3			-		x(100)
255.9	3			x(100)		x(97.2)
257.9	3			x(97.2)		x(31.5)
259.9	3			x(31.5)		-
287.8	4				-	
289.8	4				x(77.2)	
291.8	4				x(100)	
293.8	4				x(48.6)	
295.8	4				x(10.5)	
321.8	5					-
323.8	5					x(61.7)
325.8	5					x(100)
327.8	5					x(64.8)
329.8	5					x(21)

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Table 2. Characteristic masses and isotope patterns of the characteristic masses for hexachlorobiphenyl through decachlorobiphenyl congeners.

		PCB Congener Class (Number of Congeners)				
Characteristic Masses	Number of Chlorines	CL6-BP (42)	CL7-BP (24)	CL8-BP (12)	CL9-BP (3)	CL10-BP (1)
287.8	4	x(77.2)				
289.8	4	x(100)				
291.8	4	x(48.6)				
293.8	4	x(10.5)				
321.8	5		x(61.7)			
323.8	5		x(100)			
325.8	5		x(64.8)			
327.8	5		x(21)			
329.8	5		-			
355.8	6	-		x(51.4)		
357.8	6	x(51.4)		x(100)		
359.8	6	x(100)		x(81)		
361.8	6	x(81)		x(35)		
363.8	6	x(35)		-		
389.7	7		-		x(44.1)	
391.7	7		x(44.1)		x(100)	
393.7	7		x(100)		x(97.2)	
395.7	7		x(97.2)		(17)	
397.7	7		x(17)		-	
423.7	8			-		x(34)
425.7	8			x(34)		x(88.2)
427.7	8			x(88.2)		x(100)
429.7	8			x(100)		x(64.8)
431.7	8			x(64.8)		x(26.2)
433.7	8			x(26.2)		-
459.6	9				x(26.5)	
461.6	9				x(77.2)	
463.6	9				x(100)	
465.6	9				x(75.6)	
467.6	9				x(36.7)	
493.6	10					x(21.2)
495.6	10					x(68.6)
497.6	10					x(100)
499.6	10					x(86.4)
501.6	10					x(19)

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Appendix C. Listing of Procedure Files and Examples of Computer outputs Produced from the Analysis of the System Performance Standard. (SYSPRF Transfer File.)

MONS 024784

```
:du,sysprf,1
:**    :TR,SYSPRF,filename1
:**
:**   This transfer file is designed to calculate RRFs for compounds in the
:**         system performance standard and to give various displays which can
:**         be used to judge system performance.
:**
:**       1G - filename1
:**       2G -
:**       3G -
:**       4G -
:**
:**
:**   The following R-globals are used.
:**       1R - anthracene-d10 m/e 188 area
:**       2R - 2,6-dimethylphenol m/e 122 area       22R - RR for 2,6-dimephnol
:**       3R - 2,6-dimethylaniline m/e 121 area      23R - RR for 2,6-dimeanln
:**       4R - 1-decanol m/e 69 area                 24R - RR for 1-decenol
:**       5R - pentachlorophenol 266 area            25R - RR for pntchlorophnl
:**       6R - 1-octadecene m/e 97 area              26R - RR for 1-octdcene
:**       7R - n-octadecane m/e 85 area              27R - RR for n-octadecane
:**       8R - DFTPP m/e 198 area                    28R - RR for DFTPP
:**       9R - n-eicosane m/e 85 area                29R - RR for n-eicosane
:**      10R - pyrene m/e 202 area                   30R - RR for pyrene
:**      11R - n-heneicosane m/e 85 area             31R - RR for n-henelcosane
:**      12R - methyl stearate m/e 74  area          32R - RR for methyl strate
:**      13R - chrysene m/e 228 area                 33R - RR for chrysene
:**
:SV,1,9,IH
:RU,RPW
:RU,NEW
:RU,CRT,CA,FF
:RU,TIM
:RU,STW,,1,0:75,0:40
:RU,STW,,2,0:75,60:80
:RU,STW,,3,0:75,80:100
:RU,STW,,10,15:090,23:73
:RU,STW,,4,0:75,40:50
:RU,STW,,5,0:75,50:60
:RU,STW,,6,0:75,40:60
:**RU,DEV,2648,1
:**  This section calculates DFTPP m/e 198 area.
:RU,FI,1G
:RU,MCA,198,,,,22:24
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,8,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXG,,,3.5
:RU,RDP,3,DFTPP Spectrum Criterion Test
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 198.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:RU,RD
```

MONS 024785

```
:RU,CRT,CA
:RU,TUNER,=DFTPP
:RU,CRT,AL,FF
:RU,NEW
:**  This section calculates anthracene-d10 area.
:RU,FI,1G
:RU,MCA,188,,,,21:23
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,1,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of Anthracene-d10
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 188.
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,DR,10,X
:RU,GR,,1
:RU,NEW
:** The following calculates 2,6-dimethylphenol m/e 122 area.
:RU,MCA,122,,,,9:11
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,2,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of 2,6-Dimethylphenol
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 122.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates 2,6-dimethylaniline m/e 121 area.
:RU,MCA,121,,,,10:12
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,3,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of 2,6-Dimethylaniline
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 121.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates 1-decanol m/e 69 area.
:RU,MCA,069,,,,13:15
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,4,WNDOW1
```

MONS 024786

```
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of 1-Decanol
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 69.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates pentachlorophenol m/e 266 area.
:RU,MCA,265.7,,,,21:23
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,5,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of Pentachlorophenol
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 266.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates 1-octadecene m/e 97 area.
:RU,MCA,097,,,,21:23
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,6,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of 1-Octadecene
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 97.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates n-octadecane m/e 85 area.
:RU,MCA,085,,,,21:23
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,7,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of n-Octadecane
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 85.
:RU,DR,10,X
:RU,SHO,1
```

```
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** This section calculates DFTPP m/e 198 area.
:RU,FI,1G
:RU,MCA,198,,,,22:24
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,8,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of DFTPP
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 198.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates n-eicosane m/e 85 area.
:RU,MCA,85,,,,24:26
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,9,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of n-Eicosane
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 85.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates pyrene m/e 202 area.
:RU,MCA,202,,,,25:27
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,10,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of Pyrene
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 202.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates n-henaicosane m/e 85 area.
:RU,MCA,085,,,,25.5:27
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,11,WNDOW1
```

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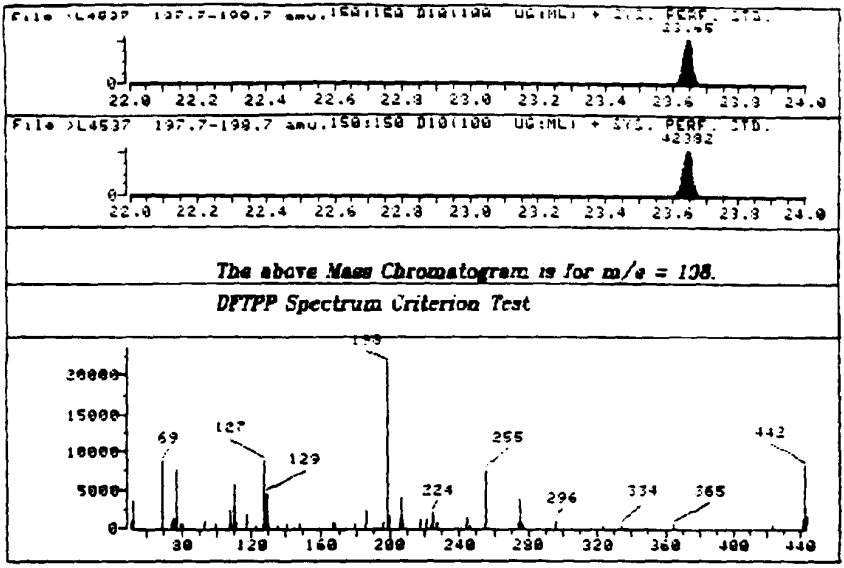
```
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of n-Heneicosane
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 85.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates methyl stearate m/e 74 area.
:RU,MCA,074,,,,25:27
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,12,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of Methyl Stearate
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 74.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:** The following calculates chrysene m/e 228 area.
:RU,MCA,228,,,,29:31
:RU,INT,,,1,,X,,WNDOW1
:RU,PF,1,,13,WNDOW1
:RU,DR,3,Y,,,,10523B,031463B
:RU,DR,2,Y,,,,10723B,031463B
:RU,DR,1,X,,,,110B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,3,Mass Spectrum of Chrysene
:RU,RDP,2,,
:RU,RDP,1,The above Mass Chromatogram is for m/e = 228.
:RU,DR,10,X
:RU,SHO,1
:RU,SHO,4
:RU,SHO,5
:RU,GR,,1
:RU,NEW
:RU,CRT,CA,FF
:AN,*************************************************************************
:AN,*  The following summarizes relative response factors for compound  *
:AN,*      characteristic masses versus anthracene-d10 m/e 188.         *
:AN,*                                                                   *
:AN,*     (RR = (Compound Characteristic Mass Area)/(m/e 188 Area)      *
:AN,*************************************************************************
:AN,2,6-dimethylphenol RR:
:RU,RCA,22,2R,DI,1R
:AN,2,6-dimethylaniline RR:
:RU,RCA,23,3R,DI,1R
:AN,1-decanol RR:
:RU,RCA,24,4R,DI,1R
```

```
:AN,pentachlorophenol RR:
:RU,RCA,25,5R,DI,1R
:AN,1-octadecene RR:
:RU,RCA,26,6R,DI,1R
:AN,n-octadecane RR:
:RU,RCA,27,7R,DI,1R
:AN,DFTPP RR:
:RU,RCA,28,8R,DI,1R
:RU,CRT,AL,CA
:AN,n-eicosane RR:
:RU,RCA,29,9R,DI,1R
:AN,pyrene RR:
:RU,RCA,30,10R,DI,1R
:AN,n-heneicosane RR:
:RU,RCA,31,11R,DI,1R
:AN,methyl stearate RR:
:RU,RCA,32,12R,DI,1R
:AN,chrysene RR:
:RU,RCA,33,13R,DI,1R
:*********************************************************************
:RU,CRT,AL,CA,FF
:RU,EC,,,21:23
:RU,CRT,CA
:RU,INT,,,4,,X
:RU,CRT,AL,CA
:RU,DR,3,X,,,,10523B,31463B
:RU,DR,2,X,,,,10723B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,1,Chromatographic Resolution Evaluation for Pentachloro-
:RU,RDP,2,,
:RU,RDP,3,phenol; Anthracene-d10; 1-Octadecene; and n-Octadecene.
:RU,DR,10,X
:RU,SHO,6
:RU,GR,,1
:RU,NEW
:RU,EC,,,24:27
:RU,CRT,CA,FF
:RU,INT,,,4,,X
:RU,CRT,AL,CA
:RU,DR,3,X,,,,10523B,31463B
:RU,DR,2,X,,,,10723B,31463B
:RU,AR,OURCXC,,,3,5
:RU,RDP,1,Chromatographic Resolution Evaluation for Eicosane;
:RU,RDP,2,,
:RU,RDP,3,Pyrene; Heneicosane; and Methyl Stearate.
:RU,DR,10,X
:RU,SHO,6
:RU,GR,,1
:RU,NEW
:SV,9G,,IH
:TR
```



GC/MS PERFORMANCE STANDARD

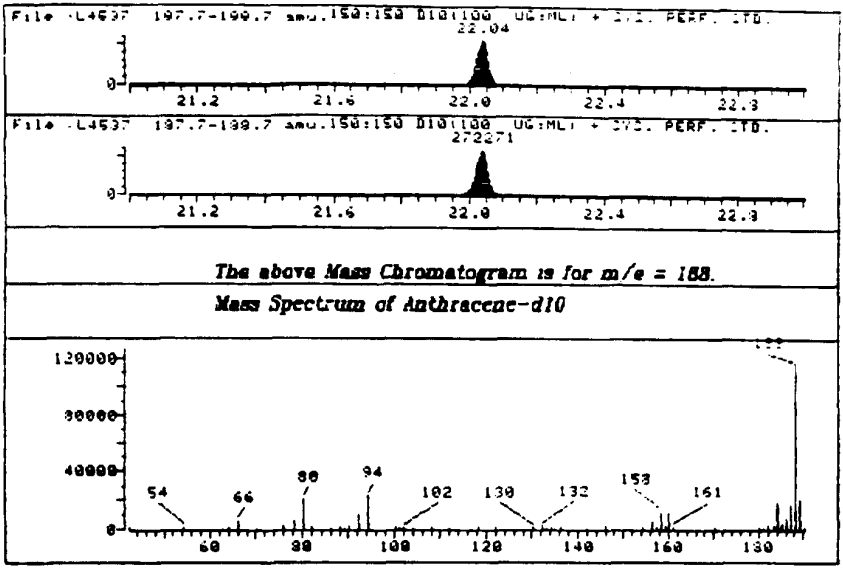
Decafluorotriphenylphospine (DFTPP)

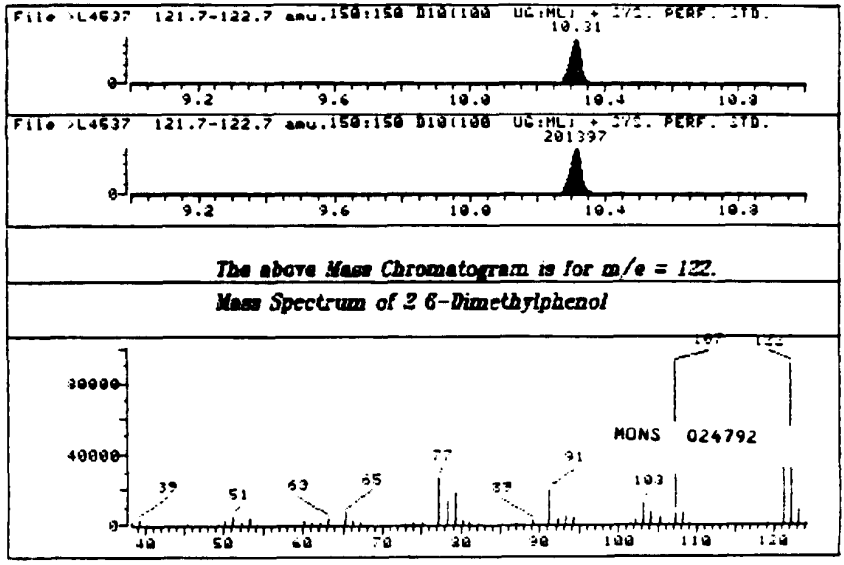
m/z	Ion Abundance Criteria	% Relative Abundance Base Peak	% Relative Abundance Appropriate Peak	Status
51	30-60% of mass 198	15.82	15.82	No Good
68	Less then 2% of mass 69	0.00	0.00	Ok
69	(reference only)	40.72	40.72	Ok
70	Less then 2% of mass 69	0.00	0.00	Ok
127	40-60% of mass 198	40.40	40.40	Ok
197	Less then 1% of mass 198	0.00	0.00	Ok
198	Base peak, 100% relative abundance	100.00	100.00	Ok
199	5-9% of mass 198	6.84	6.84	Ok
275	10-30% of mass 198	16.93	16.93	Ok
365	Greater then 1% of mass 198	1.67	1.67	Ok
441	Less then mass 443	6.18	99.39	Ok
442	Greater then 40% of mass 198	37.85	37.85	No Good
443	17-23% of mass 442	6.91	18.26	Ok

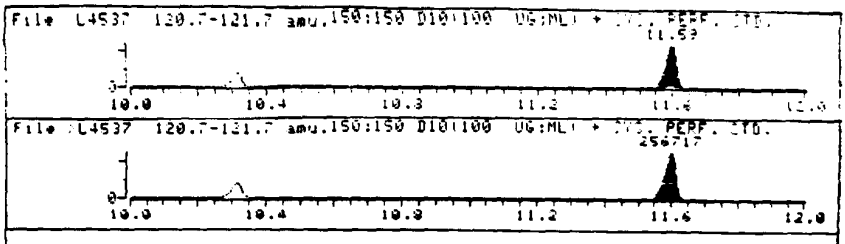
Injection Date: 11/19/87
Injection Time: 16:47
Data File: /L4537
Scan: 4669

MONS 024791

Spectrum fails specified criteria.

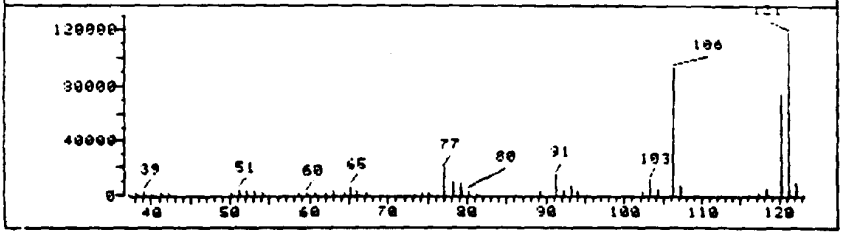

File >L4637 197.7-199.7 amu.150:150 D10(100 UG:ML) + 375. PERF. STD.
22.04
21.2
21.6
22.0
22.4
22.8
File >L4637 197.7-199.7 amu.150:150 D10(100 UG:ML) + 375. PERF. STD.
272271
21.2
21.6
22.0
22.4
22.8
The above Mass Chromatogram is for m/e = 188.
Mass Spectrum of Anthracene-d10
120000
80000
40000
54
66
80
94
102
130
132
158
161
60
80
100
120
140
160
180

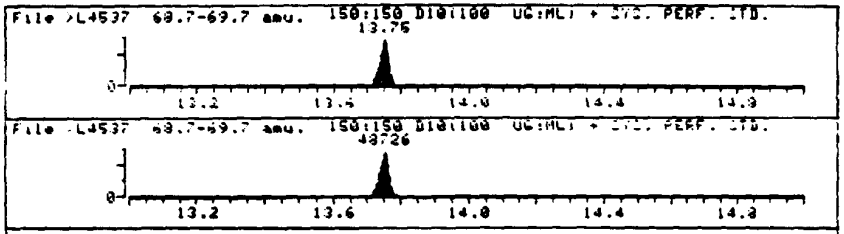

File >L4637 121.7-122.7 amu.150:150 D10(100 UG:ML) + 375. PERF. STD.
10.31
9.2
9.6
10.0
10.4
10.8
File >L4637 121.7-122.7 amu.150:150 D10(100 UG:ML) + 375. PERF. STD.
201397
9.2
9.6
10.0
10.4
10.8
The above Mass Chromatogram is for m/e = 122.
Mass Spectrum of 2 6-Dimethylphenol
80000
40000
39
51
63
65
77
91
103
MONS 024792
40
50
60
70
80
90
100
110
120



The above Mass Chromatogram is for m/e = 121.

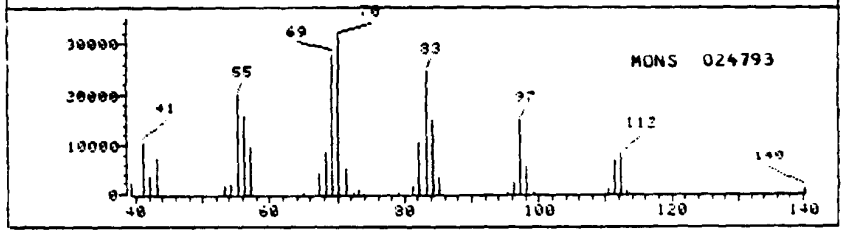
Mass Spectrum of 2 6-Dimethylaniline

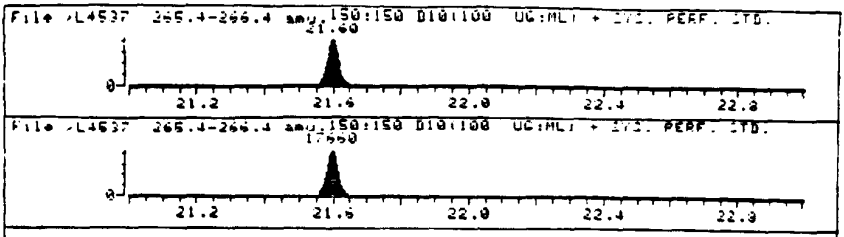




The above Mass Chromatogram is for m/e = 69.

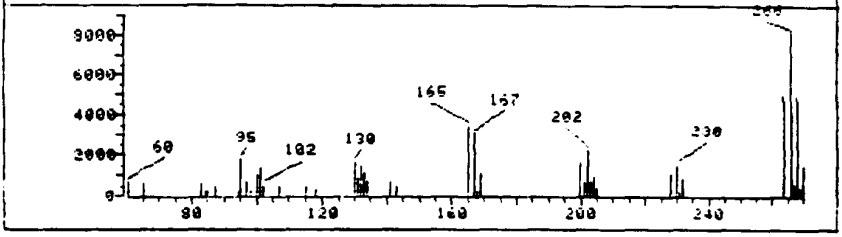
Mass Spectrum of 1-Decanol

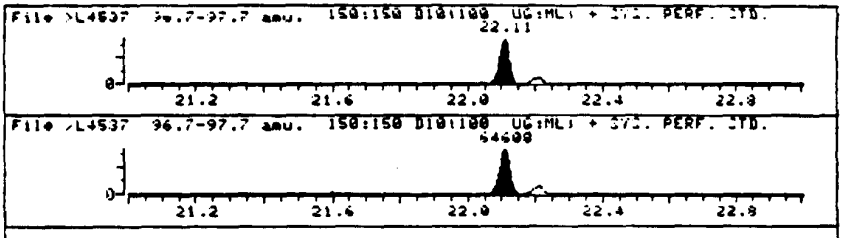




The above Mass Chromatogram is for m/e = 266.

