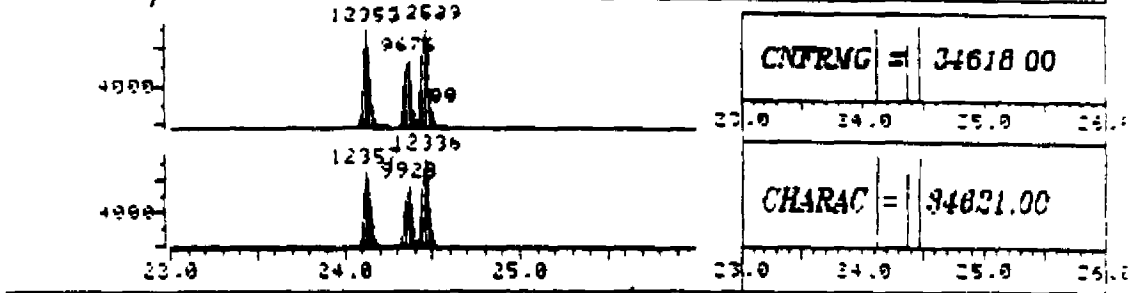
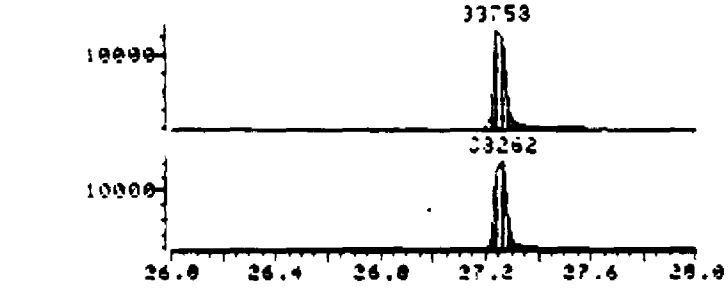


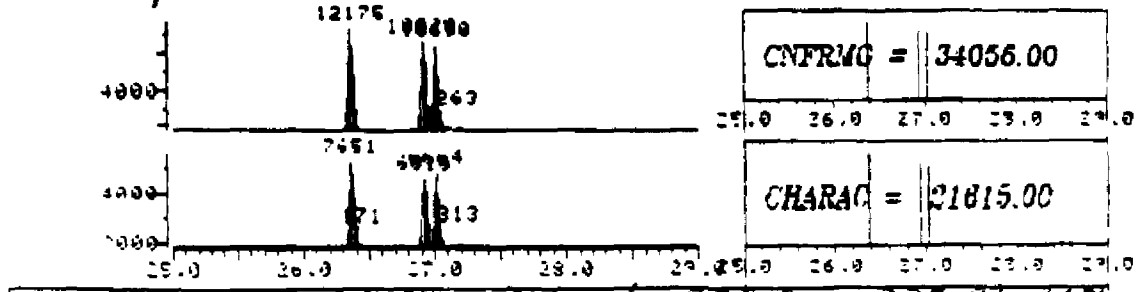
SUM-CL3-biphenyl (m/e 255.9 & 257.9) #10
GC/MS FILE = >14494



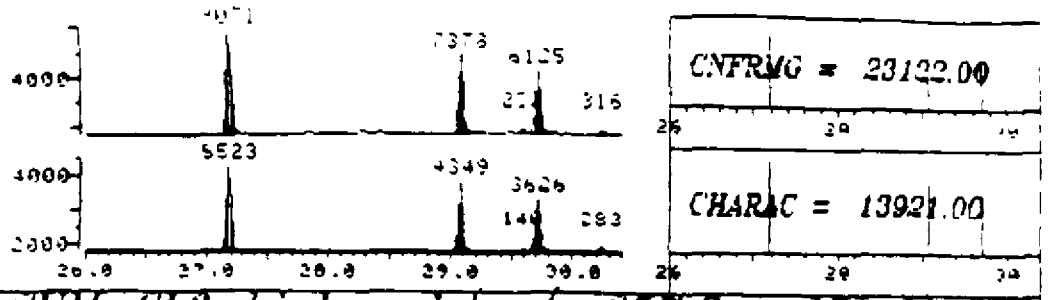
SUM-CL4-biphenyl (m/e 289.8 & 291.8) #13
GC/MS FILE = >14494