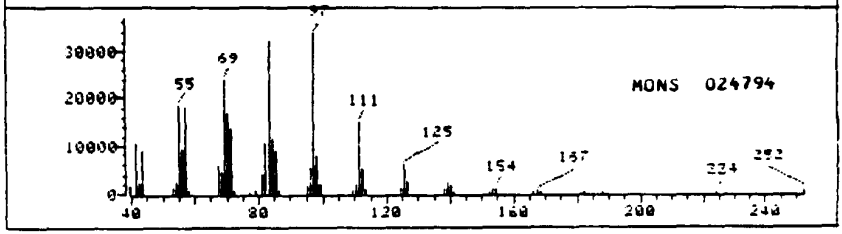
Mass Spectrum of Pentachlorophenol

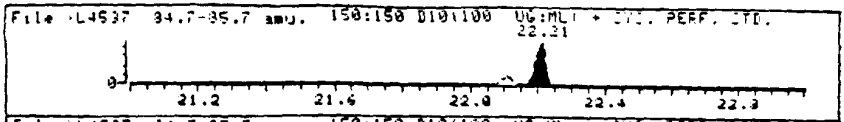


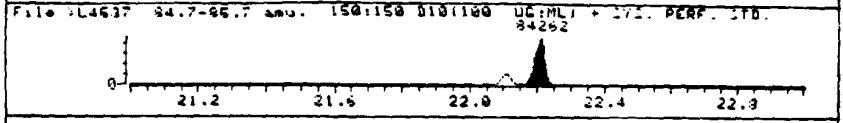


The above Mass Chromatogram is for m/e = 97.

Mass Spectrum of 1-Octadecene

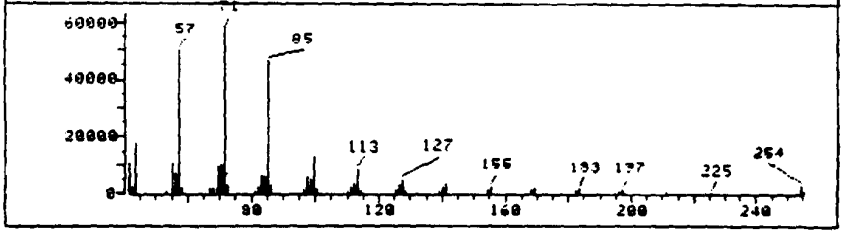


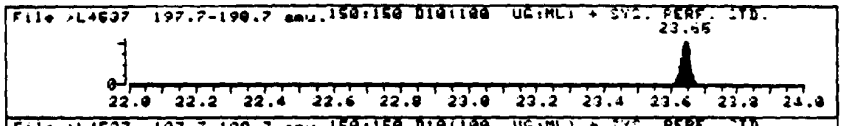


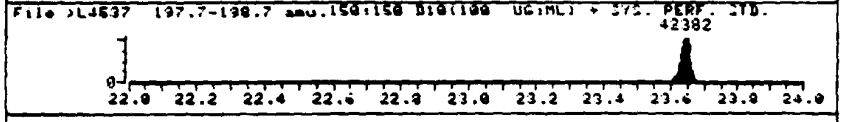


The above Mass Chromatogram is for m/e = 85.

Mass Spectrum of n-Octadecane

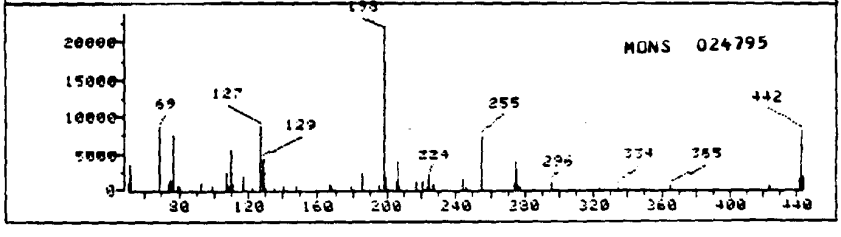


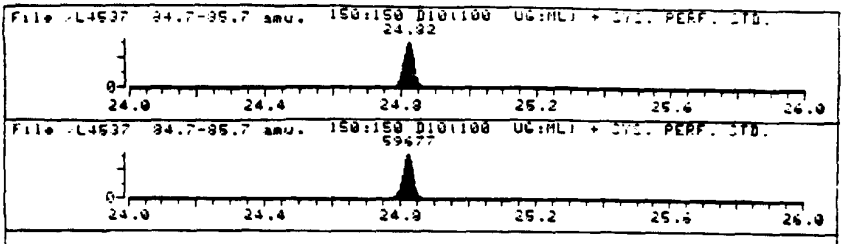




The above Mass Chromatogram is for m/e = 198.

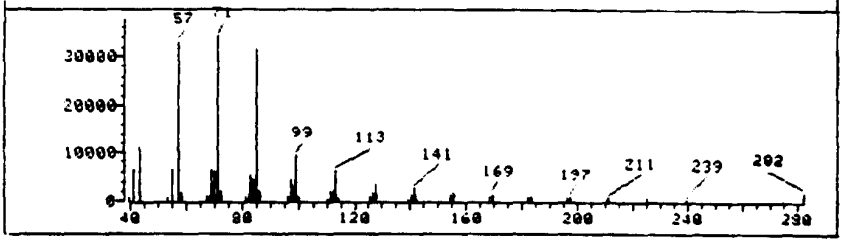
Mass Spectrum of DFTPP

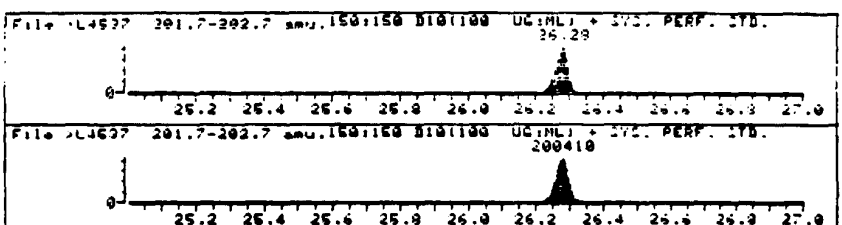




The above Mass Chromatogram is for m/e = 85.

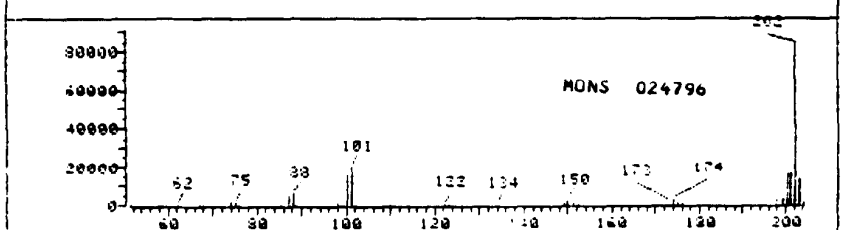
Mass Spectrum of n-Eicosane

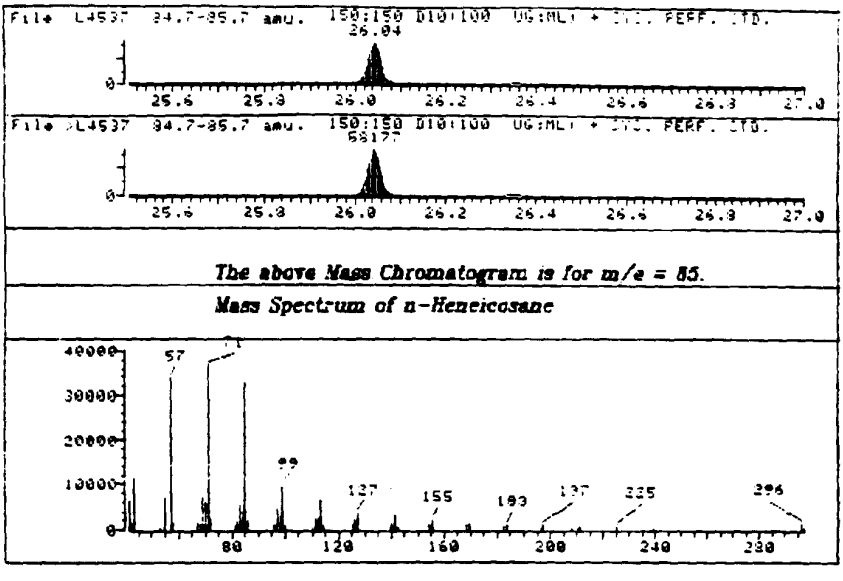




The above Mass Chromatogram is for m/e = 202.

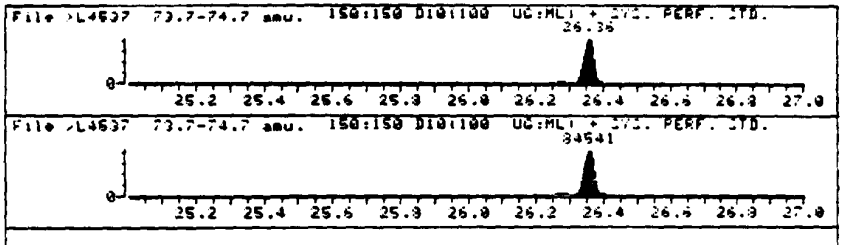
Mass Spectrum of Pyrene





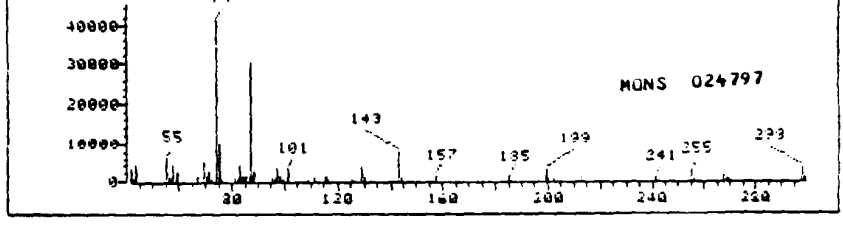
The above Mass Chromatogram is for m/e = 85.

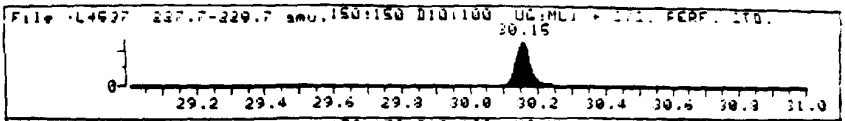
Mass Spectrum of n-Heneicosane

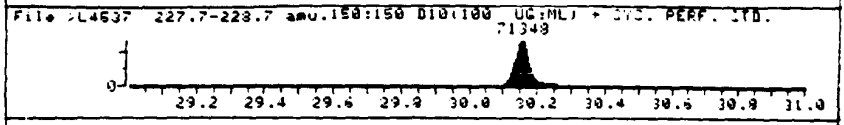


The above Mass Chromatogram is for m/e = 74.

Mass Spectrum of Methyl Stearate

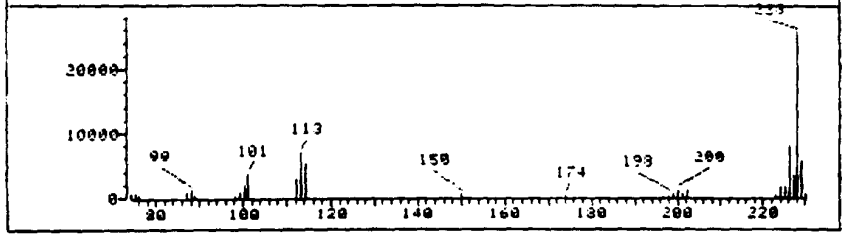






The above Mass Chromatogram is for m/e = 228.

Mass Spectrum of Chrysene



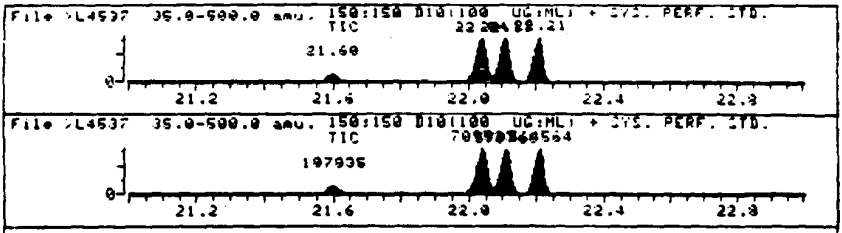
```
**********************************************************************
*  The Following summarizes relative response factors for compound
*       characteristic masses versus anthracene-d10 m/e 188
*
*      (RR = (Compound Characteristic Mass Area)/(m/e 188 Area)
**********************************************************************
2,6-dimethylphenol RR:

R-Global(22) =          .7397

2,6-dimethylaniline RR:

R-Global(23) =          .9429

1-decanol RR:

R-Global(24) =          .1790

pentachlorophenol RR:

R-Global(25) =          .0649

1-octadecene RR:

R-Global(26) =          .2373

n-octadecane RR:

R-Global(27) =          .3095

DFTPP RR:

R-Global(28) =          .1557


n-eicosane RR:

R-Global(29) =          .2192

pyrene RR:

R-Global(30) =          .7361

n-heneicosane RR:

R-Global(31) =          .2137

methyl stearate RR:

R-Global(32) =          .3105

chrysene RR:

R-Global(33) =          .2620
```

>L4537 150:150 D10(100 UG/ML) + SYS. PERF. STD

35.0-500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	21.601	4197	4207	4218	55838	107835	107835	15.20	5.49
2	22.040	4291	4306	4314	312260	711310	709523	100.00	36.16
3	22.111	4314	4322	4333	305338	579768	579768	81.71	29.55
4	22.214	4333	4345	4354	314455	564564	564564	79.57	28.78

Sum of corrected areas 1961690



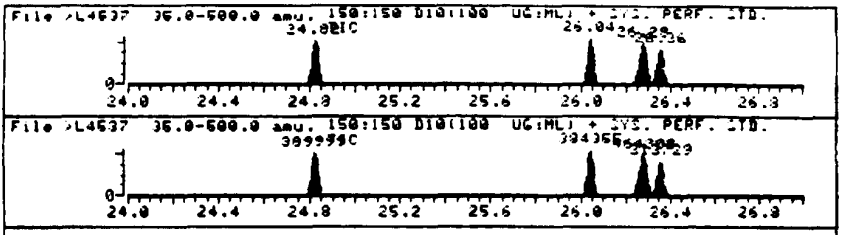
Chromatographic Resolution Evaluation for Pentachlorophenol; Anthracene-d10; 1-Octadecene; and n-Octadecene.

.L4537 150:150 D10(100 UG/ML) + SYS. PERF. STD #0

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max	% of total
1	24.819	4924	4933	4946	207021	389999	389999	84.00	25.122
2	26.042	5199	5209	5219	218277	384355	384355	82.78	24.759
3	26.281	5248	5263	5271	199483	466042	464308	100.00	29.905
4	26.357	5271	5280	5293	165097	316852	313729	67.57	20.209

Sum of corrected areas: 1552371



Chromatographic Resolution Evaluation for Eicosane; Pyrene; Henicosane; and Methyl Stearate.

Appendix D. Listing of PCB Congeners Used for Instrument Calibration and Procedure Files and Examples of Computer Outputs Produced in Incidental PCB Analyses. (PCCALC and PCOUTP Transfer Files.)

MONS 024802

Table D-1. Summary of Internal, Surrogate, and Native Standards used for PCB Chlorine Class Calibration and Native and Surrogate Spiking Studies.

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 instrument calibration)
2-monochlorobiphenyl
3-monochlorobiphenyl
4-monochlorobiphenyl
2,2'-dichlorobiphenyl
2,5-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

```
:du,pccalc,1
:**                                          Time Stamp - <871116.1004>
:**    :TR,PCCALC,filename1,output_filename,nnn
:**
:**    This transfer file is designed to calculate relative responses for com-
:**         pounds in samples and extracts analyzed for incidental PCBs.
:**
:**
:**         1G = mass spectrometric filename (>Lxxxx)
:**         2G = output_filename (PGxxxx)
:**         3G = 0 if sample not a standard, and
:**              nnn if sample is a standard.  (Note that nnn is the concen-
:**              tration of the standard in ug/mL.)
:**
:**
:**    The following R-globals are used.
:**         1R = anthracene-d10 m/e 188 area          31R = m/e 190 area
:**         2R = CL-biphenyl #1      m/e 188 area     32R = m/e 190 area
:**         3R =       "     #2      m/e 188 area     33R = m/e 190 area
:**         4R =       "     #3      m/e 188 area     34R = m/e 190 area
:**         5R = 4-CL-biphenyl-6Cl3 m/e 194 area      35R = m/e 196 area
:**         6R = CL2-biphenyl #1     m/e 222 area     36R = m/e 224 area
:**         7R =       "     #2      m/e 222 area     37R = m/e 224 area
:**         8R =       "     #3      m/e 222 area     38R = m/e 224 area
:**         9R =       "     #4      m/e 222 area     39R = m/e 224 area
:**        10R = CL3-biphenyl #1     m/e 255.9 area   40R = m/e 257.9 area
:**        11R =       "     #2      m/e 255.9 area   41R = m/e 257.9 area
:**        12R =       "     #3      m/e 255.9 area   42R = m/e 257.9 area
:**        13R = CL4-biphenyl #1     m/e 289.8 area   43R = m/e 291.8 area
:**        14R =       "     #2      m/e 289.8 area   44R = m/e 291.8 area
:**        15R =       "     #3      m/e 289.8 area   45R = m/e 291.8 area
:**        16R = CL4-BP-12Cl3 Surr.  m/e 301.8 area   46R = m/e 303.8 area
:**        17R = CL5-biphenyl #1     m/e 323.9 area   47R = m/e 325.9 area
:**        18R =       "     #2      m/e 323.9 area   48R = m/e 325.9 area
:**        19R =       "     #3      m/e 323.9 area   49R = m/e 325.9 area
:**        20R = CL6-biphenyl #1     m/e 357.8 area   50R = m/e 359.8 area
:**        21R =       "     #2      m/e 357.8 area   51R = m/e 359.8 area
:**        22R =       "     #3      m/e 357.8 area   52R = m/e 359.8 area
:**        23R = CL7-biphenyl #1     m/e 323.9 area   53R = m/e 325.9 area
:**        24R =       "     #2      m/e 323.9 area   54R = m/e 325.9 area
:**        25R = CL8-biphenyl        m/e 427.7 area   55R = m/e 429.7 area
:**        26R = CL8-BP-12Cl3 Surr.  m/e 439.7 area   56R = m/e 441.7 area
:**        27R = CL9-biphenyl        m/e 461.6 area   57R = m/e 463.6 area
:**        28R = CL10-biphenyl       m/e 497.6 area   58R = m/e 499.6 area
:**        29R = CL10-BP-12Cl3 Surr  m/e 509.6 area   59R = m/e 511.6 area
:**
:**
:**              In addition, R-globals 61R through 90R are used to store
:**                  delta-RTs of compounds 1R through 30R.  (Delta-RTs
:**                  are RTs of the first ion of the compound minus RTs of
:**                  the second ion of the compound.)
:SV,1,9,IH
:RU,RPN
:RU,RSE
:RU,NEW
:RU,CRT,CA,FF
:RU,MSR,1G
:RU,CRT,AL,CA,FF                                                    MONS  024804
:RU,TIM
:RU,STW,,1,0:75,10:30
```

```
:RU,STW,,2,0:75,30:48
:RU,STW,,3,0:75,60:080
:RU,STW,,4,0:75,80:098
:RU,STW,,10,0:75,50:60
:** This section calculates anthracene-d10 area.
:RU,FI,1G
:TR,IN#SU1,,,,188,21.5,22.4,1,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,Anthracene-d10 (m/e = 188 and 190) #1
:RU,RDP,2,GC/MS FILE =,1G
:RU,SHO,10
:RU,DR,10,X
:** The following calculates CL1-biphenyl Areas (m/e 188 and 190)
:TR,IN#SU2,,,,188,17.5,20,2,3
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL1-biphenyl (m/e = 188 and 190) #2-#4
:RU,RDP,2,GC/MS FILE =,1G
:RU,SHO,10
:RU,DR,10,X
:RU,GR,,1
:RU,NEW
:** The following calculates 4-CL-biphenyl-6C13 (m/e = 194 and 196)
:TR,IN#SU1,,,,194,18.5,20,5,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,4-CL-biphenyl-6C13 (194 and 196) #5
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL2-biphenyl (m/e = 222 and 224)
:TR,IN#SU2,,,,222,19,22.4,6,4
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL2-biphenyl (m/e = 222 and 224) #6-#9
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL3-biphenyl (m/e = 255.9 and 257.9)
:TR,IN#SU1,,,,255.9,21.4,24,10,3
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL3-biphenyl (m/e 255.9 & 257.9) #10-#12
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CLA-biphenyl (289.8 and 291.8)
:TR,IN#SU2,,,,289.8,23,26,13,3
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CLA-biphenyl (m/e 289.8 & 291.8) #13-#15
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CLA-biphenyl-12C13 (301.8 and 303.8)
:TR,IN#SU1,,,,301.8,26,28,16,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CLA-biphenyl-12C13 (301.8 and 303.8) #16
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
```

MONS 024805

```
:** The following calculates CL5-biphenyl (323.9 and 325.9)
:TR,IN=SU2,,,,323.9,25,29,17,3
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL5-biphenyl (m/e 323.9 & 325.9) #17-#19
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL6-biphenyl (357.8 and 359.8)
:TR,IN=SU1,,,,359.8,26,30.4,20,3
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL6-biphenyl (m/e 357.8 & 359.8) #20-#22
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL7-biphenyl (323.9 and 325.9)
:TR,IN=SU2,,,,393.8,29,30.5,23,2
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL7-biphenyl (m/e 323.9 & 325.9) #23-#24
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL8-biphenyl (427.7 and 429.7)
:TR,IN=SU1,,,,427.7,31,34,25,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL8-biphenyl (m/e 427.7 and 429.7) #25
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL8-biphenyl-12C13 (439.7 and 441.7)
:TR,IN=SU2,,,,439.7,30,31,26,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL8-biphenyl-12C13 (439.7 and 441.7) #26
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:**  The following calulcates CL9-biphenyl (461.6 and 463.6)
:TR,IN=SU1,,,,461.6,31,33,27,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL9-biphenyl (m/e 461.6 and 463.6) #27
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:**  The following calculates CL10-biphenyl (497.6 and 499.6)
:TR,IN=SU2,,,,497.6,33,35,28,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL10-biphenyl (m/e 497.6 and 499.6) #28
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:**  The following calculates CL10-biphenyl-12C13 (509.6 and 511.6)          MONS  024806
:TR,IN=SU1,,,,509.6,33,35,29,1
:RU,AR,PCBLBL,,,3,5
```

```
:RU,RDP,1,CL10-biphenyl-12C13 (509.6 and 511.6) #29
:RU,RDP,2,GC/MS FILE -,1G
:RU,DR,10,X
:RU,SNO,10
:RU,GR,,1
:RU,NEW
:TR,PCOUTP
:RU,CRT,FF,CA
:RU,WAIT,15
:DU,2G,1
:RU,CRT,AL,FF
:**  Use :SL,6,xxxxxx:sc,WRST,6  to setup spooling output to xxxxxx:sc and 6.
:**     then, XLU,,6 to direct all RDP outputs to LU 6.
:**     then, CS,6 to close xxxxxx:sc and outspool to lu 6.
:**     Now xxxxxx:sc is ready to transfer to Lotus 1-2-3.
:TR

:cu,pcoutp,1
FMGR 067
:du,pcoutp,1
:**    :TR,PCOUTP,filename,output_filename    Time Stamp - <870902.1259>
:**
:**    This transfer file is designed to output an ASCII file which can be used
:**         as an input file for data reduction of incidental PCB analysis
:**         data.  The following summarize globals used:
:**
:**       1G = mass spectrometric filename (>Lxxxx)
:**       2G = output filename
:**       3G =
:**
:**
:**
:**    The following R-globals are used.
:**
:**       1R = anthracene-d10 m/e 188 area          31R = m/e 190 area
:**       2R = CL-biphenyl #1     m/e 188 area      32R = m/e 190 area
:**       3R =       "        #2     m/e 188 area      33R = m/e 190 area
:**       4R =       "        #3     m/e 188 area      34R = m/e 190 area
:**       5R = 4-CL-biphenyl-6C13 m/e 194 area      35R = m/e 196 area
:**       6R = CL2-biphenyl #1    m/e 222 area      36R = m/e 224 area
:**       7R =       "        #2     m/e 222 area      37R = m/e 224 area
:**       8R =       "        #3     m/e 222 area      38R = m/e 224 area
:**       9R =       "        #4     m/e 222 area      39R = m/e 224 area
:**      10R = CL3-biphenyl #1    m/e 255.9 area    40R = m/e 257.9 area
:**      11R =       "        #2     m/e 255.9 area    41R = m/e 257.9 area
:**      12R =       "        #3     m/e 255.9 area    42R = m/e 257.9 area
:**      13R = CLA-biphenyl #1    m/e 289.8 area    43R = m/e 291.8 area
:**      14R =       "        #2     m/e 289.8 area    44R = m/e 291.8 area
:**      15R =       "        #3     m/e 289.8 area    45R = m/e 291.8 area
:**      16R = CLA-BP-12C13 Surr. m/e 301.8 area    46R = m/e 303.8 area
:**      17R = CL5-biphenyl #1    m/e 323.9 area    47R = m/e 325.9 area
:**      18R =       "        #2     m/e 323.9 area    48R = m/e 325.9 area
:**      19R =       "        #3     m/e 323.9 area    49R = m/e 325.9 area
:**      20R = CL6-biphenyl #1    m/e 357.8 area    50R = m/e 359.8 area
:**      21R =       "        #2     m/e 357.8 area    51R = m/e 359.8 area
:**      22R =       "        #3     m/e 357.8 area    52R = m/e 359.8 area
:**      23R = CL7-biphenyl #1    m/e 323.9 area    53R = m/e 325.9 area
:**      24R =       "        #2     m/e 323.9 area    54R = m/e 325.9 area
:**      25R = CL8-biphenyl        m/e 427.7 area    55R = m/e 429.7 area
:**      26R = CL8-BP-12C13 Surr. m/e 439.7 area    56R = m/e 441.7 area
```

```
:**      27R = CL9-biphenyl        m/e 461.6 area    57R = m/e 463.6 area
:**      28R = CL10-biphenyl       m/e 497.6 area    58R = m/e 499.6 area
:**      29R = CL10-BP-12C13 Surr m/e 509.6 area     59R = m/e 511.6 area
:**
:**
:**             In addition, R-globals 61R through 89R are used to store
:**                 delta-RTs of the corresponding compounds.  (Delta-RTs
:**                 are RTs of characteristic ion minus RTs of confirming ion)
:SV,1,9,IH
:RU,RPN
:RU,NEW
:RU,FI,1G
:CR,2G::PR:4:14
:SL,6,2G,WRST,6
:RU,XLU,,6
:LL,6
:DU,PCOUT3,6
:RU,MSH,1G
:RU,RDP,,Anthracene-d10 Area = ,1R, and Std. Conc. =,3G, ug/mL.
:DU,PCOUT1,6
:** The following calculates anthracene-d10 m/e 188 RRs, etc.
:RU,RSE,94:1R,95:31R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,001,94R,95R,61R,93R
:** The following calculates CL1-biphenyl #1 (m/e 188 and 190)
:RU,RSE,94:2R,95:32R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,002,94R,95R,62R,93R,1
:** The following calculates CL1-biphenyl #2 (m/e 188 and 190)
:RU,RSE,94:3R,95:33R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,003,94R,95R,63R,93R,1
:** The following calculates CL1-biphenyl #3 (m/e 188 and 190)
:RU,RSE,94:4R,95:34R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,004,94R,95R,64R,93R,1
:** The following calculates 4-CL-biphenyl-6C13 (m/e 194 and 196)
:RU,RSE,94:5R,95:35R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,005,94R,95R,65R,93R,1
:** The following calculates CL2-biphenyl #1 (m/e 222 and 224)
:RU,RSE,94:6R,95:36R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,006,94R,95R,66R,93R,2
:** The following calculates CL2-biphenyl #2 (m/e 222 and 224)
:RU,RSE,94:7R,95:37R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
```

```
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,007,94R,95R,67R,93R,2
:** The following calculates CL2-biphenyl #3 (m/e 222 and 224)
:RU,RSE,94:8R,95:38R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,008,94R,95R,68R,93R,2
:** The following calculates CL2-biphenyl #4 (m/e 222 and 224)
:RU,RSE,94:9R,95:39R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,009,94R,95R,69R,93R,2
:** The following calculates CL3-biphenyl #1 (m/e 255.9 and 257.9)
:RU,RSE,94:10R,95:40R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,010,94R,95R,70R,93R,3
:** The following calculates CL3-biphenyl #2 (m/e 255.9 and 257.9)
:RU,RSE,94:11R,95:41R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,011,94R,95R,71R,93R,3
:** The following calculates CL3-biphenyl #3 (m/e 255.9 and 257.9)
:RU,RSE,94:12R,95:42R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,012,94R,95R,72R,93R,3
:** The following calculates CLA-biphenyl #1 (m/e 289.8 and 291.8)
:RU,RSE,94:13R,95:43R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,013,94R,95R,73R,93R,4
:** The following calculates CLA-biphenyl #2 (m/e 289.8 and 291.8)
:RU,RSE,94:14R,95:44R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,014,94R,95R,74R,93R,4
:** The following calculates CLA-biphenyl #3 (m/e 289.8 and 291.8)
:RU,RSE,94:15R,95:45R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,015,94R,95R,75R,93R,4
:** The following calculates CLA-biphenyl-12C13 (m/e 301.8 and 303.8)
:RU,RSE,94:16R,95:46R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,016,94R,95R,76R,93R,4
:** The following calculates CL5-biphenyl #1 (m/e 223.9 and 225.9)
:RU,RSE,94:17R,95:47R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
```

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```
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,017,94R,95R,77R,93R,5
:** The following calculates CL5-biphenyl #2 (m/e 223.9 and 225.9)
:RU,RSE,94:18R,95:48R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,018,94R,95R,78R,93R,5
:** The following calculates CL5-biphenyl #3 (m/e 223.9 and 225.9)
:RU,RSE,94:19R,95:49R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,019,94R,95R,79R,93R,5
:** The following calculates CL6-biphenyl #1 (m/e 357.8 and 359.8)
:RU,RSE,94:20R,95:50R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,020,94R,95R,80R,93R,6
:** The following calculates CL6-biphenyl #2 (m/e 357.8 and 359.8)
:RU,RSE,94:21R,95:51R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,021,94R,95R,81R,93R,6
:** The following calculates CL6-biphenyl #3 (m/e 357.8 and 359.8)
:RU,RSE,94:22R,95:52R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,022,94R,95R,82R,93R,6
:** The following calculates CL7-biphenyl #1 (m/e 323.9 and 325.9)
:RU,RSE,94:23R,95:53R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,023,94R,95R,83R,93R,7
:** The following calculates CL7-biphenyl #2 (m/e 323.9 and 325.9)
:RU,RSE,94:24R,95:54R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,024,94R,95R,84R,93R,7
:** The following calculates CL8-biphenyl #1 (m/e 427.7 and 429.7)
:RU,RSE,94:25R,95:55R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,025,94R,95R,85R,93R,8
:** The following calculates CL8-biphenyl-12C13 (m/e 439.7 and 441.7)
:RU,RSE,94:26R,95:56R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,026,94R,95R,86R,93R,8
:** The following calculates CL9-biphenyl (m/e 461.6 and 463.6)
:RU,RSE,94:27R,95:57R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
```

MONS 024810

```
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,027,94R,95R,87R,93R,9
:** The following calculates CL10-biphenyl (m/e 497.6 and 499.6)
:RU,RSE,94:28R,95:58R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,028,94R,95R,88R,93R,10
:** The following calculates CL10-biphenyl-12C13 (m/e 509.6 and 511.6)
:RU,RSE,94:29R,95:59R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,029,94R,95R,89R,93R,10
:**  Use :SL,6,xxxxxx:sc,WRST,6  to setup spooling output to xxxxxx:sc and 6.
:**      then, XLU,,6 to direct all RDP outputs to LU 6.
:**      then, CS,6 to close xxxxxx:sc and outspool to lu 6.
:**      Now xxxxxx:sc is ready to transfer to Lotus 1-2-3.
:CS,6
:LL,1
:RU,XLU,,1
:SV,9G,,IH
:TR
```

```
:du,in#sul,1
:**
:**     :TR,IN#SU1,,,,4G,5G,6G,7G,8G
:**
:**         This transfer file should be treated as a subroutine to integrate
:**           one component's characteristic ion area and it's confirming
:**           ion area.  (The confirming ion is always assumed to be 2 masses
:**           above the characteristic ion.)
:**
:**         where:  4G = characteristic mass.  Later, R-global for confirming
:**                      ion area storage.
:**                 5G = starting retention time.  Later, R-global for delta-
:**                      RT storage.
:**                 6G = ending retention time.  Later, the peak # being stored
:**                 7G = R-global number for storage of characteristic ion area
:**                 8G = # of characteristic ion peaks to integrate.
:**
:**
:**
:SV
:RU,RSE,4G
:RU,RCA,0,OR,AD,2
:RU,MCA,4G,OR,,,5G:6G
:RU,INT,,,8G,,X
:SE,,,,,,1
:RU,PF,6G,,7G,,,90
:CA,4,7G,+,30
:CA,5,7G,+,60
:**    4G = 7G+30 (FOR CONFIRMING ION AREA STORAGE)
:**    5G = 7G+60 (FOR DELTA-RT STORAGE)
:**    Z = CONFIRMING ION; Y = CHARACTERISTIC ION; X = MASS SPECTRUM
:**       OF SELECTED ION MAXIMUM (NOT USED).
:**
:RU,RRH,SA,98,Y
:RU,RRH,BA,99,Y
:RU,DR,3,Y,AL,,,10720B,031463B,98R:99R
:RU,INT,,,,,Z
:RU,RSE,0
:RU,RCA,,0OR,SU,93R
:**   OR now contains -RT of characteristic ion.
:**
:RU,PF,OR,,4G,,,90
:**   DELTA-RT = (confirming ion RT) - (characteristic ion RT)
:**
:RU,RCA,5G,93R,AD,OR
:RU,RD
:RU,RRH,SA,98,Z
:RU,RRH,BA,99,Z
:RU,DR,4,Z,AL,,,10720B,021462B,98R:99R
:RU,STW,,10,0:75,50:60
:** BRANCH POINT!!!
```

```
:RU,RSE,0
:RU,RCA,,OR,SU,93R
:RU,PF,OR,,4G,,,90
:RU,RCA,5G,93R,AD,OR
:RU,RD
:IF,1,EQ,1,-16
:TR

:du,in#su2,1
:**
:**     :TR,IN#SU2,,,,4G,5G,6G,7G,8G
:**
:**         This transfer file should be treated as a subroutine to integrate
:**           one component's characteristic ion area and it's confirming
:**           ion area.  (The confirming ion is always assumed to be 2 masses
:**           above the characteristic ion.)
:**
:**         where:  4G = characteristic mass.  Later, R-global for confirming
:**                      ion area storage.
:**                 5G = starting retention time.  Later, R-global for delta-
:**                      RT storage.
:**                 6G = ending retention time.  Later, the peak # being stored
:**                 7G = R-global number for storage of characteristic ion area
:**                 8G = # of characteristic ion peaks to integrate.
:**
:**
:**
:SV
:RU,RSE,4G
:RU,RCA,0,OR,AD,2
:RU,MCA,4G,OR,,,5G:6G
:RU,INT,,,8G,,X
:SE,,,,,,1
:RU,PF,6G,,7G,,,90
:CA,4,7G,+,30
:CA,5,7G,+,60
:**   4G = 7G+30 (FOR CONFIRMING ION AREA STORAGE)
:**   5G = 7G+60 (FOR DELTA-RT STORAGE)
:**   Z = CONFIRMING ION; Y = CHARACTERISTIC ION; X = MASS SPECTRUM
:**     OF SELECTED ION MAXIMUM (NOT USED).
:**
:RU,RRM,SA,98,Y
:RU,RRM,BA,99,Y
:RU,DR,1,Y,AL,,,10720B,031463B,98R:99R
:RU,INT,,,,,Z
:RU,RSE,0
:RU,RCA,,OOR,SU,93R
:**  OR now contains -RT of characteristic ion.
:**
:RU,PF,OR,,4G,,,90
:**   DELTA-RT = (confirming ion RT) - (characteristic ion RT)
:**
:RU,RCA,5G,93R,AD,OR
:RU,RD
:RU,RRM,SA,98,Z
:RU,RRM,BA,99,Z
:RU,DR,2,Z,AL,,,10720B,021462B,98R:99R
:RU,STW,,10,0:75,0:10
:** BRANCH POINT!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!
:IF,8G,EQ,6G,+14
```

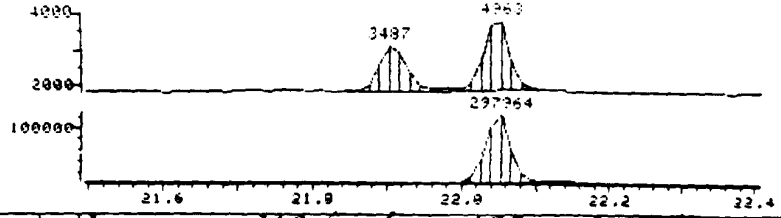
```
:RU,RD
:CA,4,4G,+,1
:CA,5,5G,+,1
:CA,6,6G,+,1
:CA,7,7G,+,1
:RU,INT,,,8G,,X
:RU,PF,6G,,7G,,,90
:RU,INT,,,,,Z
:RU,RSE,0
:RU,RCA,,0R,SU,93R
:RU,PF,0R,,4G,,,90
:RU,RCA,5G,93R,AD,0R
:RU,RD
:IF,1,EQ,1,-16
:TR
```

MONS 024814

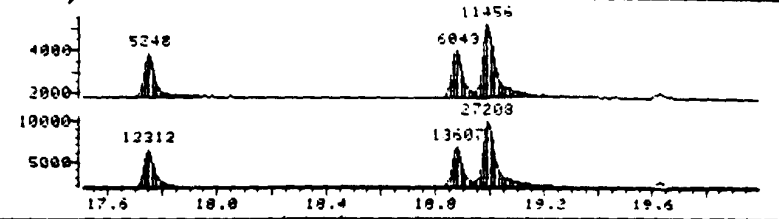
MS data file header from >L4525

Sample: 1uL 150:150 D10(10 U Operator: MASON MS2 11/13/87 22:00
Misc: G:ML) + HS SPK 2236-42 #21 BTL#21
Sys. #: 2 MS model: 87 SW/HW rev: IA ALS #: ***
Method file: PCSC25 Tuning file: MT8602 No. of extra records: 1
Source temp.: 200 Analyzer temp.: 0 Transfer line temp: 0

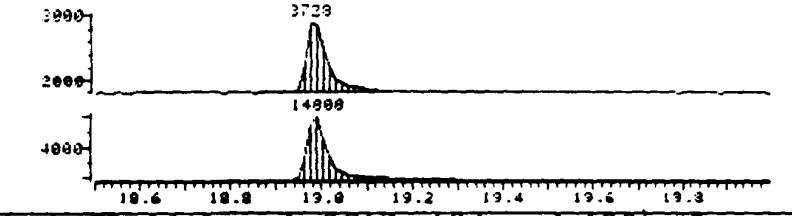
Chromatographic temperatures:	0	0	0	0	0
Chromatographic times, min:	0.0	0.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	0.0	0.0	0.0	0.0	0.0



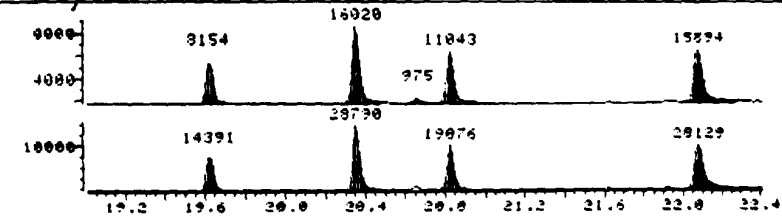
Anthracene-d10 (m/e = 188 and 190) #1
GC/MS FILE = >14525



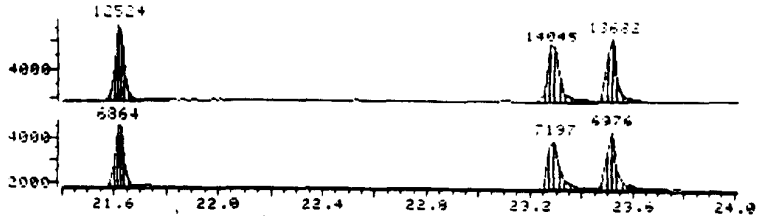
CL1-biphenyl (m/e = 188 and 190) #2-#4
GC/MS FILE = >14525



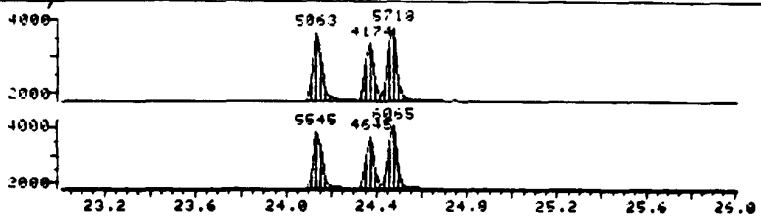
4-CL-biphenyl-6CL3 (194 and 196) #5
GC/MS FILE = >14525



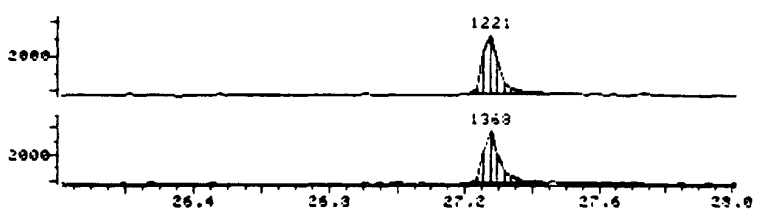
CL2-biphenyl (m/e = 222 and 224) #6-#9
GC/MS FILE = >14525



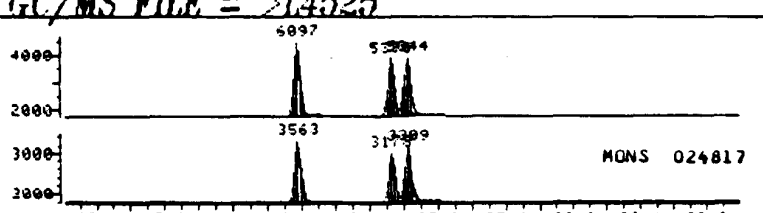
CL3-biphenyl (m/e 255.9 & 257.9) #10-#12
GC/MS FILE = >L4525



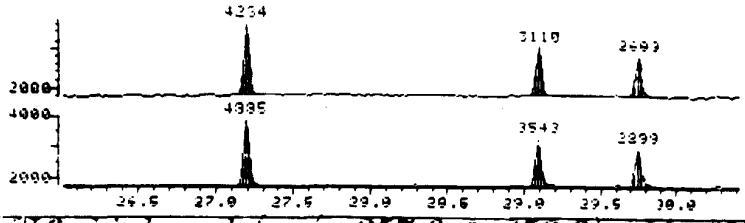
CL4-biphenyl (m/e 289.8 & 291.8) #13-#15
GC/MS FILE = >L4525



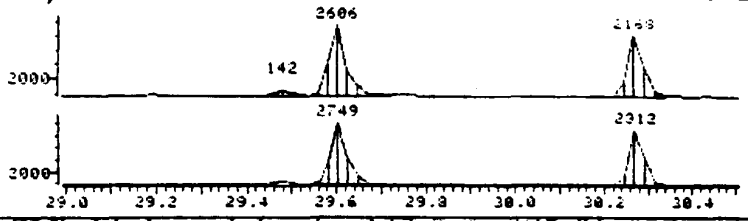
CL4-biphenyl-12C13 (301.8 and 303.8) #16
GC/MS FILE = >L4525



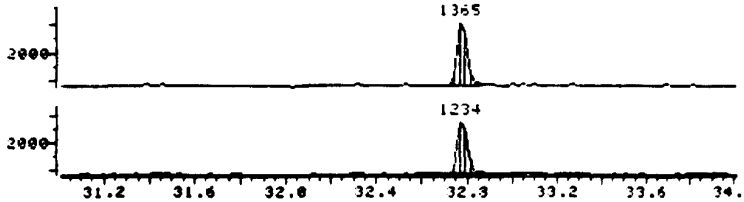
CL5-biphenyl (m/e 323.9 & 325.9) #17-#19
GC/MS FILE = >L4525



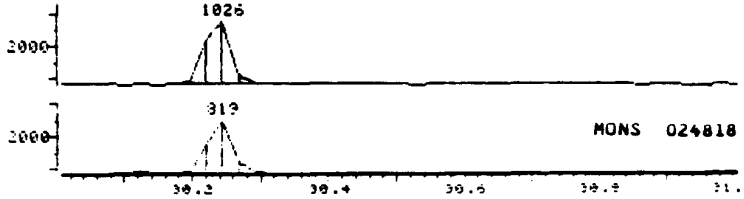
CL6-biphenyl (m/e 357.8 & 359.8) #20-#22
GC/MS FILE = >14525



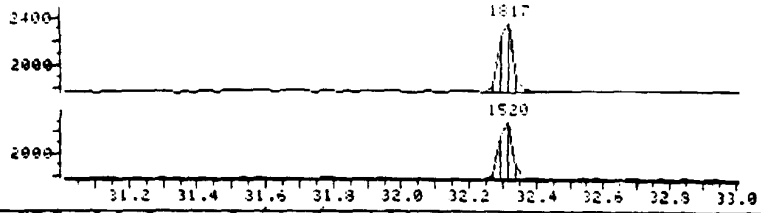
CL7-biphenyl (m/e 323.9 & 325.9) #23-#24
GC/MS FILE = >14525



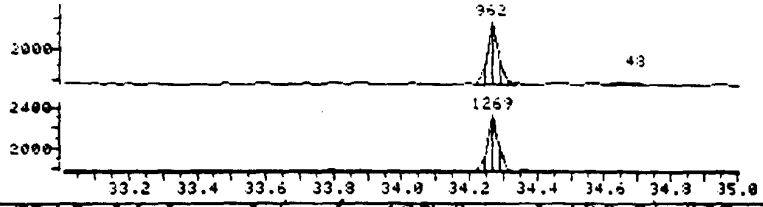
CL8-biphenyl (m/e 427.7 and 429.7) #25
GC/MS FILE = >14525



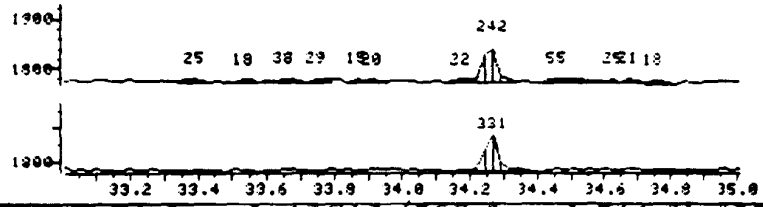
CL8-biphenyl-13C13 (439.7 and 441.7) #26
GC/MS FILE = >14525



CL9-biphenyl (m/e 461.6 and 463.6) #27
GC/MS FILE = >14525



CL10-biphenyl (m/e 497.6 and 499.6) #28
GC/MS FILE = >14525



CL10-biphenyl-12C13 (509.6 and 511.6) #29
GC/MS FILE = >14525