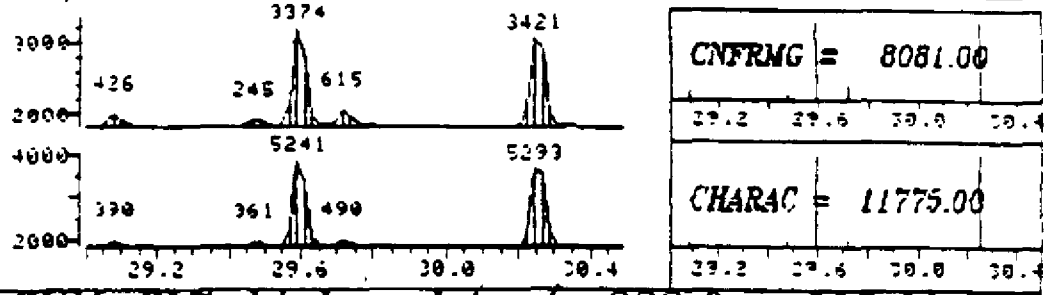
CL4-biphenyl-13C13 (301.8 and 303.8) #16
GC/MS FILE = >14494



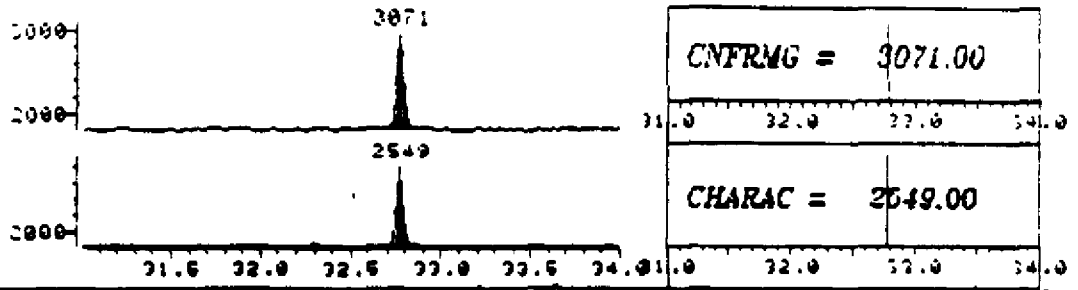
SUM-CL5-biphenyl (m/e 323.9 & 325.9) #17
GC/MS FILE = >14494



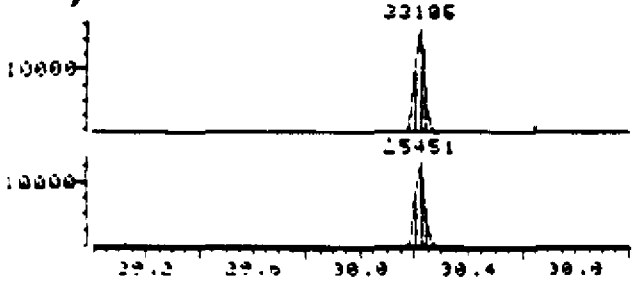
SUM-CL6-biphenyl (m/e 357.8 & 359.8) #20
GC/MS FILE = >14494



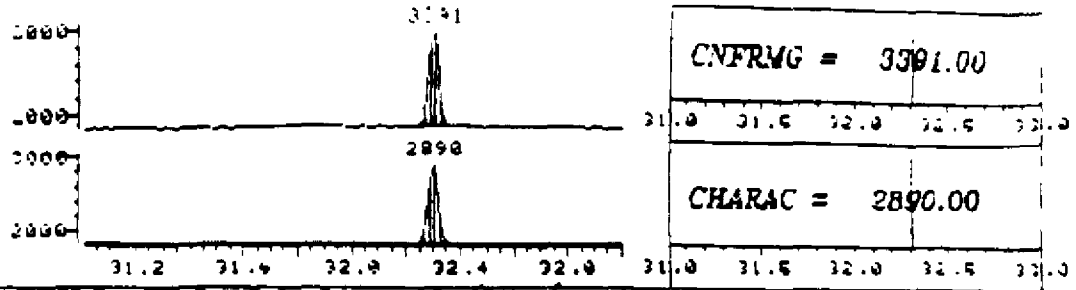
SUM-CL7-biphenyl (m/e 323.9 & 325.9) #23
GC/MS FILE = >14494



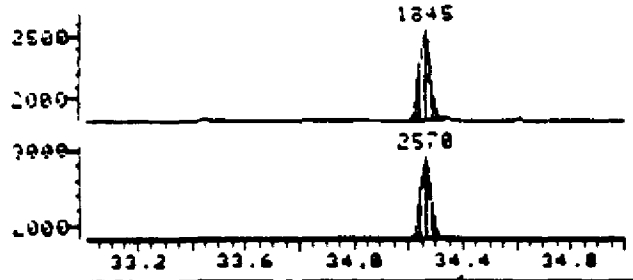
SUM-CL8-biphenyl (m/e 427.7 and 429.7) #25
GC/MS FILE = >14494