```
RU,WAIT,15
DU,P?4525,1
!!!
```

012	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
013	186	170	.00E+00	[illegible]	4
014	156	140	.00E+00	[illegible]	4
015	204	.192	.00E+00	[illegible]	[illegible]
016	.0459	.0410	.00E+00	112.04	4
017	120	205	.00E+00	58.44	5
018	.107	181	.00E+00	58.90	5
019	.111	.179	.00E+00	61.92	5
020	.164	142	.00E+00	115.38	6
021	119	.104	.00E+00	113.92	6
022	.0973	.0900	.00E+00	108.05	6
023	.0923	.0875	.00E+00	105.49	7
024	.0776	.0728	.00E+00	106.64	[illegible]
025	.0414	.0458	.00E+00	90.40	8
026	.0275	.0344	.00E+00	79.82	8
027	.0510	.0610	.00E+00	83.65	9
028	.0426	.0323	.00E+00	131.91	10
029	.0111	.00812	.00E+00	136.78	10

RU,CRT,AL,FF

MONS 024821

Appendix E. Listing of Procedure Files and Examples of Computer Outputs Produced in Incidental PCB Analyses. (PTCALC and PTOUTP Files.)

MONS 024822

```
:du,ptcalc.l
:**                                              Time Stamp - <871119.0842>
:**    :TR,PTCALC,filename1,output_filename,nnn
:**
:**    This transfer file is designed to calculate total relative responses for
:**         compounds in samples and extracts analyzed for incidental PCBs
:**         using MS acquisition method PCSC25.
:**
:**        1G = mass spectrometric filename (>Lxxxx)
:**        2G = output_filename (PTxxxx)
:**        3G = 0 if sample not a standard, and
:**             nnn if sample is a standard.  (Note that nnn is the concen-
:**             tration of the standard in ug/mL.)
:**
:**
:**    The following R-globals are used.
:**        1R = anthracene-d10 m/e 188 area          31R = m/e 190 area
:**        2R = SUM-CL-biphenyl    m/e 188 area      32R = m/e 190 area
:**        3R = NOT USED           m/e 188 area      33R = m/e 190 area
:**        4R =     "              m/e 188 area      34R = m/e 190 area
:**        5R = 4-CL-biphenyl-6C13 m/e 194 area      35R = m/e 196 area
:**        6R = SUM-CL2-biphenyl   m/e 222 area      36R = m/e 224 area
:**        7R = NOT USED           m/e 222 area      37R = m/e 224 area
:**        8R =     "              m/e 222 area      38R = m/e 224 area
:**        9R =     "              m/e 222 area      39R = m/e 224 area
:**       10R = SUM-CL3-biphenyl   m/e 255.9 area    40R = m/e 257.9 area
:**       11R = NOT USED           m/e 255.9 area    41R = m/e 257.9 area
:**       12R =     "              m/e 255.9 area    42R = m/e 257.9 area
:**       13R = SUM-CL4-biphenyl   m/e 289.8 area    43R = m/e 291.8 area
:**       14R = NOT USED           m/e 289.8 area    44R = m/e 291.8 area
:**       15R =     "              m/e 289.8 area    45R = m/e 291.8 area
:**       16R = CL4-BP-12C13 Surr. m/e 301.8 area    46R = m/e 303.8 area
:**       17R = SUM-CL5-biphenyl   m/e 323.9 area    47R = m/e 325.9 area
:**       18R = NOT USED           m/e 323.9 area    48R = m/e 325.9 area
:**       19R =     "              m/e 323.9 area    49R = m/e 325.9 area
:**       20R = SUM-CL6-biphenyl   m/e 359.8 area    50R = m/e 361.8 area
:**       21R = NOT USED           m/e 359.8 area    51R = m/e 361.8 area
:**       22R =     "              m/e 359.8 area    52R = m/e 361.8 area
:**       23R = SUM-CL7-biphenyl   m/e 393.8 area    53R = m/e 393.8 area
:**       24R = NOT USED           m/e 393.8 area    54R = m/e 393.8 area
:**       25R = SUM-CL8-biphenyl   m/e 427.7 area    55R = m/e 429.7 area
:**       26R = CL8-BP-12C13 Surr. m/e 439.7 area    56R = m/e 441.7 area
:**       27R = SUM-CL9-biphenyl   m/e 461.6 area    57R = m/e 463.6 area
:**       28R = CL10-biphenyl      m/e 497.6 area    58R = m/e 499.6 area
:**       29R = CL10-BP-12C13 Surr m/e 509.6 area    59R = m/e 511.6 area
:**
:**       97R = number of peaks detected in characteristic ion chromatogram.
:SV,1,9,IH
:RU,RPW
:RU,RSE
:RU,NEW
:RU,CRT,CA,FF
:RU,MSH,1G
:RU,CRT,AL,CA,FF
:RU,TIH
:RU,STW,,1,0:50,10:30
:RU,STW,,2,0:50,30:48
:RU,STW,,3,0:50,60:080
:RU,STW,,4,0:50,80:098
:RU,STW,,5,50:75,10:030
```

```
:RU,STW,,6,50:75,30:048
:RU,STW,,7,50:75,60:080
:RU,STW,,8,50:75,80:098
:RU,STW,,10,0:75,50:60
:**  This section calculates anthracene-d10 area.
:RU,FI,1G
:TR,IT*SU1,,,,188,21.5,22.4,1
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,31R
:RU,RDP,7,CHARAC =, 1R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,Anthracene-d10 (m/e = 188 and 190) #1
:RU,RDP,2,GC/MS FILE =,1G
:RU,SNO,10
:RU,DR,10,X
:** The following calculates CL1-biphenyl Areas (m/e 188 and 190)
:TR,IT*SU2,,,,188,17.5,20,2
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 32R
:RU,RDP,7,CHARAC =, 2R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL1-biphenyl (m/e = 188 and 190) #2
:RU,RDP,2,GC/MS FILE =,1G
:RU,SNO,10
:RU,DR,10,X
:RU,GR,,1
:RU,NEW
:** The following calculates 4-CL-biphenyl-6C13 (m/e = 194 and 196)
:TR,IT*SU1,,,,194,18.4,20,5,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,4-CL-biphenyl-6C13 (194 and 196) #5
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SNO,10
:** The following calculates CL2-biphenyl (m/e = 222 and 224)
:TR,IT*SU2,,,,222,19,22.4,6
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 36R
:RU,RDP,7,CHARAC =, 6R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL2-biphenyl (m/e = 222 and 224) #6
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SNO,10
:RU,GR,,1
```

```
:RU,NEW
:** The following calculates CL3-biphenyl (m/e = 255.9 and 257.9)
:TR,IT=SU1,,,,255.9,21,26,10
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,40R
:RU,RDP,7,CHARAC =,10R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL3-biphenyl (m/e 255.9 & 257.9) #10
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL4-biphenyl (289.8 and 291.8)
:TR,IT=SU2,,,,289.8,22.8,28,13
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 43R
:RU,RDP,7,CHARAC =,13R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL4-biphenyl (m/e 289.8 & 291.8) #13
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL4-biphenyl-12C13 (301.8 and 303.8)
:TR,IT=SU1,,,,301.8,26,28,16,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL4-biphenyl-12C13 (301.8 and 303.8) #16
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL5-biphenyl (323.9 and 325.9)
:TR,IT=SU2,,,,323.9,24,29.4,17
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 47R
:RU,RDP,7,CHARAC =,17R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL5-biphenyl (m/e 323.9 & 325.9) #17
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL6-biphenyl (359.8 and 361.8)
:TR,IT=SU1,,,,359.8,25,31,20
:RU,RIF,97R,EQ,0
```

```
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,50R
:RU,RDP,7,CHARAC =,20R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL6-biphenyl (m/e 359.8 & 361.8) #20
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL7-biphenyl (393.8 and 395.8)
:TR,IT#SU2,,,,393.8,28,33,23
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,  53R
:RU,RDP,7,CHARAC =,23R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL7-biphenyl (m/e 393.8 & 395.8) #23
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL8-biphenyl (427.7 and 429.7)
:TR,IT#SU1,,,,427.7,30,34,25
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,55R
:RU,RDP,7,CHARAC =,25R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL8-biphenyl (m/e 427.7 & 429.7) #25
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL8-biphenyl-12C13 (439.7 and 441.7)
:TR,IN#SU2,,,,439.7,30,31,26,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL8-biphenyl-12C13 (439.7 and 441.7) #26
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:**  The following calulcates CL9-biphenyl (461.6 and 463.6)
:TR,IT#SU1,,,,461.6,32,34,27
:RU,RIF,97R,EQ,0
:IF,1P,EQ,0,1
:IF,1P,EQ,1P,4
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,57R
```

MONS 024826

```
:RU,RDP,7,CHARAC =,27R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,SUM-CL9-biphenyl (m/e 461.6 & 463.6) #27
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:**  The following calculates CL10-biphenyl (497.6 and 499.6)
:TR,IN#SU2,,,,497.6,33,35,28,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL10-biphenyl (m/e 497.6 and 499.6) #28
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:**  The following calculates CL10-biphenyl-12C13 (509.6 and 511.6)
:TR,IN#SU1,,,,509.6,33,35,29,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL10-biphenyl-12C13 (509.6 and 511.6) #29
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:TR,PTOUTP
:RU,CRT,FF,CA
:RU,WAIT,15
:DU,2G,1
:RU,CRT,AL,FF
:**  Use :SL,6,xxxxxx:sc,WRST,6  to setup spooling output to xxxxxx:sc and 6.
:**      then, XLU,,6 to direct all RDP outputs to LU 6.
:**      then, CS,6 to close xxxxxx:sc and outspool to lu 6.
:**      Now xxxxxx:sc is ready to transfer to Lotus 1-2-3.
:TR
```

```
:du,ptoutp,1
:**    :TR,PTOUTP,filename,output_filename   Time Stamp - <871113.1407>
:**
:**   This transfer file is designed to output an ASCII file which can be used
:**         as an input file for data reduction of total incidental PCB
:**         analysis data.  The following summarize globals used:
:**
:**       1G = mass spectrometric filename (>Lxxxx)
:**       2G = output filename
:**       3G =
:**
:**
:**
:**   The following R-globals are used.
:**
:**       1R = anthracene-d10 m/e 188 area          31R = m/e 190 area
:**       2R = SUM-CL-biphenyl    m/e 188 area      32R = m/e 190 area
:**       3R = NOT USED           m/e 188 area      33R = m/e 190 area
:**       4R =      "             m/e 188 area      34R = m/e 190 area
:**       5R = 4-CL-biphenyl-6C13 m/e 194 area      35R = m/e 196 area
:**       6R = SUM-CL2-biphenyl   m/e 222 area      36R = m/e 224 area
:**       7R = NOT USED           m/e 222 area      37R = m/e 224 area
:**       8R =      "             m/e 222 area      38R = m/e 224 area
:**       9R =      "             m/e 222 area      39R = m/e 224 area
:**      10R = SUM-CL3-biphenyl   m/e 255.9 area    40R = m/e 257.9 area
:**      11R = NOT USED           m/e 255.9 area    41R = m/e 257.9 area
:**      12R =      "             m/e 255.9 area    42R = m/e 257.9 area
:**      13R = SUM-CL4-biphenyl   m/e 289.8 area    43R = m/e 291.8 area
:**      14R = NOT USED           m/e 289.8 area    44R = m/e 291.8 area
:**      15R =      "             m/e 289.8 area    45R = m/e 291.8 area
:**      16R = CL4-BP-12C13 Surr. m/e 301.8 area    46R = m/e 303.8 area
:**      17R = SUM-CL5-biphenyl   m/e 323.9 area    47R = m/e 325.9 area
:**      18R = NOT USED           m/e 323.9 area    48R = m/e 325.9 area
:**      19R =      "             m/e 323.9 area    49R = m/e 325.9 area
:**      20R = SUM-CL6-biphenyl   m/e 359.8 area    50R = m/e 361.8 area
:**      21R = NOT USED           m/e 359.8 area    51R = m/e 361.8 area
:**      22R =      "             m/e 359.8 area    52R = m/e 361.8 area
:**      23R = SUM-CL7-biphenyl   m/e 393.8 area    53R = m/e 395.8 area
:**      24R = NOT USED           m/e 393.8 area    54R = m/e 395.8 area
:**      25R = SUM-CL8-biphenyl   m/e 427.7 area    55R = m/e 429.7 area
:**      26R = CL8-BP-12C13 Surr. m/e 439.7 area    56R = m/e 441.7 area
:**      27R = SUM-CL9-biphenyl   m/e 461.6 area    57R = m/e 463.6 area
:**      28R = CL10-biphenyl      m/e 497.6 area    58R = m/e 499.6 area
:**      29R = CL10-BP-12C13 Surr m/e 509.6 area    59R = m/e 511.6 area
:**
:**
:SV,1,9,IH
:RU,RPW
:RU,NEW
:RU,FI,1G
:CR,2G::PR:4:14
:SL,6,2G,WRST,6
:RU,XLU,,6
:LL,6
:DU,PTOUT3,6
:RU,MSH,1G
:RU,RDP,,Anthracene-d10 Area = ,1R, and Std. Conc. =,3G, ug/mL.
:DU,PTOUT1,6
:** The following calculates anthracene-d10 m/e 188 RRs, etc.
:RU,RSE,94:1R,95:31R
```

MONS 024828

```
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,011
:** NOT USED!! The following calculates CL3-biphenyl #3 (m/e 255.9 and 257.9)
:**,RSE,94:12R,95:42R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,012
:** The following calculates CL4-biphenyl #1 (m/e 289.8 and 291.8)
:RU,RSE,94:13R,95:43R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,013,94R,95R,73R,93R,4
:** NOT USED!! The following calculates CL4-biphenyl #2 (m/e 289.8 and 291.8)
:**,RSE,94:14R,95:44R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,014
:** NOT USED!! The following calculates CL4-biphenyl #3 (m/e 289.8 and 291.8)
:**,RSE,94:15R,95:45R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,015
:** The following calculates CL4-biphenyl-12C13 (m/e 301.8 and 303.8)
:RU,RSE,94:16R,95:46R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,016,94R,95R,76R,93R,4
:** The following calculates SUM-CL5-biphenyl #1 (m/e 223.9 and 225.9)
:RU,RSE,94:17R,95:47R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,017,94R,95R,77R,93R,5
:** NOT USED!! The following calculates CL5-biphenyl #2 (m/e 223.9 and 225.9)
:**,RSE,94:18R,95:48R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,018
:** NOT USED!! The following calculates CL5-biphenyl #3 (m/e 223.9 and 225.9)
:**,RSE,94:19R,95:49R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,019
:** The following calculates SUM-CL6-biphenyl #1 (m/e 359.8 and 361.8)
:RU,RSE,94:20R,95:50R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,020,94R,95R,80R,93R,6
:** NOT USED!! The following calculates CL6-biphenyl #2 (m/e 359.8 and 361.8)
:**,RSE,94:21R,95:51R
```

```
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,001,94R,95R,61R,93R
:** The following calculates SUM-CL1-biphenyl #1 (m/e 188 and 190)
:RU,RSE,94:2R,95:32R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,002,94R,95R,62R,93R,1
:** NOT USED!!The following calculates CL1-biphenyl #2 (m/e 188 and 190)
:**,RSE,94:3R,95:33R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,003
:** NOT USED!! The following calculates CL1-biphenyl #3 (m/e 188 and 190)
:**,RSE,94:4R,95:34R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,004
:** The following calculates 4-CL-biphenyl-6C13 (m/e 194 and 196)
:RU,RSE,94:5R,95:35R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,005,94R,95R,65R,93R,1
:** The following calculates SUM-CL2-biphenyl #1 (m/e 222 and 224)
:RU,RSE,94:6R,95:36R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,006,94R,95R,66R,93R,2
:** NOT USED!! The following calculates CL2-biphenyl #2 (m/e 222 and 224)
:**,RSE,94:7R,95:37R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,007
:** NOT USED!! The following calculates CL2-biphenyl #3 (m/e 222 and 224)
:**,RSE,94:8R,95:38R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,008
:** NOT USED!! The following calculates CL2-biphenyl #4 (m/e 222 and 224)
:**,RSE,94:9R,95:39R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,009
:** The following calculates SUM-CL3-biphenyl #1 (m/e 255.9 and 257.9)
:RU,RSE,94:10R,95:40R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,010,94R,95R,70R,93R,3
:** NOT USED!! The following calculates CL3-biphenyl #2 (m/e 255.9 and 257.9)
:**,RSE,94:11R,95:41R
```

```
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,021
:** NOT USED!! The following calculates CL6-biphenyl #3 (m/e 359.8 and 361.8)
:**,RSE,94:22R,95:52R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,022
:** The following calculates SUM-CL7-biphenyl #1 (m/e 393.8 and 395.8)
:RU,RSE,94:23R,95:53R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,023,94R,95R,83R,93R,7
:** NOT USED!! The following calculates CL7-biphenyl #2 (m/e 393.8 and 395.8)
:**,RSE,94:24R,95:54R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,024
:** The following calculates SUM-CL8-biphenyl #1 (m/e 427.7 and 429.7)
:RU,RSE,94:25R,95:55R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,025,94R,95R,85R,93R,8
:** The following calculates CL8-biphenyl-12C13 (m/e 439.7 and 441.7)
:RU,RSE,94:26R,95:56R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,026,94R,95R,86R,93R,8
:** The following calculates SUM-CL9-biphenyl (m/e 461.6 and 463.6)
:RU,RSE,94:27R,95:57R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,027,94R,95R,87R,93R,9
:** The following calculates CL10-biphenyl (m/e 497.6 and 499.6)
:RU,RSE,94:28R,95:58R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,028,94R,95R,88R,93R,10
:** The following calculates CL10-biphenyl-12C13 (m/e 509.6 and 511.6)
:RU,RSE,94:29R,95:59R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,029,94R,95R,89R,93R,10
:**  Use :SL,6,xxxxxx:sc,WRST,6  to setup spooling output to xxxxxx:sc and 6.
:**      then, XLU,,6 to direct all RDP outputs to LU 6.
:**      then, CS,6 to close xxxxxx:sc and outspool to lu 6.
:**      Now xxxxxx:sc is ready to transfer to Lotus 1-2-3.
:CS,6
:LL,1                                                          MONS  024831
:RU,XLU,,1
:SV,9G,,IH
```

```
:du,it#sul,1
:**
:**     :TR,IT#SU1,,,,4G,5G,6G,7G
:**
:**         This transfer file should be treated as a subroutine to integrate
:**           one component's characteristic ion area and it's confirming
:**           ion area.  (The confirming ion is always assumed to be 2 masses
:**           above the characteristic ion.)
:**
:**         where:  4G = characteristic mass.  Later, R-global for confirming
:**                      ion area storage.  Even later, total confirming
:**                      ions area.
:**                 5G = starting retention time.
:**                 6G = ending retention time.
:**                 7G = R-global number for storage of characteristic ion
:**                      area.  Later, total characteristic ions area.
:**
:**               97R = number of peaks detected in characteristic ion chrom.
:**               98R = minimum in ion chromatograms.
:**               99R = maximum in ion chromatograms.
:**
:SV
:RU,STW,,10,50:75,60:98
:RU,RSE,4G
:RU,RCA,0,0R,AD,2
:RU,MCA,4G,0R,,,5G:6G
:RU,INT,,-100,30,,X,,CHARAC
:RU,RGA,97,1P
:CA,4,7G,+,30
:**   4G = 7G+30 (FOR CONFIRMING ION AREA STORAGE)
:**   Y = CONFIRMING ION; X = CHARACTERISTIC ION;
:**
:**  The following two steps sets 4G and 7G "R" globals to 0.00.
:RU,RCA,4G
:RU,RCA,7G
:RU,RRH,SA,98,X
:RU,RRH,BA,99,X
:RU,DR,3,X,AL,,,10720B,031463B,98R:99R
:RU,INT,,-100,,,Y,,CONFRM
:**
:**
:RU,RRH,SA,98,Y
:RU,RRH,BA,99,Y
:RU,DR,4,Y,AL,,,10720B,021462B,98R:99R
:RU,RDP,,# PEAKS =,97R
:RU,RSE,4G:0,7G:0
:DP,1P
:RU,RIF,97R,EQ,0
:DP,1P
:IF,1P,EQ,0,1
:TR
:**  JMP +1
:**  JMP +2
:RU,RCH,CONFRM
:RU,RCH,CHARAC
:RU,ADD,0.04,T
:RU,RCH,CHARAC
:RU,SUB,T,0.04,T
:RU,RCH,CHARAC
:RU,DR,7,X,5G:6G,,,11B,10421B
```

MONS 024832

```
:RU,SUM,X
:RU,RRH,BA,7G,X
:RU,DR,8,Y,5G:6G,,,11B,10421B
:RU,SUM,Y
:RU,RRH,BA,4G,Y
:TR

:du,ic#su2,1
:**
:**     :TR,IT#SU2,,,,4G,5G,6G,7G
:**
:**         This transfer file should be treated as a subroutine to integrate
:**           one component's characteristic ion area and it's confirming
:**           ion area.  (The confirming ion is always assumed to be 2 masses
:**           above the characteristic ion.)
:**
:**         where:  4G = characteristic mass.  Later, R-global for confirming
:**                      ion area storage.  Even later, total confirming ions
:**                      area.
:**                 5G = starting retention time.
:**                 6G = ending retention time.
:**                 7G = R-global number for storage of characteristic ion
:**                      area.  Later, total characteristic ions area.
:**
:**                97R = number of peaks detected in characteristic ion chrom.
:**                98R = minimum in ion chromatograms.
:**                99R = maximum in ion chromatograms.
:**
:**
:**
:SV
:RU,STW,,10,50:75,10:48
:RU,RSE,4G
:RU,RCA,0,OR,AD,2
:RU,MCA,4G,OR,,,5G:6G
:RU,INT,,-100,30,,X,,CHARAC
:RU,RCA,97,1P
:CA,4,7G,+,30
:**   4G = 7G+30 (FOR CONFIRMING ION AREA STORAGE)
:**   Y = CONFIRMING ION; X = CHARACTERISTIC ION;
:**
:**  The following two steps set the 4G and 7G "R" values to 0.
:RU,RCA,4G
:RU,RCA,7G
:RU,RRH,SA,98,X
:RU,RRH,BA,99,X
:RU,DR,1,X,AL,,,10720B,031463B,98R:99R
:RU,INT,,-100,,,Y,,CONFRM
:**
:**
:RU,RRH,SA,98,Y
:RU,RRH,BA,99,Y
:RU,DR,2,Y,AL,,,10720B,021462B,98R:99R
:RU,RSE,4G:0,7G:0
:RU,RDP,,# PEAKS =,97R
:DP,1P
:RU,RIF,97R,EQ,0
:DP,1P
:IF,1P,EQ,0,1
:TR
```

```
:** JMP +1
:** JMP +2
:RU,RCH,CONFRM
:RU,RCH,CHARAC
:RU,ADD,0.04,T
:RU,RCH,CHARAC
:RU,SUB,T,0.04,T
:RU,RCH,CHARAC
:RU,DR,5,X,5G:6G,,,11B,10421B
:RU,SUM,X
:RU,RRH,BA,7G,X
:RU,DR,6,Y,5G:6G,,,11B,10421B
:RU,SUM,Y
:RU,RRH,BA,4G,Y
:TR
```

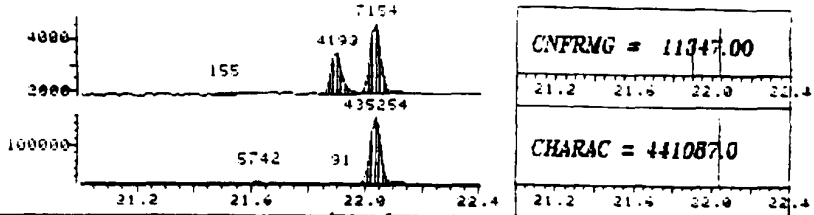
MONS 024834

MS data file header from -L4494

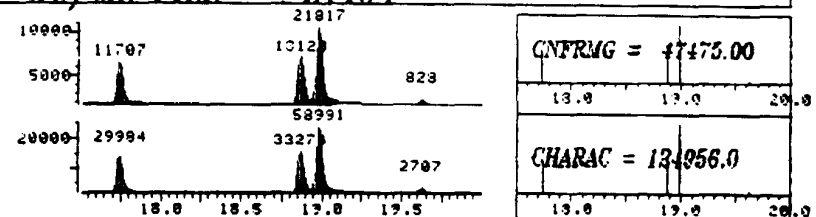
Sample 10L 130:130 D10(10UG Operator: MASON MS2 10/30/87 14:43
Misc ML) + LOW NATIVE SPK (1 UG/ML) #01 BTL# 1
Sys. #: 2 MS model: 87 SW/HW rev. IA ALS # ***
Method file: PCSC25 Tuning file: MT8602 No. of extra records 4
Source temp. 200 Analyzer temp. 0 Transfer line temp 0

Chromatographic temperatures	0.	0	0	0	0
Chromatographic times, min :	0.0	0.0	0.0	0.0	0.0
Chromatographic rate, deg/min.	0.0	0.0	0.0	0.0	0.0

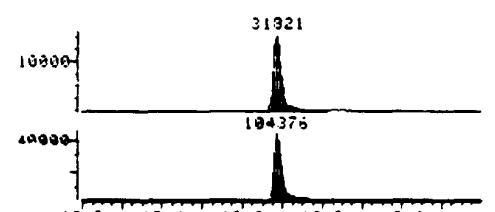
MONS 024835



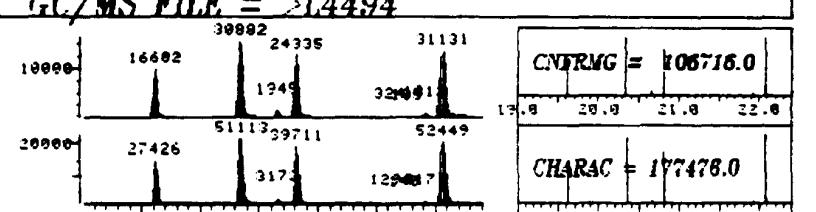
Anthracene-d10 (m/e = 188 and 190) #1
GC/MS FILE = >14494



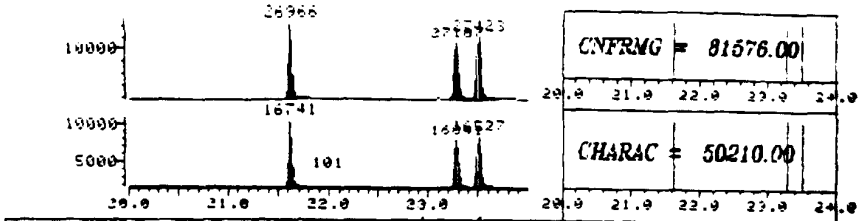
SUM-CL1-biphenyl (m/e = 188 and 190) #2
GC/MS FILE = >14494



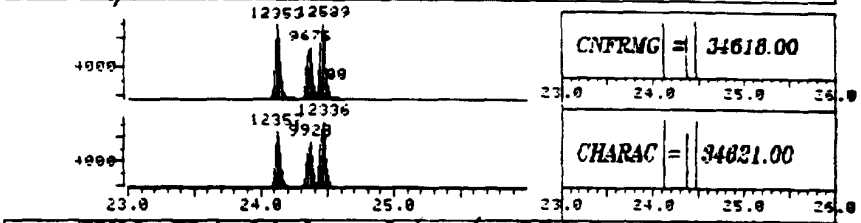
4-CL-biphenyl-6C13 (194 and 196) #5
GC/MS FILE = >14494



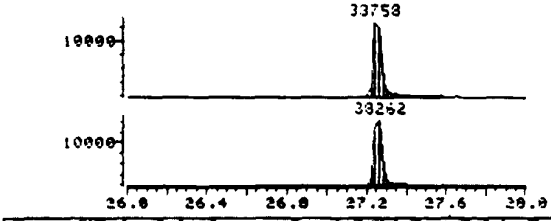
SUM-CL2-biphenyl (m/e = 222 and 224) #6
GC/MS FILE = >14494



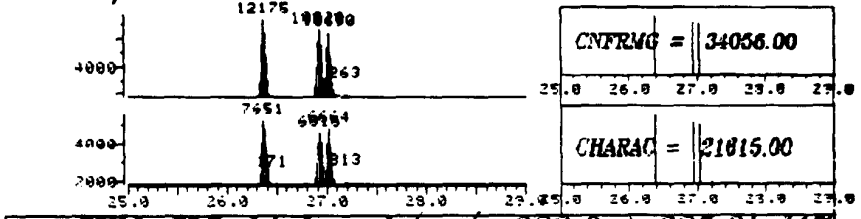
SUM-CL3-biphenyl (m/e 255.9 & 257.9) #10
GC/MS FILE = >14494



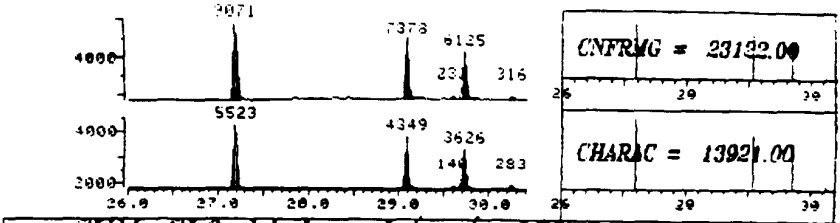
SUM-CL4-biphenyl (m/e 289.8 & 291.8) #13
GC/MS FILE = >14494



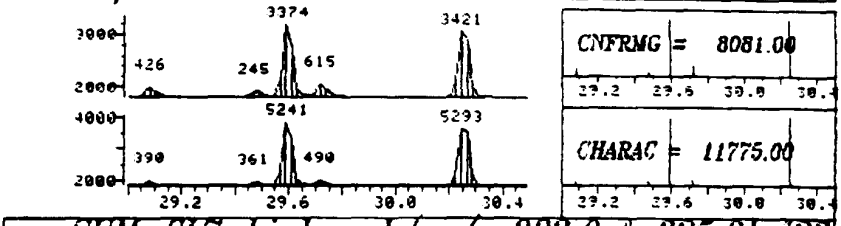
CL4-biphenyl-12C13 (301.8 and 303.8) #16
GC/MS FILE = >14494



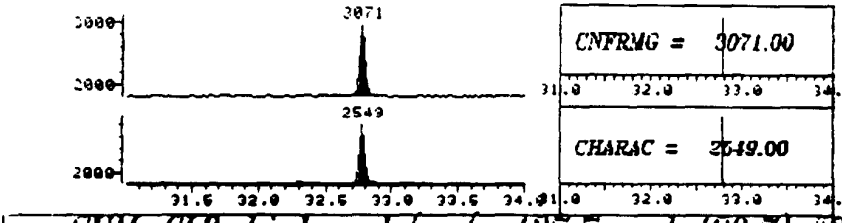
SUM-CL5-biphenyl (m/e 323.9 & 325.9) #17
GC/MS FILE = >14494



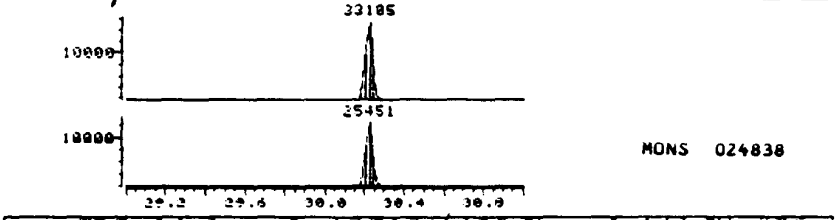
SUM-CL6-biphenyl (m/e 357.8 & 359.8) #20
GC/MS FILE = >14494



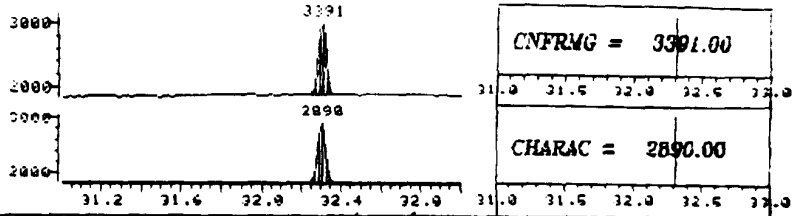
SUM-CL7-biphenyl (m/e 323.9 & 325.9) #23
GC/MS FILE = >14494



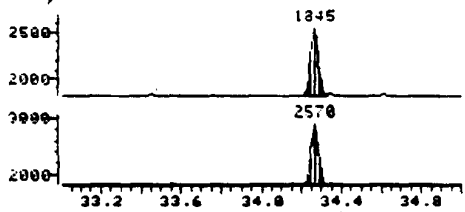
SUM-CL8-biphenyl (m/e 427.7 and 429.7) #25
GC/MS FILE = >14494



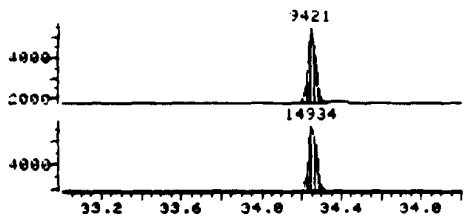
CL8-biphenyl-13C13 (439.7 and 441.7) #26
GC/MS FILE = >14494



SUM-CL9-biphenyl (m/e 461.6 and 463.6) #27
GC/MS FILE = >14494



CL10-biphenyl (m/e 497.6 and 499.6) #28
GC/MS FILE = >14494



CL10-biphenyl-12C13 (509.6 and 511.6) #29
GC/MS FILE = >14494

```
RU,WHIT,15
DU,PT4494,1
!!!
```

011					
012					
013	.785	.785	.00E+00	100.01	4
014					
015					
016	.867	.765	-.0204	113.34	4
017	.490	.772	.00E+00	63.47	5
018					
019					
020	.316	.524	.00E+00	60.21	6
021					
022					
023	.267	.183	.00E+00	145.71	7
024					
025	.0578	.0696	.00E+00	83.00	8
026	.577	.751	.00E+00	76.88	8
027	.0655	.0769	.00E+00	85.23	9
028	.0583	.0418	.00E+00	139.30	10
029	.339	.214	.00E+00	158.52	10

:RU,CRT,AL,FF

MONS 024841

Appendix F. Listing of PCB Congers Present in PCB Window Standardand Procedure Files and Examples of Computer Outputs Produced in Incidental PCB Analyses. (PTWNDW and PTOUTP Transfer Files.)

MONS 024842

Table F-1. Summary of PCB Congeners Present in the PCB Windowing Standard from Wellington Laboratories.

First-Eluting PCB Isomer	Last-Eluting PCB Isomer	#Cls
3-CL1-biphenyl	4-CL1-biphenyl	1
2,6-CL2-biphenyl	4,4'-CL2-biphenyl	2
2,4,6-CL3-biphenyl	3,4,4'-CL3-biphenyl	3
2,2',6,6'-CL4-biphenyl	3,3',4,4'-CL4-biphenyl	4
2,2',4,6,6'-CL5-biphenyl	3,3',4,4',5-CL5-biphenyl	5
2,2',4,4',6,6'-CL6-biphenyl	3,3',4,4',5,5'-CL6-biphenyl	6
2,2',3,4',5,6,6'-CL7-biphenyl	2,3,3',4,4',5,5'-CL7-biphenyl	7
2,2',3,3',5,5',6,6'-CL8-biphenyl	2,3,3',4,4',5,5',6-CL8-biphenyl	8
2,2',3,3',4,5,5',6,6'-CL9-biphenyl	2,2',3,3',4,4',5,5',6-CL9-biphenyl	9
CL10-biphenyl		10

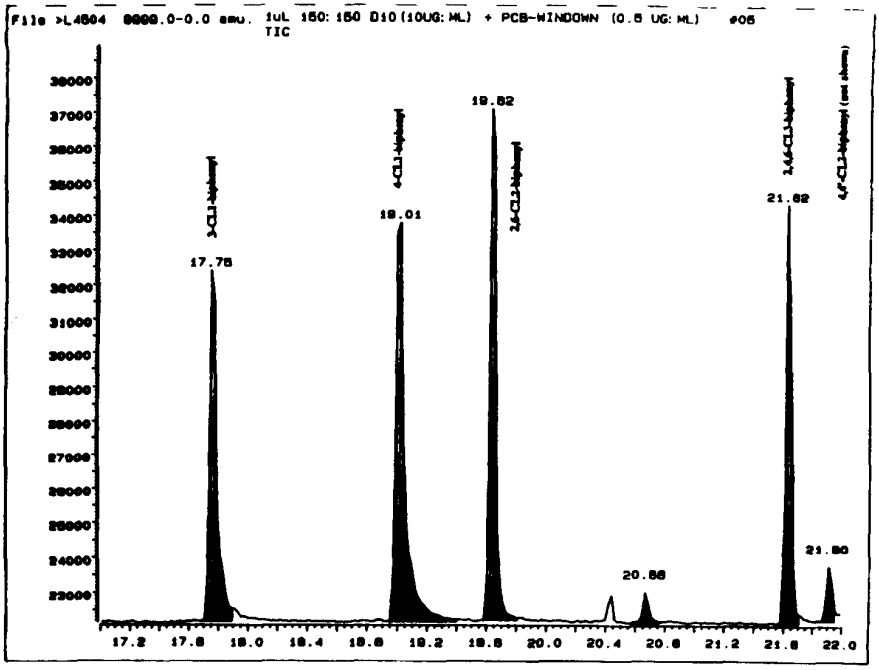


Figure F-1a. ESC's chromatogram obtained from the analysis of the Wellington Laboratories PCB Window Standard using Standard Analysis Method M8AN25, shown in Appendix A. (CL1-biphenyls through CL3-biphenyls)

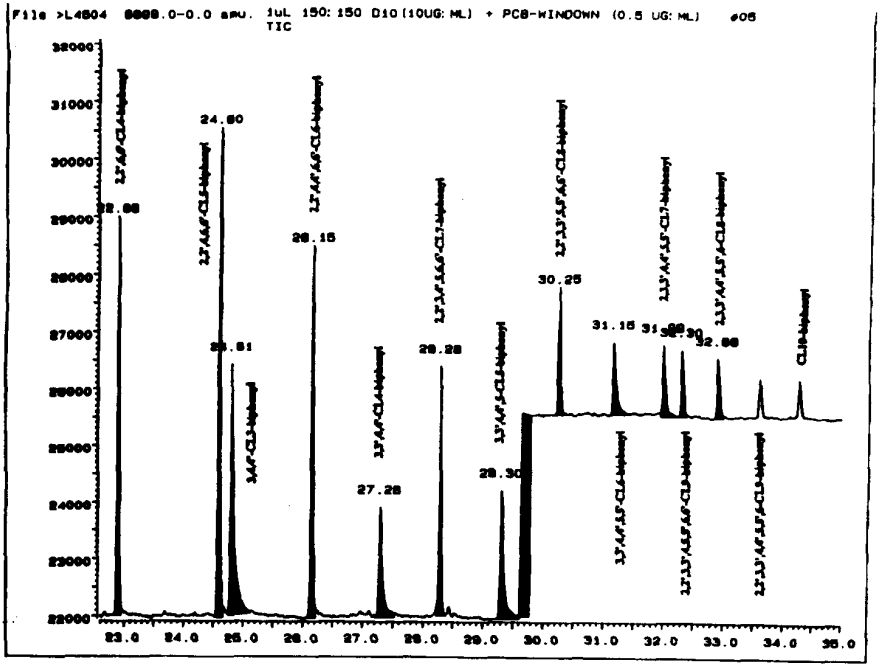


Figure F-1h. ESC's chromatogram obtained from the analysis of the Wellington Laboratories PCB Window Standard using Standard Analysis Method MRAN24 shown in Appendix A. (C13-biphenyls through C13-10-biphenyl)

```
:du,ptwndw,1
:**                                                Time Stamp - <871118.1421>
:**    :TR,PTWNDW,filename1,output_filename,nnn
:**
:**    This transfer file is designed to calculate total relative responses for
:**          compounds in samples and extracts analyzed for incidental PCBs
:**          using MS acquisition method PCSC25.
:**
:**          1G = mass spectrometric filename (>Lxxxx)
:**          2G = output_filename (PTxxxx)
:**          3G = 0 if sample not a standard, and
:**               nnn if sample is a standard.  (Note that nnn is the concen-
:**               tration of the standard in ug/mL.)
:**
:**
:**    The following R-globels are used.
:**         1R = enthracene-d10 m/e 188 area          31R = m/e 190 area
:**         2R = SUM-CL-biphenyl    m/e 188 area      32R = m/e 190 area
:**         3R = NOT USED           m/e 188 area      33R = m/e 190 area
:**         4R =     "              m/e 188 area      34R = m/e 190 area
:**         5R = 4-CL-biphenyl-6Cl3 m/e 194 area      35R = m/e 196 area
:**         6R = SUM-CL2-biphenyl   m/e 222 area      36R = m/e 224 area
:**         7R = NOT USED           m/e 222 area      37R = m/e 224 area
:**         8R =     "              m/e 222 area      38R = m/e 224 area
:**         9R =     "              m/e 222 area      39R = m/e 224 area
:**        10R = SUM-CL3-biphenyl   m/e 255.9 area    40R = m/e 257.9 area
:**        11R = NOT USED           m/e 255.9 area    41R = m/e 257.9 area
:**        12R =     "              m/e 255.9 area    42R = m/e 257.9 area
:**        13R = SUM-CL4-biphenyl   m/e 289.8 area    43R = m/e 291.8 area
:**        14R = NOT USED           m/e 289.8 area    44R = m/e 291.8 area
:**        15R =     "              m/e 289.8 area    45R = m/e 291.8 area
:**        16R = CL4-BP-12C13 Surr. m/e 301.8 area    46R = m/e 303.8 area
:**        17R = SUM-CL5-biphenyl   m/e 323.9 area    47R = m/e 325.9 area
:**        18R = NOT USED           m/e 323.9 area    48R = m/e 325.9 area
:**        19R =     "              m/e 323.9 area    49R = m/e 325.9 area
:**        20R = SUM-CL6-biphenyl   m/e 359.8 area    50R = m/e 361.8 area
:**        21R = NOT USED           m/e 359.8 area    51R = m/e 361.8 area
:**        22R =     "              m/e 359.8 area    52R = m/e 361.8 area
:**        23R = SUM-CL7-biphenyl   m/e 393.8 area    53R = m/e 393.8 area
:**        24R = NOT USED           m/e 393.8 area    54R = m/e 393.8 area
:**        25R = SUM-CL8-biphenyl   m/e 427.7 area    55R = m/e 429.7 area
:**        26R = CL8-BP-12C13 Surr. m/e 439.7 area    56R = m/e 441.7 area
:**        27R = SUM-CL9-biphenyl   m/e 461.6 area    57R = m/e 463.6 area
:**        28R = CL10-biphenyl      m/e 497.6 area    58R = m/e 499.6 area
:**        29R = CL10-BP-12C13 Surr m/e 509.6 area    59R = m/e 511.6 area
:**
:**
:SV,1,9,IH
:RU,RPN
:RU,RSE
:RU,NEW
:RU,CRT,CA,FF
:RU,MSH,1G
:RU,CRT,AL,CA,FF
:RU,TIM
:RU,STW,,1,0:50,10:30
:RU,STW,,2,0:50,30:48
:RU,STW,,3,0:50,60:080
:RU,STW,,4,0:50,80:098
:RU,STW,,5,50:75,10:030
```

MONS 024846