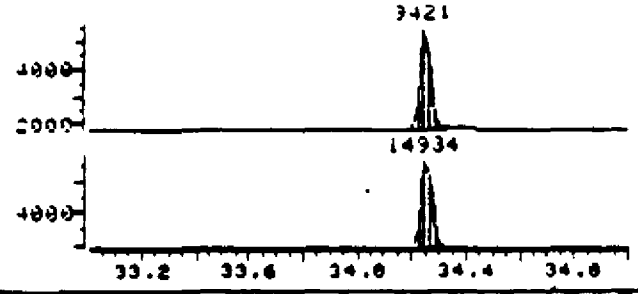
CL8-biphenyl-13C13 (439.7 and 441.7) #26
GC/MS FILE = >14494



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

```
#0,WHITE,15
D0,PT4494,,
!!!
```

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	03	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

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 221200

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

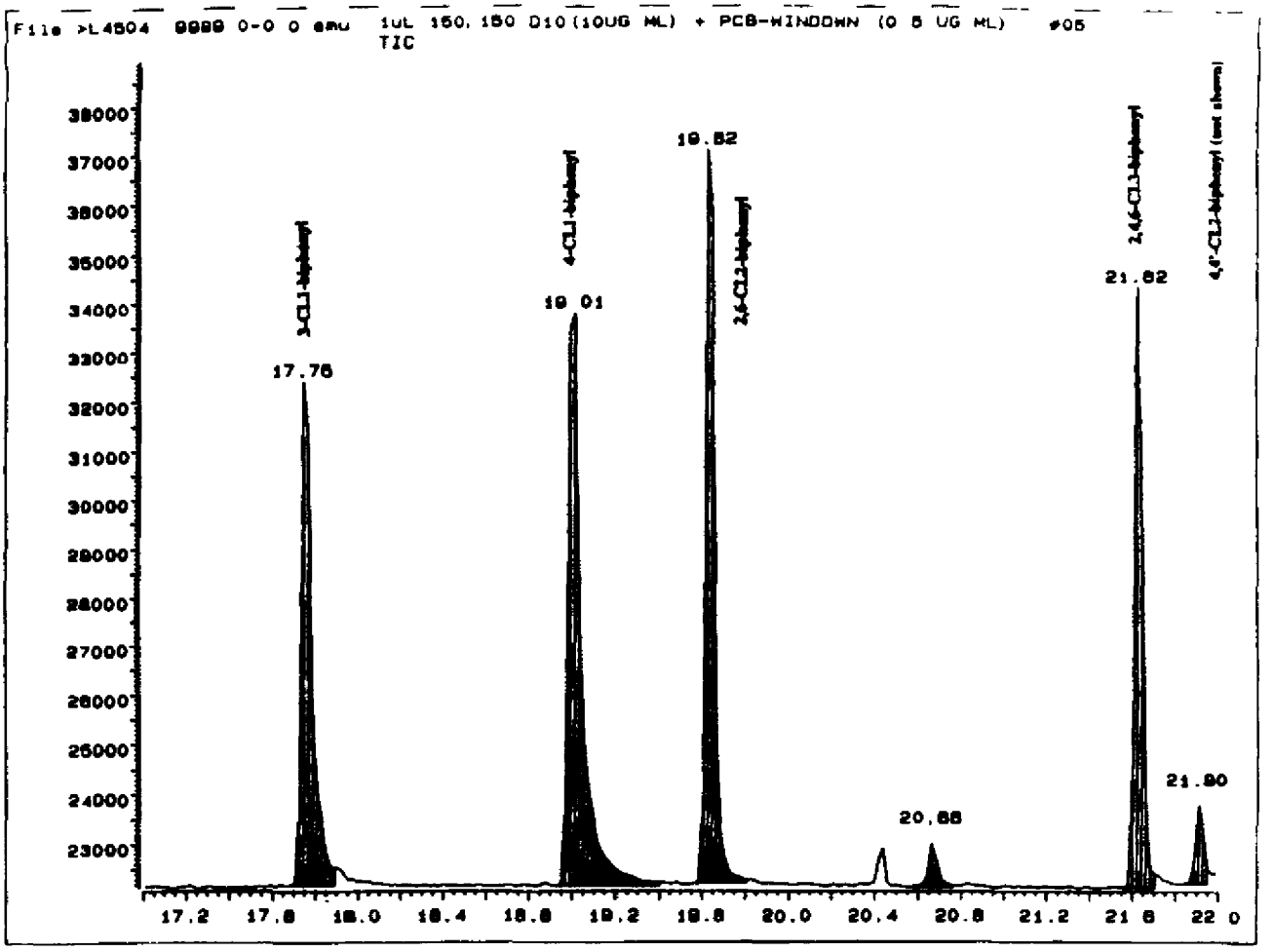


Figure F-1a. ESC's chromatogram obtained from the analysis of the Wellington Laboratories PCB Window Standard using Standard Analysis Method MSAN25, shown in Appendix A. (CL1-biphenyls through CL3-biphenyls)