```
:RU,STW,,6,50:75,30:048
:RU,STW,,7,50:75,60:080
:RU,STW,,8,50:75,80:098
:RU,STW,,10,0:75,50:60
:**  This section calculates anthracene-d10 area.
:RU,FI,1G
:TR,IT*SU1,,,,188,21.5,22.4,1
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,31R
:RU,RDP,7,CHARAC =, 1R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,Anthracene-d10 (m/e = 188 and 190) #1
:RU,RDP,2,GC/MS FILE =,1G
:RU,SHO,10
:RU,DR,10,X
:** The following calculates CL1-biphenyl Areas (m/e 188 and 190)
:TR,IT*SU2,,,,188,17.5,20,2
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 32R
:RU,RDP,7,CHARAC =, 2R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,3-CL1-BP & 4-CL1-BP (m/e = 188 & 190)
:RU,RDP,2,GC/MS FILE =,1G
:RU,SHO,10
:RU,DR,10,X
:RU,GR,,1
:RU,NEW
:** The following calculates 4-CL-biphenyl-6C13 (m/e = 194 and 196)
:TR,IN*SU1,,,,194,18.4,20,5,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,4-CL-biphenyl-6C13 (194 and 196) #5
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL2-biphenyl (m/e = 222 and 224)
:TR,IT*SU2,,,,222,19,22.4,6
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 36R
:RU,RDP,7,CHARAC =, 6R
:RU,DR,10,X
:RU,STW,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,26- & 44'-CL2-BP (m/e = 222 and 224) #6
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL3-biphenyl (m/e = 255.9 and 257.9)
:TR,IT*SU1,,,,255.9,21,26,10
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,40R
:RU,RDP,7,CHARAC =,10R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
```

```
:RU,RDP,1,246- & 344'-CL3-BP (m/e 255.9 & 257.9)
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL4-biphenyl (289.8 and 291.8)
:TR,IT*SU2,,,,289.8,22.8,28,13
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 43R
:RU,RDP,7,CHARAC =,13R
:RU,DR,10,X
:RU,STV,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,22'66'- & 33'44'-CL4(m/e 289.8 & 291.8)
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL4-biphenyl-12C13 (301.8 and 303.8)
:TR,IN*SU1,,,,301.8,26,28,16,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL4-biphenyl-12C13 (301.8 and 303.8) #16
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL5-biphenyl (323.9 and 325.9)
:TR,IT*SU2,,,,323.9,24,29.4,17
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 47R
:RU,RDP,7,CHARAC =,17R
:RU,DR,10,X
:RU,STV,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,22'466'- & 33'44'5-CL5(m/e 323.9 & 325.9)
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL6-biphenyl (359.8 and 361.8)
:TR,IT*SU1,,,,359.8,25,31,20
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,50R
:RU,RDP,7,CHARAC =,20R
:RU,DR,10,X
:RU,STV,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,22'44'66'- & 33'44'55'-CL6(m/e 359.8)
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL7-biphenyl (393.8 and 395.8)
:TR,IT*SU2,,,,393.8,28,33,23
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =, 53R
:RU,RDP,7,CHARAC =,23R
:RU,DR,10,X
:RU,STV,,10,0:75,00:10
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,22'34'566'- & 233'44'55'-CL7(m/e 393.8)
```

```
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL8-biphenyl (427.7 and 429.7)
:TR,IT#SU1,,,,427.7,30,34,25
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,55R
:RU,RDP,7,CHARAC =,25R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,22'33'55'66'- & 233'44'55'6-CL8(m/e 427.7)
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL8-biphenyl-12C13 (439.7 and 441.7)
:TR,IN#SU2,,,,439.7,30,31,26,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL8-biphenyl-12C13 (439.7 and 441.7) #26
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calulcates CL9-biphenyl (461.6 and 463.6)
:TR,IT#SU1,,,,461.6,32,34,27
:RU,AR,CNFCHA,,,3,5
:RU,RDP,1,CNFRMG =,57R
:RU,RDP,7,CHARAC =,27R
:RU,DR,10,X
:RU,STW,,10,0:75,50:60
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,22'33'455'66' & 22'33'44'55'6-CL9(m/e 461.6)
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:** The following calculates CL10-biphenyl (497.6 and 499.6)
:TR,IN#SU2,,,,497.6,33,35,28,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL10-biphenyl (m/e 497.6 and 499.6) #28
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:** The following calculates CL10-biphenyl-12C13 (509.6 and 511.6)
:TR,IN#SU1,,,,509.6,33,35,29,1
:RU,AR,PCBLBL,,,3,5
:RU,RDP,1,CL10-biphenyl-12C13 (509.6 and 511.6) #29
:RU,RDP,2,GC/MS FILE =,1G
:RU,DR,10,X
:RU,SHO,10
:RU,GR,,1
:RU,NEW
:TR,PTOUTP
:RU,CRT,FF,CA
:RU,WAIT,15
:DU,2G,1
```

```
:RU,CRT,AL,FF
:**  Use :SL,6,xxxxxx:sc,WRST,6  to setup spooling output to xxxxxx:sc and 6.
:**      then, XLU,,6 to direct all RDP outputs to LU 6.
:**      then, CS,6 to close xxxxxx:sc and outspool to lu 6.
:**      Now xxxxxx:sc is ready to transfer to Lotus 1-2-3.
:TR
```

```
:du,ptoutp,1
:**    :TR,PTOUTP,filename,output_filename   Time Stamp - <871113.1407>
:**
:**    This transfer file is designed to output an ASCII file which can be used
:**          as an input file for data reduction of total incidental PCB
:**          analysis data.  The following summarize globals used:
:**
:**       1G = mass spectrometric filename (>Lxxxx)
:**       2G = output filename
:**       3G =
:**
:**
:**
:**    The following R-globals are used.
:**
:**       1R = anthracene-d10 m/e 188 area          31R = m/e 190 area
:**       2R = SUM-CL-biphenyl    m/e 188 area      32R = m/e 190 area
:**       3R = NOT USED           m/e 188 area      33R = m/e 190 area
:**       4R =     "              m/e 188 area      34R = m/e 190 area
:**       5R = 4-CL-biphenyl-6C13 m/e 194 area      35R = m/e 196 area
:**       6R = SUM-CL2-biphenyl   m/e 222 area      36R = m/e 224 area
:**       7R = NOT USED           m/e 222 area      37R = m/e 224 area
:**       8R =     "              m/e 222 area      38R = m/e 224 area
:**       9R =     "              m/e 222 area      39R = m/e 224 area
:**      10R = SUM-CL3-biphenyl   m/e 255.9 area    40R = m/e 257.9 area
:**      11R = NOT USED           m/e 255.9 area    41R = m/e 257.9 area
:**      12R =     "              m/e 255.9 area    42R = m/e 257.9 area
:**      13R = SUM-CL4-biphenyl   m/e 289.8 area    43R = m/e 291.8 area
:**      14R = NOT USED           m/e 289.8 area    44R = m/e 291.8 area
:**      15R =     "              m/e 289.8 area    45R = m/e 291.8 area
:**      16R = CL4-BP-12C13 Surr. m/e 301.8 area    46R = m/e 303.8 area
:**      17R = SUM-CL5-biphenyl   m/e 323.9 area    47R = m/e 325.9 area
:**      18R = NOT USED           m/e 323.9 area    48R = m/e 325.9 area
:**      19R =     "              m/e 323.9 area    49R = m/e 325.9 area
:**      20R = SUM-CL6-biphenyl   m/e 359.8 area    50R = m/e 361.8 area
:**      21R = NOT USED           m/e 359.8 area    51R = m/e 361.8 area
:**      22R =     "              m/e 359.8 area    52R = m/e 361.8 area
:**      23R = SUM-CL7-biphenyl   m/e 393.8 area    53R = m/e 395.8 area
:**      24R = NOT USED           m/e 393.8 area    54R = m/e 395.8 area
:**      25R = SUM-CL8-biphenyl   m/e 427.7 area    55R = m/e 429.7 area
:**      26R = CL8-BP-12C13 Surr. m/e 439.7 area    56R = m/e 441.7 area
:**      27R = SUM-CL9-biphenyl   m/e 461.6 area    57R = m/e 463.6 area
:**      28R = CL10-biphenyl      m/e 497.6 area    58R = m/e 499.6 area
:**      29R = CL10-BP-12C13 Surr m/e 509.6 area    59R = m/e 511.6 area
:**
:**
:SV,1,9,IH
:RU,RFW
:RU,NEW
:RU,FI,1G
:CR,2G::PR:4:14
:SL,6,2G,WRST,6
:RU,XLU,,6
:LL,6
:DU,PTOUT3,6
:RU,MSH,1G
:RU,RDF,,Anthracene-d10 Area = ,1R, and Std. Conc. =,3G, ug/mL.
:DU,PTOUT1,6
:** The following calculates anthracene-d10 m/e 188 RRs, etc.          MONS  024851
:RU,RSE,94:1R,95:31R
```

```
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,001,94R,95R,61R,93R
:** The following calculates SUM-CL1-biphenyl #1 (m/e 188 and 190)
:RU,RSE,94:2R,95:32R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,002,94R,95R,62R,93R,1
:** NOT USED!!The following calculates CL1-biphenyl #2 (m/e 188 and 190)
:**,RSE,94:3R,95:33R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,003
:** NOT USED!! The following calculates CL1-biphenyl #3 (m/e 188 and 190)
:**,RSE,94:4R,95:34R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,004
:** The following calculates 4-CL-biphenyl-6C13 (m/e 194 and 196)
:RU,RSE,94:5R,95:35R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,005,94R,95R,65R,93R,1
:** The following calculates SUM-CL2-biphenyl #1 (m/e 222 and 224)
:RU,RSE,94:6R,95:36R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,006,94R,95R,66R,93R,2
:** NOT USED!! The following calculates CL2-biphenyl #2 (m/e 222 and 224)
:**,RSE,94:7R,95:37R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,007
:** NOT USED!! The following calculates CL2-biphenyl #3 (m/e 222 and 224)
:**,RSE,94:8R,95:38R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,008
:** NOT USED!! The following calculates CL2-biphenyl #4 (m/e 222 and 224)
:**,RSE,94:9R,95:39R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,009
:** The following calculates SUM-CL3-biphenyl #1 (m/e 255.9 and 257.9)
:RU,RSE,94:10R,95:40R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,010,94R,95R,70R,93R,3
:** NOT USED!! The following calculates CL3-biphenyl #2 (m/e 255.9 and 257.9)
:**,RSE,94:11R,95:41R
```

```
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,011
:** NOT USED!! The following calculates CL3-biphenyl #3 (m/e 255.9 and 257.9)
:**,RSE,94:12R,95:42R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,012
:** The following calculates CL4-biphenyl #1 (m/e 289.8 and 291.8)
:RU,RSE,94:13R,95:43R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,013,94R,95R,73R,93R,4
:** NOT USED!! The following calculates CL4-biphenyl #2 (m/e 289.8 and 291.8)
:**,RSE,94:14R,95:44R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,014
:** NOT USED!! The following calculates CL4-biphenyl #3 (m/e 289.8 and 291.8)
:**,RSE,94:15R,95:45R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,015
:** The following calculates CL4-biphenyl-12C13 (m/e 301.8 and 303.8)
:RU,RSE,94:16R,95:46R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,016,94R,95R,76R,93R,4
:** The following calculates SUM-CL5-biphenyl #1 (m/e 223.9 and 225.9)
:RU,RSE,94:17R,95:47R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,017,94R,95R,77R,93R,5
:** NOT USED!! The following calculates CL5-biphenyl #2 (m/e 223.9 and 225.9)
:**,RSE,94:18R,95:48R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,018
:** NOT USED!! The following calculates CL5-biphenyl #3 (m/e 223.9 and 225.9)
:**,RSE,94:19R,95:49R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,019
:** The following calculates SUM-CL6-biphenyl #1 (m/e 359.8 and 361.8)
:RU,RSE,94:20R,95:50R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,020,94R,95R,80R,93R,6
:** NOT USED!! The following calculates CL6-biphenyl #2 (m/e 359.8 and 361.8)
:**,RSE,94:21R,95:51R
```

```
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,021
:** NOT USED!! The following calculates CL6-biphenyl #3 (m/e 359.8 and 361.8)
:**,RSE,94:22R,95:52R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,022
:** The following calculates SUM-CL7-biphenyl #1 (m/e 393.8 and 395.8)
:RU,RSE,94:23R,95:53R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,023,94R,95R,83R,93R,7
:** NOT USED!! The following calculates CL7-biphenyl #2 (m/e 393.8 and 395.8)
:**,RSE,94:24R,95:54R
:**,RCA,94,94R,DI,1R,MU,10
:**,RCA,95,95R,DI,1R,MU,10
:**,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,024
:** The following calculates SUM-CL8-biphenyl #1 (m/e 427.7 and 429.7)
:RU,RSE,94:25R,95:55R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,025,94R,95R,85R,93R,8
:** The following calculates CL8-biphenyl-12C13 (m/e 439.7 and 441.7)
:RU,RSE,94:26R,95:56R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,026,94R,95R,86R,93R,8
:** The following calculates SUM-CL9-biphenyl (m/e 461.6 and 463.6)
:RU,RSE,94:27R,95:57R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,027,94R,95R,87R,93R,9
:** The following calculates CL10-biphenyl (m/e 497.6 and 499.6)
:RU,RSE,94:28R,95:58R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,028,94R,95R,88R,93R,10
:** The following calculates CL10-biphenyl-12C13 (m/e 509.6 and 511.6)
:RU,RSE,94:29R,95:59R
:RU,RCA,94,94R,DI,1R,MU,10
:RU,RCA,95,95R,DI,1R,MU,10
:RU,RCA,93,94R,DI,95R,MU,100
:RU,RDP,,029,94R,95R,89R,93R,10
:**  Use :SL,6,xxxxxx:sc,WRST,6  to setup spooling output to xxxxxx:sc and 6.
:**      then, XLU,,6 to direct all RDP outputs to LU 6.
:**      then, CS,6 to close xxxxxx:sc and outspool to lu 6.
:**      Now xxxxxx:sc is ready to transfer to Lotus 1-2-3.
:CS,6
:LL,1                                                              MONS   024854
:RU,XLU,,1
:SV,9G,,IH
```

```
:du,it#sul,1
:**
:**     :TR,IT#SU1,,,,4G,5G,6G,7G
:**
:**         This transfer file should be treated as a subroutine to integrate
:**           one component's characteristic ion area and it's confirming
:**           ion area.  (The confirming ion is always assumed to be 2 masses
:**           above the characteristic ion.)
:**
:**         where:  4G = characteristic mass.  Later, R-global for confirming
:**                      ion area storage.  Even later, total confirming
:**                      ions area.
:**                 5G = starting retention time.
:**                 6G = ending retention time.
:**                 7G = R-global number for storage of characteristic ion
:**                      area.  Later, total characteristic ions area.
:**
:**                97R = number of peaks detected in characteristic ion chrom.
:**                98R = minimum in ion chromatograms.
:**                99R = maximum in ion chromatograms.
:**
:SV
:RU,STW,,10,50:75,60:98
:RU,RSE,4G
:RU,RCA,0,0R,AD,2
:RU,MCA,4G,0R,,,5G:6G
:RU,INT,,-100,30,,X,,CHARAC
:RU,RCA,97,1P
:CA,&,7G,+,30
:**   4G = 7G+30 (FOR CONFIRMING ION AREA STORAGE)
:**   Y = CONFIRMING ION; X = CHARACTERISTIC ION;
:**
:**  The following two steps sets 4G and 7G "R" globals to 0.00.
:RU,RCA,4G
:RU,RCA,7G
:RU,RRM,SA,98,X
:RU,RRM,BA,99,X
:RU,DR,3,X,AL,,,10720B,031463B,98R:99R
:RU,INT,,-100,,,Y,,CONFRM
:**
:**
:RU,RRM,SA,98,Y
:RU,RRM,BA,99,Y
:RU,DR,4,Y,AL,,,10720B,021462B,98R:99R
:RU,RDP,,# PEAKS =,97R
:RU,RSE,4G:0,7G:0
:DP,1P
:RU,RIF,97R,EQ,0
:DP,1P
:IF,1P,EQ,0,1
:TR
:**  JMP +1
:**  JMP +2
:RU,RCH,CONFRM
:RU,RCH,CHARAC
:RU,ADD,0.04,T
:RU,RCH,CHARAC
:RU,SUB,T,0.04,T
:RU,RCH,CHARAC
:RU,DR,7,X,5G:6G,,,11B,10421B
```

MONS 024855

```
:RU,SUM,X
:RU,RRH,8A,7G,X
:RU,DR,8,Y,5G:6G,,,11B,10421B
:RU,SUM,Y
:RU,RRH,8A,4G,Y
:TR
```

MONS 024856

```
:du,it*su2,1
:**
:**     :TR,IT*SU2,,,,4G,5G,6G,7G
:**
:**         This transfer file should be treated as a subroutine to integrate
:**           one component's characteristic ion area and it's confirming
:**           ion area.  (The confirming ion is always assumed to be 2 massas
:**           above the charscteristic ion.)
:**
:**         where:  4G = characteristic mass.  Later, R-global for confirming
:**                      ion area storage.  Even later, total confirming ions
:**                      area.
:**                 5G = starting retention time.
:**                 6G = ending retention time.
:**                 7G = R-global number for storage of characteristic ion
:**                      area.  Later, total characteristic ions area.
:**
:**                97R = number of peaks detected in characteristic ion chrom.
:**                98R = minimum in ion chromatograms.
:**                99R = maximum in ion chromatograms.
:**
:**
:**
:SV
:RU,STV,,10,50:75,10:48
:RU,RSE,4G
:RU,RCA,0,0R,AD,2
:RU,MCA,4G,0R,,,5G:6G
:RU,INT,,-100,30,,X,,CHARAC
:RU,RCA,97,1P
:CA,4,7G,+,30
:**   4G = 7G+30 (FOR CONFIRMING ION AREA STORAGE)
:**   Y = CONFIRMING ION; X = CHARACTERISTIC ION;
:**
:**  The following two steps set the 4G and 7G "R" values to 0.
:RU,RCA,4G
:RU,RCA,7G
:RU,RRM,SA,98,X
:RU,RRM,BA,99,X
:RU,DR,1,X,AL,,,10720B,031463B,98R:99R
:RU,INT,,-100,,,Y,,CONFRM
:**
:**
:RU,RRM,SA,98,Y
:RU,RRM,BA,99,Y
:RU,DR,2,Y,AL,,,10720B,021462B,98R:99R
:RU,RSE,4G:0,7G:0
:RU,RDP,,# PEAKS =,97R
:DP,1P
:RU,RIF,97R,EQ,0
:DP,1P
:IF,1P,EQ,0,1
:TR
:** JMP +1
:** JMP +2
:RU,RCH,CONFRM
:RU,RCH,CHARAC
:RU,ADD,0.04,T
:RU,RCH,CHARAC
:RU,SUB,T,0.04,T
```

MONS 024857

```
:RU,RCH,CHARAC
:RU,DR,5,X,5G:6G,,,11B,10421B
:RU,SUM,X
:RU,RRH,BA,7G,X
:RU,DR,6,Y,5G:6G,,,11B,10421B
:RU,SUM,Y
:RU,RRH,BA,4G,Y
:TR
```

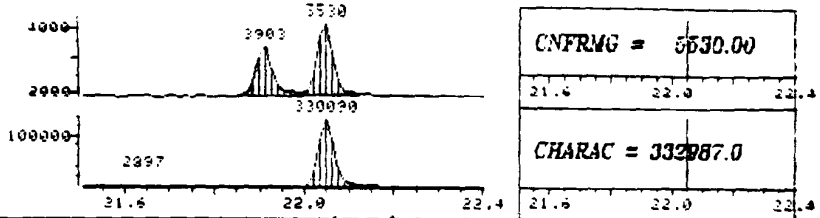
MONS 024858

MS data file header from >L4536

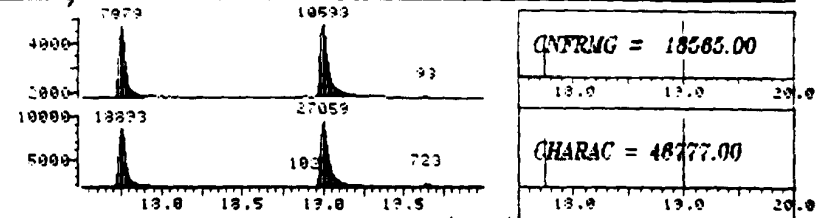
Sample: 10L 150:150 D10(10 U Operator: MASON MS2 11/14/97 13:45
Misc G:ML) + PCB-WINDOWN (0.5 UG ML) #43 BTL#43
Sys. #: 2 MS model: 87 SW/HW rev. IA ALS # ***
Method file PCSC25 Tuning file MT8602 No. of extra records 0
Source temp. 200 Analyzer temp.: 0 Transfer line temp 0

Chromatographic temperatures	0.	0.	0.	0.	0.
Chromatographic times, min.	0.0	0.0	0.0	0.0	0.0
Chromatographic rate, deg/min	0.0	0.0	0.0	0.0	0.0

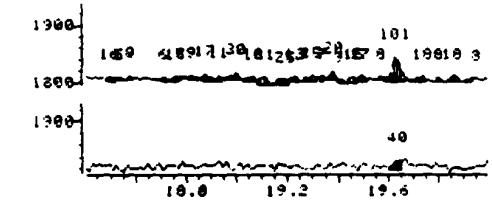
MONS 024859



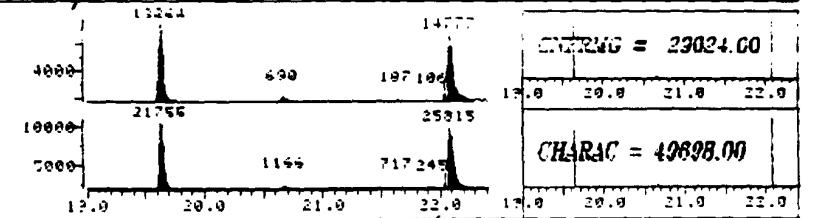
Anthracene-d10 (m/e = 188 and 190) #1
GC/MS FILE = >14536



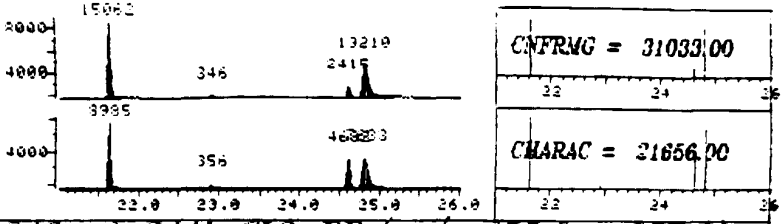
3-CL1-BP & 4-CL1-BP (m/e = 188 & 190)
GC/MS FILE = >14536



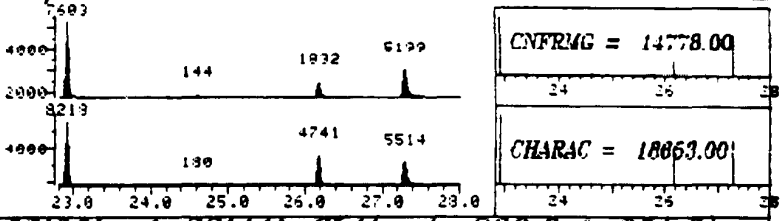
4-CL-biphenyl-6CI3 (194 and 196) #5
GC/MS FILE = >14536



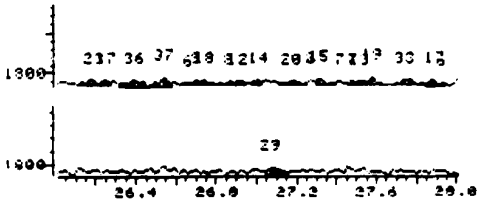
26- & 44'-CL2-BP (m/e = 222 and 224) #6
GC/MS FILE = >14536



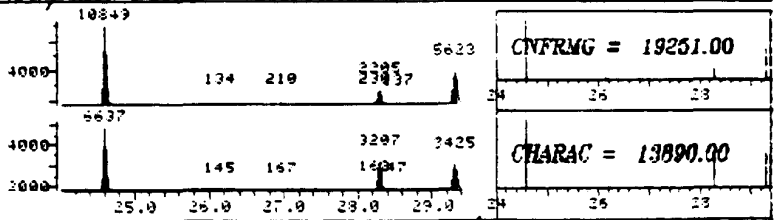
246- & 344'-CL3-BP (m/e 255.9 & 257.9)
GC/MS FILE = >L4536



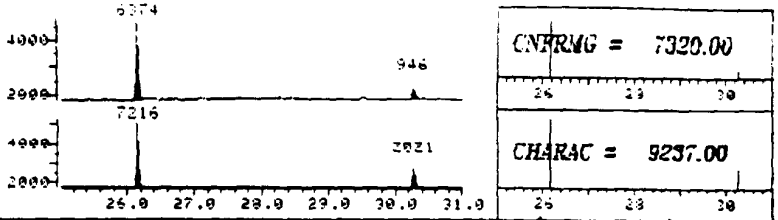
22'66'- & 33'44'-CL4(m/e 289.8 & 291.8)
GC/MS FILE = >L4536



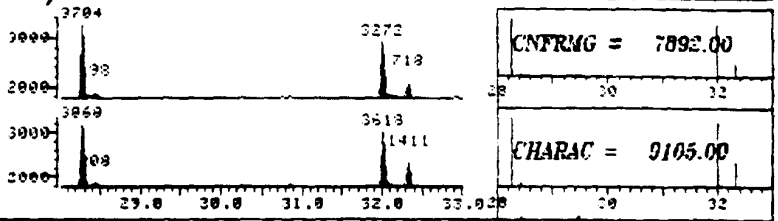
CL4-biphenyl-12C13 (301.8 and 303.8) #16
GC/MS FILE = >L4536



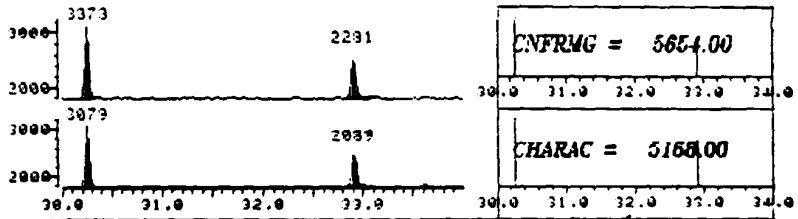
22'466'- & 33'44'5-CL5(m/e 323.9 & 325.9)
GC/MS FILE = >L4536



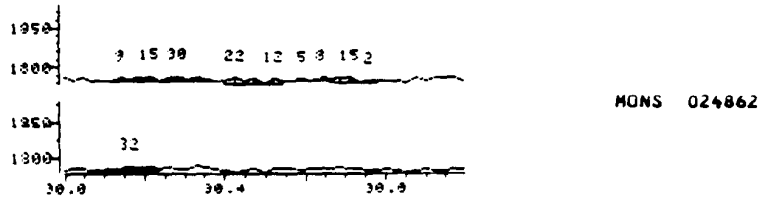
22'44'66'- & 33'44'55'-CL6(m/e 359.8)
GC/MS FILE = >L4536



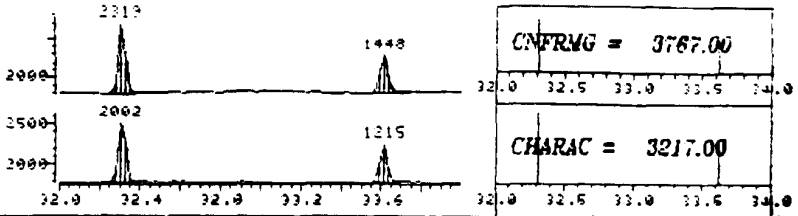
22'34'566'- & 233'44'55'-CL7(m/e 393.8)
GC/MS FILE = >L4536



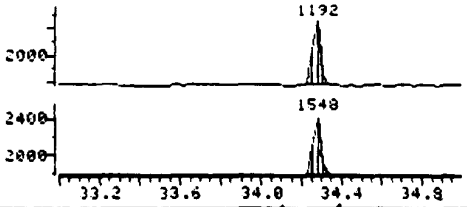
22'33'55'66'- & 233'44'55'6-CL8(m/e 427.7)
GC/MS FILE = >L4536



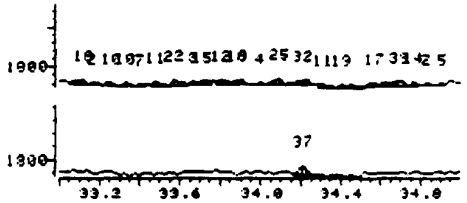
CL8-biphenyl-13C13 (439.7 and 441.7) #26
GC/MS FILE = >L4536



22'33'455'66' & 22'33'44'55'6-CL9(m/e 461.6)
GC/MS FILE = >L4536



CL10-biphenyl (m/e 497.6 and 499.6) #28
GC/MS FILE = >L4536



CL10-biphenyl-12C13 (509.6 and 511.6) #29
GC/MS FILE = >L4536

```
RU,WAIT,15
OU,P?4536,1
!!!
```

012				
013	560	144	00E+00	126.22 4
014				
015				
016	.000871	.000420	-.0613	207.14 4
017	.417	.578	.00E+00	72.15 5
018				
019				
020	.277	.220	.00E+00	126.19 6
021				
022				
023	.273	.237	.00E+00	115.37 7
024				
025	.155	.170	.00E+00	91.40 8
026	.000961	.000270	-.0238	355.56 8
027	.0966	.113	.00E+00	85.40 9
028	.0465	.0358	.00E+00	129.87 10
029	.00111	.000961	.00E+00	115.63 10

RU,CRT,AL,FF

MONS 024865

Wellington
Laboratories

P.O. Box 1261 291 Woodlawn Road West, Unit 2
Guelph, Ontario N1H 6N6
Telephone (519) 822-2436

Dear Researcher:

Enclosed is PCBMX-A, a mixture of chlorobiphenyl congeners in solution in iso-octane.

This solution/mixture is designed to be used in setting retention time 'windows' on a DB-5 (J&W) capillary column with MS detection. It contains the first (lowest retention time) and last (longest retention time) eluting isomers of each PCB congener group (monochloro- to nonachloro-) and also contains biphenyl and decachlorobiphenyl.

You will note that we have substituted (i) 3-monochlorobiphenyl for 2-monochlorobiphenyl and (ii) 2,4,6-trichlorobiphenyl for 2,2',6-trichlorobiphenyl. We have found that these pairs of compounds coelute and, unfortunately, we were unable to prepare the necessary isomers at an acceptable purity.

We trust that you will find this solution/mixture of good quality and, hopefully, helpful in your research.

Thank you for choosing our products.

B.G. Chittim
WELLINGTON LABORATORIES

MONS 024866

CHEMICAL STANDARD DOCUMENTATION SHEET

PCB WINDOW-DEFINING MIXTURE

SOLVENT: iso-octane

CONCENTRATIONS: 4.0 ug/ml of each congener (± 0.25 ug/ml)

RETENTION TIME *	COMPOUND
14.851	BIPHENYL
16.592	3-MONOCHLOROBIPHENYL♭
17.654	4-MONOCHLOROBIPHENYL
18.196	2,6-DICHLOROBIPHENYL
19.668	2,4,6-TRICHLOROBIPHENYL♦
20.245	4,4'-DICHLOROBIPHENYL
20.938	2,2',6,6'-TETRACHLOROBIPHENYL
22.341	2,2',4,6,6'-PENTACHLOROBIPHENYL
22.511	3,4,4'-TRICHLOROBIPHENYL
23.622	2,2',4,4',6,6'-HEXACHLOROBIPHENYL
24.544	3,3',4,4'-TETRACHLOROBIPHENYL
25.387	2,2',3,4',5,6,6'-HEPTACHLOROBIPHENYL
26.233	3,3',4,4',5-PENTACHLOROBIPHENYL
27.015	2,2',3,3',5,5',6,6'-OCTACHLOROBIPHENYL
27.760	3,3',4,4',5,5'-HEXACHLOROBIPHENYL
28.451	2,3,3',4,4',5,5'-HEPTACHLOROBIPHENYL
28.730	2,2',3,3',4,5,5',6,6'-NONACHLOROBIPHENYL
29.213	2,3,3',4,4',5,5',6-OCTACHLOROBIPHENYL
29.792	2,2',3,3',4,4',5,5',6-NONACHLOROBIPHENYL
30.386	2,2',3,3',4,4',5,5',6,6'-DECACHLOROBIPHENYL

*Refer to attached chromatogram

♭Substituted for 2-monochlorobiphenyl

♦Substituted for 2,2',6-trichlorobiphenyl

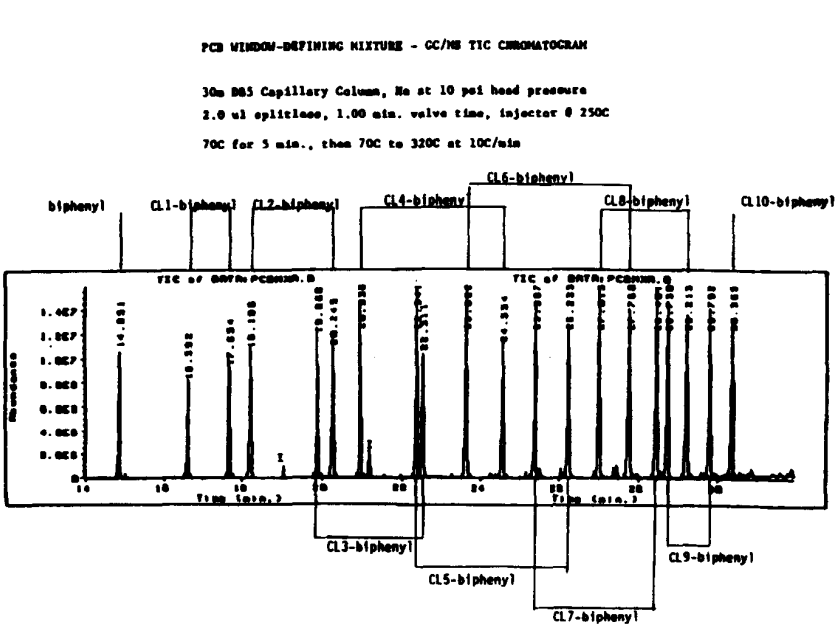
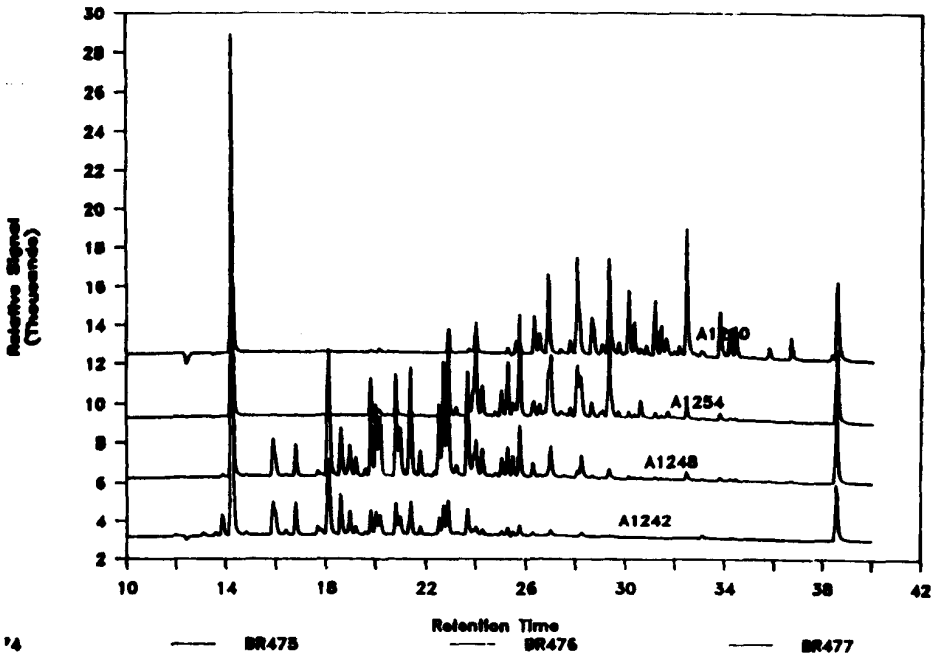


Figure F-2. Total ion chromatogram of PCB Window Standard supplied by Wellington Laboratories.

Appendix G. Chromatograms Obtained from the Comparison of DB-5 and SB-30 Capillary Columns.

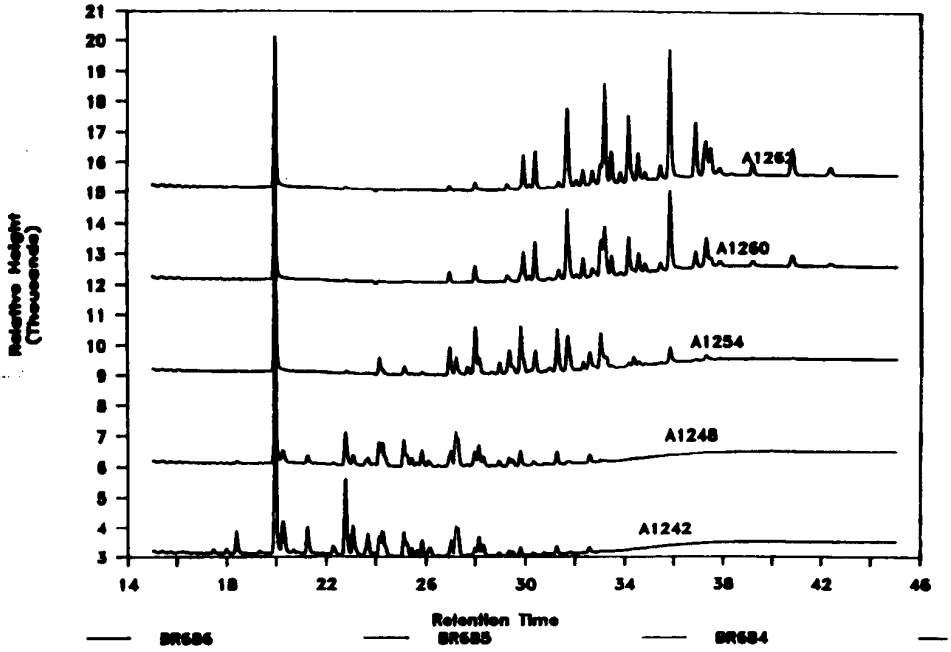
MONS 024869

FIGURE G-1. DB-5 Column Chromatographic Separations of Four Aroclor Formulations



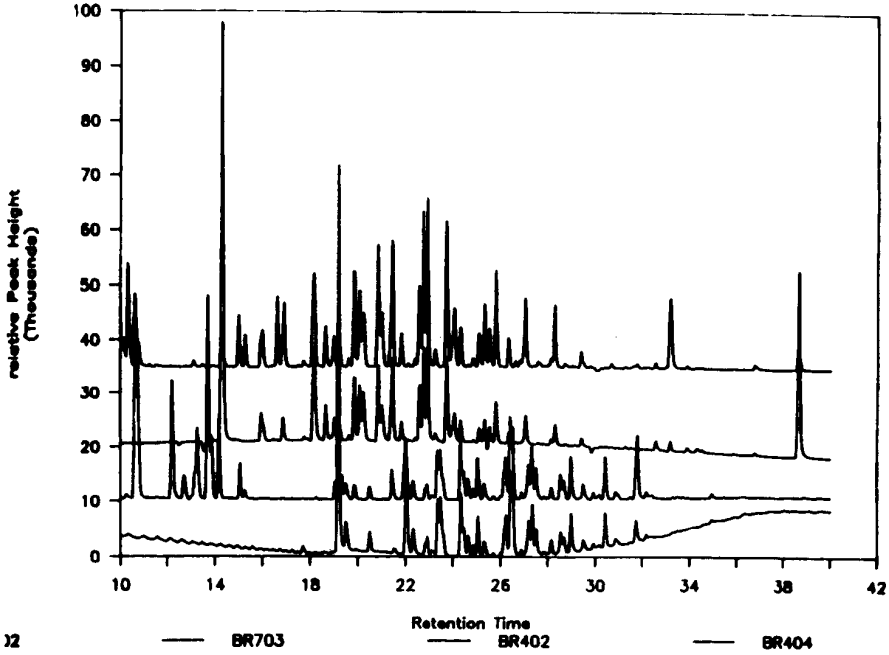
MONS 024870

FIGURE G-2. Biphenyl (SB-30) Column Chromatographic Separation of Five Aroclor Formulations



MONS 024871

FIGURE G-3. Comparison of A Sample and Aroclor 1248 on a DB-5 and SB-30 Capillary Column



MONS 024872

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

Quality Assurance/Quality Control Terminology

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

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

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

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

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

(continued)

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

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

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

(continued)

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

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

Further Information

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

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

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

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

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

QAQCTE:MM

Analyte Extraction Terminology

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

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

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

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

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

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

(continued)

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

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

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

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

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

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

(continued)

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

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

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

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

(continued)

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

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

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

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

(continued)

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

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

Further Information

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

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

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

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

ANEXTE:MH

Instrumental Analysis Terminology

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

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

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

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

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

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

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

(continued)

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

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

RRF = RR/(Analyte Concentration)

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

Analyte Extract Concentration = RR/RRF

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

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

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

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

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

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

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

(continued)

Further Information

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

INANTE:MH

Monsanto

ES&H/ESC
(Co./Div./Dept./Location

SAMPLING ANALYSIS
(Type of Report)

REPORT NO.: MSL-7431
ESC-UAG-87-179

JOB/PROJECT NO.: 760-24-710
760-24-734
760-24-733

DATE: December 21, 1987

TITLE: EVALUATION OF CLOR-N-OIL KITS FOR SCREENING OF CHLORINATED HYDROCARBONS AT SUPERFUND SITES AND FOR THE DETERMINATION OF DILUTION AMOUNTS IN GC/EC PCB ANALYSES.

AUTHORS: D. E. McKenzie and B. M. Hughes

ABSTRACT: Clor-N-Oil kits are sold by Dexsil Corporation for screening electrical transformers for PCBs. The U.S. EPA, however, requires that all PCB analyses ultimately be conducted using methods similar to EPA Methods 608, 625 or 1625. These methods use GC/EC or GC/MS instrumentation to analyze for specific components in the PCB formulations. However, ESC has (See attached Abstract)

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APPROVAL DATE: March 4, 1988

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REPT. NO.: MSL-7431 AUTHORS: D. E. McKenzie and B. M. Hughes
ESC-UAG-87-179 TITLE: Evaluation of Clor-N-Oil Kits for Screening of Chlorinated Hydrocarbons at Superfund Sites and for the Determination of Dilution Amounts in GC/EC PCB Analyses.
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ABSTRACT: (Cont.'d)

demonstrated the usefulness of these kits in two areas. These areas include uses for : 1) screening for chlorinated hydrocarbons which may be present in soil for the Brio/DOP Superfund Site; and 2) determining if oil dilution is required in gas chromatography/electron capture (GC/EC) analyses of PCBs in oils. This report is a summary of the use of the Clor-N-Oil kits for these purposes.

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Time Stamp - <871221.1119>

RESEARCH REPORT

DATE: December 24, 1987

PROJECT NO: 760.24-710
760.24-734
760.24-733

TO: D. E. Danzer (F3WB)
J. Kanyok (F3WB)
R. E. Guilliams (1200)
W. W. Varnado (G4WM)
C. E. McLaughlin (E3NF)
J. R. Craddock (A2NE)
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FROM:

Environmental Sciences Center
Monsanto Company
800 N. Lindbergh Blvd.
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Phone No: (314) 694-1464

Title: Evaluation of Clor-N-Oil Kits for Screening of Chlorinated Hydrocarbons at Superfund Sites and for the Determination of Dilution Amounts in GC/EC PCB Analyses.

ABSTRACT

Clor-N-Oil kits are sold by Dexsil Corportation for screening electrical transformers for PCBs. The US EPA, however, requires that all PCB analyses ultimately be conducted using methods similar to EPA Methods 608, 625 or 1625. These methods use GC/EC or GC/MS instrumentation to analyze for specific components in the PCB formulations. However, ESC has demonstrated the usefulness of these kits in two areas. These areas include uses for: 1) screening for chlorinated hydrocarbons which may be present in soil for the Brio/DOP Superfund Site; and 2) determining if oil dilution is required in gas chromatography/electron capture (GC/EC) analyses of PCBs in oils. This report is a summary of the use of the Clor-N-Oil kits for these purposes.

(continued)

INTRODUCTION

Screening Approach for Chlorinated Hydrocarbons at Superfund Sites

One of the important parameters of concern at the Brio/DOP site is the amount of total chlorinated hydrocarbons which may be present at a particular location. The actual excavation and cleanup of the site could be greatly simplified if almost immediate information could be obtained on this class of chemical compounds which may be present in various trenches, borings, etc. A commercially available kit, which was developed for the analysis of polychlorinated biphenyls (PCBs) in transformer fluids, was evaluated for the screening of contaminated soils at approximately 1000 ug total chlorinated hydrocarbons per gram of soil. This preliminary study showed the feasibility of this approach, and could be evaluated for the Brio/DOP Superfund Site if further studies are warranted.

During 1987, ESC was heavily involved with the study of organic compounds which are present at the Brio/DOP Superfund Site near Houston, Texas. The present Research Report summarizes portions of these studies supported with EPS funding of Project Numbers 760.24-710, 760.24-733, and 760.24-734. The total time charged to these core accounts is approximately 30 hours.

PCB in Oil Dilution Determination

PCB in oil analyses by gas chromatography with electron capture detection (GC/EC) is fairly straight-forward. Classically, the technique involves dilution of the oil in an organic solvent and injecting the diluted oil into the GC/EC. Difficulties arise, however, when dealing with a wide range of samples with a wide range of PCB concentrations. Simply diluting all oil samples to a predetermined dilution volume is not sufficient since the sample can have PCB concentrations ranging from < 1 ppm to 100%. GC/EC instrumentation is like any other instrumentation, in that there are finite working analysis conditions such as linear dynamic range, and column capacity that must be met to obtain correct analytical results. Therefore, the analyst must typically prepare dilutions of the oil ranging from 1,000,000 fold to 10 fold and begin analysis by injecting samples from the greatest dilution to the least dilution in order to find the appropriate level for analysis. This can be a very time-consuming and laborious process. Through the use of Clor-N-Oil kits during the past year, ESC has eliminated much of the guesswork in determining the appropriate dilution level for PCB in oil analyses.

EXPERIMENTAL-GENERAL

In order to fully appreciate the kit's use in these two areas, the principle of Clor-N-Oil kit operation will be discussed. The kit is based on the quantitative conversion of chlorine atoms in chlorinated hydrocarbons to chloride ions which are extracted into an aqueous solution and measured colorimetrically. Chlorine is converted to chloride ions by sodium salts formed by napthalene together with diglyme (a stabilizing ligand). The chloride ions are extracted into an aqueous buffer and reacted with a controlled (limited) amount of mercuric nitrate. Mercuric ion complexes with chloride ion. Diphenyl cabazone is then added which reacts with any non-complexed mercuric ion to form a vivid blue-purple color. A blue-purple color indicates PCBs are below a certain value, while a yellow-colorless solution indicates PCB concentrations above a predetermined kit level.

(continued)

Kits are marketed for three predetermined levels of screening (50, 100, and 500 ppm). These kits were designed for use on transformers and the PCB concentrations for these kits are calibrated assuming the PCB formulation is Aroclor 1242. Aroclor 1242 has the lowest percent chlorine in PCB formulations (42%) which are found in most PCB transformers. Therefore, an oil sample containing less than 21 ppm chlorine from Aroclor 1242 would contain less than 50 ppm total PCBs. Reagents in a 50 ppm kit are present at concentrations which will give a colorless response for 21 ppm chlorine. Since all kits require 5 mLs oil, a 50 ppm kit is actually designed to give a colorimetric response for less than 105 ug chloride ion.

Use for Screening Soils for Chlorinated Hydrocarbons.

Since the kits are designed for non-aqueous oils, chlorinated hydrocarbons in soils must first be extracted prior to using the kit. This methodology involves ultrasonic extraction of soils into a relatively non-volatile solvent such as octane or nonane and screening the extract. The advantages of extraction are three fold. The first being the exclusion of inorganic chloride ions which could be present in the soil and interfere with the screen. The second being the kit range of a 50 ppm kit is too low for useful field screening of samples when the Endangerment Assessment action level is on the order of 1000 ppm. However, this extraction step also serves as a dilution step. If a 1 g soil sample is extracted with 10 mL hydrocarbon solvent, and the extract is screened with a 100 ppm kit, the actual screening level is 210 ug/mL in the extract or 2100 ug/g organic chlorine in the soil. The third advantage of the extraction step is that water has a very low solubility in hydrocarbon solvents. This step results in the removal of water which is a major screening interference.

Clor-N-Oil kit instructions are located in Appendix A. A Typical soil and oil extraction method is given in Appendix B.

PCBs in Oils - Dilution Determination.

The same extraction protocol is used for oils as is used for soils with minor modification. The modification is 1 mL of oil is pipetted into a vial and 9.0 mL of solvent (typically benzene) is added to the oil. Oil samples are completely miscible in this solvent so the final volume is approximately 10 mL. This extraction method is listed in Appendix B.

Oils are screened by using the PCB in Oil Screening Protocol for Dilution Determination which is given in Appendix C. The Screening Protocol has been extended by the use of semi-titration and semi-back titration techniques. A negative screen can be forced positive by the addition of NaCl and a positive screen can be forced negative by the addition of mercuric nitrate. The amount added in either case can be correlated with the actual amount of PCBs present in the oil. This technique actually expands the range of each kit used so that an approximate amount of PCBs present in the oil can be estimated. It should be mentioned that the kits are not specific and any chlorinated organic compound will give positive results.

(continued)

Therefore askarel transformers, which contain on the order of 10 - 30 % dichlorobenzene, cannot be screened for low levels of PCBs since the dichlorobenzene is present at 100,000 - 300,000 ppm.

The equations listed in the ESC PCB in Oil Screening Protocol For Dilution Determination are experimentally derived. For example, assume a 100 ppm Clor-N-Oil kit is used to screen an oil diluted 1:10, and the screen results are negative. However, if the addition of four 100 uL aliquot of a 50 ppm NaCL solution changes the kit's response to test positive, the approximate PCB concentration can be calculated as:

$\{500 - (30.7 \times N)\} \times F$ = concentration (ppm)
$\{500 - (30.7 \times 4)\} \times 10$ = ppm = 3772 ppm = ug/g

This value indicates that the oil should be diluted 1:1000 prior to injection in GC/EC instrumentation. (In ESC's analytical system, the linear dynamic range is approximately 0.1 to 10 ug/g). As mentioned above, the value calculated is not specific for PCBs and the sample may actually contain other chlorinated organics. However the level for a first injection in this case would be 1:1000 versus a 1:1,000,000 with no screen (since with no screening information, it would be assumed that the sample was 100% PCBs).

It must be emphasized that the US EPA does not consider the value calculated from this screening technique to be determined by an acceptable analytical method. Therefore, this value is merely an indication of the dilution level required for GC/EC and/or GC/MS analysis methods which are acceptable to the US EPA. Typical GC/EC and GC/MS methods are given in Appendix E. GC/EC and GC/MS are the only analytical methods currently acceptable to the US EPA for PCB analyses.

SUMMARY

Clor-N-Oil kits can be used successfully for field screening of organic chlorine concentrations for superfund site remediation. The kits are relatively simple and inexpensive to use. Levels can be determined rapidly (approximately 40 samples in an hour) and inexpensively. The technique actually has some advantages over classical GC/EC or GC/MS in field testing. These advantages are: 1) results are obtained faster, and 2) more samples can be screened at a lower cost (4 - 7 dollars/kit) which can actually give better information from screening results since larger numbers of pit samples can be analyzed than either on-site GC/EC or GC/MS methods. Producing more information may be quite important when cleanup decisions are being made at a site that is not homogeneous, since analysis of one sample can actually give misleading results if extrapolated to a whole pit or trench. Another advantage of this type of screening approach is that soil screens can be performed either by a chemist or trained technician, without complex instrumentation in the field. The proposed screening method requires only a balance, an ultrasonic cleaner, solvents, pipets, and the kits.

Clor-N-Oil kits can also be used to determine dilution levels for PCB analyses. The kits can rapidly pin-point the required dilution of an oil sample prior to analysis. This can in-turn save a remarkable amount of the analyst's and instrument's time as well as produce more rapid PCB in oil analyses. These kits should only be used to determine levels of sample dilution, and should never be used to determine sample concentration without confirmatory GC/EC or GC/MS data.

(continued)

This report also contains a section located in Appendix E which describes important terminology used in Quality Assurance/Quality Control, Analyte Extraction, and Instrumental Analysis.

Final Report File No(s): RC7710

APPENDICES

Appendix A. Clor-N-Oil Kit Directions.

Appendix B. A Typical Soil and Oil Extraction Procedure.

Appendix C. ESC's PCB in Oil Screening Protocol.

Appendix D. Typical GC/EC and GC/MS Analysis Method for PCBs.

Appendix E. Important Terminology used in Quality Assurance/Quality Control, Analyte Extraction and Instrumental Analysis.

ANALYST(S): David E. McKenzie and B. Mason Hughes

ANALYST APPROVAL: 

Appendix A. Clor-N-Oil Kit Directions.

INSTRUCTIONS FOR
CLOR·N·OIL™ 100
PCB Screening Kit

A PCB screening test for transformer oil.

EACH KIT CONTAINS:

1. A polyethylene tube with a black dispensing cap containing a colorless ampule (bottom) and a gray ampule (top).
2. A polyethylene tube with a green cap containing 7 ml of buffer solution, a clear ampule (bottom) and a red-green ampule (top).
3. A polyethylene bellows type pipette.

READ CAUTION SECTION AND INFORMATION ON BACK BEFORE DOING TEST.
WEAR RUBBER GLOVES AND SAFETY GLASSES.

DIRECTIONS



STEP 1. Unscrew the dispenser cap from Tube 1 (with black cap). Using the polyethylene pipette, add exactly 5 ml (up to line) of transformer oil to the tube. Replace the cap securely.



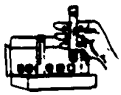
STEP 2. Break the colorless ampule (lower) in the tube by compressing the sides of the tube. Shake for 10 seconds. Break the gray ampule (top) in the tube and shake for 60 seconds. (Make sure that the colorless ampule is broken first, the gray one second.)



STEP 3. Remove the caps from both tubes and pour the buffer solution from Tube 2 (with green cap) into Tube 1. Replace the cap tightly on Tube 1 and shake vigorously for 10 seconds. Vent the tube by partially unscrewing the dispenser cap. Close securely and shake well for 10 seconds more. Vent the tube once again and tighten the cap securely. The oil should no longer appear gray.

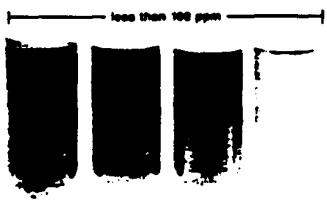


STEP 4. Stand Tube 1 upside-down on its cap and allow to settle for two minutes. If the oil layer is below the buffer layer, discontinue the test at this point as the oil is eminently pure PCB (Askarel). See photograph on back of this sheet. If the oil layer is on top of the water layer, position Tube 1 over the top of Tube 2 and open the nozzle on the black cap. Be sure to point the nozzle away from the operator while opening it, and check that the nozzle is open completely before dispensing the buffer. Dispense exactly 5 ml of the buffer solution into Tube 2 (up to line). Replace the cap on Tube 2 and close the dispenser cap on Tube 1.



STEP 5. Break the colorless ampule (bottom) in Tube 2 and shake for 10 seconds. Break the colored ampule (top), shake for 10 seconds, and observe color.

STEP 6. If the solution appears purple, the oil sample contains less than 100 ppm PCB. If it appears yellow or colorless, it MAY contain more than 100 ppm PCB and should be tested further by a PCB specific method. Disregard any color that may develop in a thin layer of oil that might form on top of the solution.



MONS 024894

DEXSIL®

DEXSIL CORPORATION
One Hamden Park Drive • Hamden, CT 06517

ABOUT THE CLOR-N-OIL TEST KIT

The Clor-N-Oil Test Kit works on the principle of chloride determination. Since PCBs contain chlorine the test kit is able to detect them. However, the test cannot distinguish between any other chlorine containing compound such as trichlorobenzene which may also be in transformer oil. This may cause a result which is known as a "false positive", i.e. the oil will indicate the presence of over 50 ppm PCBs but when analyzed by gas chromatography will show somewhat less than 50 ppm.

CHEMISTRY OF THE TEST

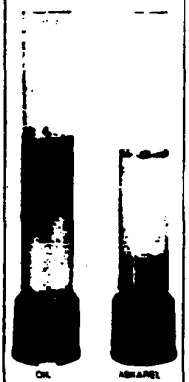
A precise amount of transformer oil is placed into Tube 1. The colorless ampule containing a catalyst is broken and thoroughly mixed with the oil. A second ampule containing metallic sodium is broken and the sodium, activated by the catalyst, strips chlorine from the PCBs forming sodium chloride. A buffer solution in water is added to the oil which neutralizes the excess sodium and extracts the sodium chloride into the water. The water layer is then separated from the oil and returned to Tube 2.

An ampule containing a precise amount of reagent is broken and mixed with the water. An indicator ampule is then broken and mixed. The color of the mixture is dependent on the amount of PCBs (chlorine) in the transformer oil sample.

SUGGESTIONS FOR USING THE CLOR-N-OIL PCB TEST KIT

- The test works on the principle of chloride detection. Therefore, contamination by salt (sodium chloride), sea water, perspiration, etc. will give a false positive result and require further testing in a laboratory.
- Never touch the ampules, the holder inside the tube, or the pipette tip, as salt may contaminate the test.
- The kit should be examined upon opening to see that all of the components are present and that all the ampules (4) are in place and not leaking. The liquid in Tube 2 (white cap) should be approximately 1/2 inch above the 5 ml line and the tube should not be leaking. The ampules are not supposed to be completely full.
- The Clor-N-Oil test will not work on a sample that contains water. If, in Step 2, the tube gets noticeably warm, builds up pressure, or takes on gray color, the sample probably contains water and the test should not be run. Another test may be tried if the oil sample is dried first.
- Perform the test in a warm, dry area with adequate light. In cold weather, a truck cab is sufficient. If a warm area is not available, Step 4 should be performed while warming Tube 1 in palm of hand.
- When drawing oil into the pipette, do not submerge tip too deeply into the oil sample. This will cause the pipette to drip.
- When inserting the pipette into the polyethylene tube, insert it all the way to the 5 ml line. This prevents oil from getting on the tube walls and reagent holder and allowing too much oil in the tube.
- Always crush the clear ampule in each tube first. If this has not been done, stop the test and start over using another complete kit. A false negative may result and allow a contaminated sample to pass without detection.
- In Step 3, tip Tube 2 to an angle of only about 45°. This will prevent the holder from sliding out.

STEP 4



OIL — ASKAREL

CONTINUE — STOP TEST

SPECIAL INSTRUCTIONS FOR ASKAREL FILLED TRANSFORMERS

- In Step 4, if the oil layer goes to the bottom (as shown in the photograph above), discontinue test at this point as the oil is nearly pure PCB (Askarel). Continuing the test further will transfer the oil into Tube 2 and leave the water layer behind, causing false results.
- This test is intended for use only with transformer oil of petroleum origin. It may not be useful for other fluids.

CAUTION

- When crushing the glass ampules, press firmly in the center of the glass ampule ONCE. Never attempt to recrush broken glass in test tube as the glass may come through the plastic and cut fingers.
- In case of accidental breakage onto skin or clothing, wash with large amounts of water. All the ampules are poisonous and should not be taken internally.
- Do not ship kits on passenger aircraft.
- Dispose of kits properly. Treat used kits as PCB waste.

The development work for this kit was sponsored by Electric Power Research Institute, Palo Alto, California and carried out by General Electric Company, Pittsfield, Massachusetts and Dexsil Corporation, Hamden, Connecticut.

MANUFACTURERS WARRANTY

This kit is warranted to be free of defects in material and workmanship until the expiration date stamped on the box. Manufacturer's sole and exclusive liability under this warranty shall be limited to replacement of any kit that is proved to be defective. Manufacturer shall not be liable for any incidental or consequential damages.

Reliable test results are highly dependent upon the care with which the directions are followed and, consequently, cannot be guaranteed.

This kit was manufactured by DEXSIL® Corporation
One Hamden Park Drive, Hamden, Connecticut 06517 (203) 288-3509 Telex 560210

MONS 024895

Appendix B. A Typical Soil and Oil Extraction Procedure.

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

ESC Standard Extraction Method No.
PCEX02

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

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

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

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

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

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

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

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

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

FLOW SHEET PCFS02

FOR ESC EXTRACTION METHOD PCEX02

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

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

EXTRACTION DATE and TIME: .....

SAMPLE COLOR AND DESCRIPTION .....

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

EXTRACT NO: .....

DATE/TIME COMPLETED .....

ANALYST: .....

ESC JOB NO: ..... MONS 024898

Appendix C. ESC's PCB in Oil Screening Protocol.

ESC PCB In Oil Screening

Protocol for Dilution Determination

Project # _____ Analyst/ Date _____

Add 3-4 mL hexane to Chlor-N-oil kit tube A (Black capped tube). [ ]

Add one mL sample extract to tube A. [ ]

Perform test as directed in Chlor-N-oil kit. Indicate if test is positive or negative in table below. [ ]

Indicate Kit type, below next to extract numbers.

Test Negative. [ ]

Add 50 ppm NaCl solution in 100 mL aliquots and indicate the number of aliquots taken until test reads positive in the table below. [ ]

[ ] Test positive.

[ ] Add 500 ppm Hg(NO3)2 solution until test is negative. Indicate the number of aliquots in the table below.

Calculate approximate PCB concentration of the sample using the equations listed below, where N is the number of 100 mL used and F is the final volume of the extract. [ ]

Kit type	For negative test (NaCl addition)	For Positive test (Hg(NO3)2 addition)
50	(250 - (30.7 x N)) x F = ppm	(250 + (17.24 x N)) x F = ppm
100	(500 - (30.7 x N)) x F = ppm	(500 + (17.24 x N)) x F = ppm
500	(2500 - (30.7 x N)) x F = ppm	(2500 + (17.24 x N)) x F = ppm

Extract number	Final volume (F)	Type kit used	result positive or negative (+ or -)	# 100 uL aliquots (N)	approx. conc. (ppm)

MONS 024900

Appendix D. Typical GC/EC and GC/MS Analysis Methods For PCBs

ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No

TITLE: Aroclor Formulations in Solvents — ECAN05

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5880 Capillary GC/EC interfaced to an HP-3357-LAS Laboratory Data System

---

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

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl through octachlorobiphenyl congeners are potential interferences. Exact retention times of most likely PCB congeners can be obtained from the analysis of Aroclors 1248, 1254, and 1260

CHROMATOGRAPHIC COLUMN: 30 m fused silica J&W DB-5 (0.25 uM) with (0.32mm ID)

COLUMN TEMPERATURE PROGRAM: 130(1)/4/300 — INJECTION TYPE: Splitless

---

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

CALCULATIONS: Internal Standard Method

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

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

External Standard Method

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

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

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

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual

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DATE OF METHOD CREATION: 10/25/83, 7/20/87 — Time Stamp - <870807.1606>

FLOW SHEET ECFS05
FOR ESC ANALYSIS METHOD ECAN05

EXTRACT ID: ---------- ESC EXTRACT # ----------

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

EC LOGBOOK PAGE NUMBER: ----------

EXTRACT COLOR AND DESCRIPTION ----------

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

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

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

DETERMINE WHETHER TO USE INTERNAL OR EXTERNAL QUANTITATION METHODOLOGY. INTERNAL QUANTITATION IS PREFERED.

[ ] BUT CAN NOT ALWAYS BE USED DUE TO INTERFERENCES.

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

INTERNAL STANDARD CALCULATION

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

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

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

EXTERNAL STANDARD CALCULATION

Aroclor Concentration = (congener area)/(RF)

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

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

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

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

MONS 024903

ESC Standard Analysis Method (SAM) ESC Standard Analysis Method No.
TITLE: Polychlorinated Compounds in Solvents MSAN25

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

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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

---

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-6C13 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-12C13 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-12C13 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-12C13 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 - <871123.1532>

(continued)

Supplemental Information to Standard Analysis Method MSAN35

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
2,5-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
decachlorobiphanyl

(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 AUTO-
SAMPLER VIAL THE SAMPLE EX-
TRACT OR STANDARD SOLUTION
CONTAINING PCB CONGENERS
WITH 150 uL INTERNAL [ ]
STANDARD SOLUTION.

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

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

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

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

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

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

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

$$\text{Total PCB Concentration} = \text{CL1-BP Concentration} + \text{CL2-BP Concentration} + \text{CL3-BP Concentration} + \ldots + \text{CL10-BP Concentration}$$

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

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

Appendix E. 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 ESG/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 proceedures are required than are included in the present study.

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

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

(continued)

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

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

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

(continued)

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

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

Further Information

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

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

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

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

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

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Analyte Extraction Terminology

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

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

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

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

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

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

(continued)

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

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

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

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

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

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

(continued)

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

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

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

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

(continued)

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

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

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

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

(continued)

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

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

Further Information

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

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

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

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

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Instrumental Analysis Terminology

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

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

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

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

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

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

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

(continued)

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

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

RRF = RR/(Analyte Concentration)

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

Analyte Extract Concentration = RR/RRF

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

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

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

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

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

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

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

(continued)

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

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

Analyte Extract Concentration = (Analyte Area)/RF

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

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

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

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

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

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

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

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

(continued)

Further Information

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

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