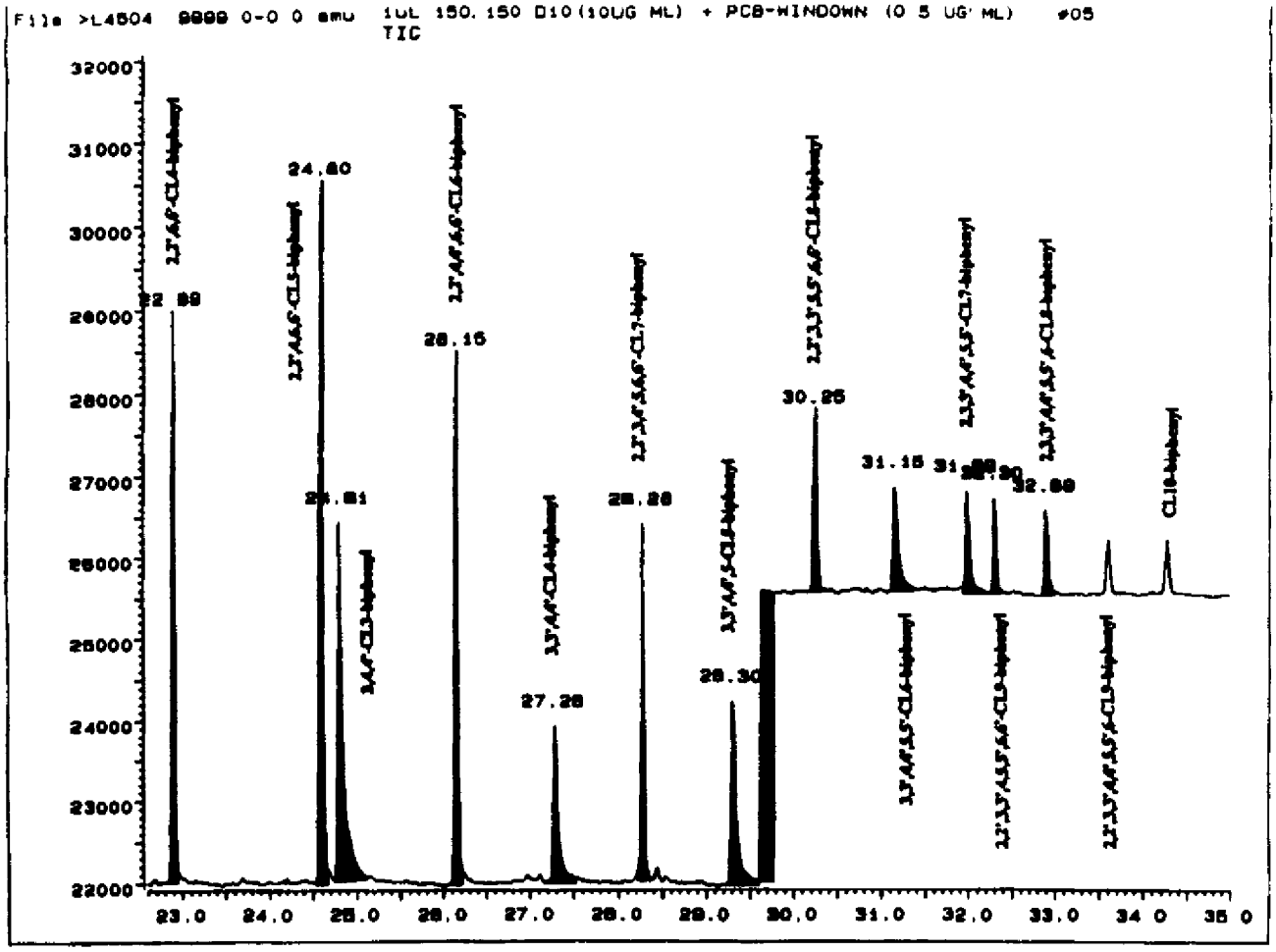


Figure F-1b. ESC's chromatogram obtained from the analysis of the Wellington Laboratories PCB Window Standard using Standard Analysis Method MSAN25 shown in Appendix A (CL Biphenyls through CL Biphenyl)

```
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-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
**
**
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 221204

```
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-6Cl3 (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-6Cl3 (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
```

MONS 221205

```
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,STW,,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,STW,,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,STW,,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,STW,,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
```

MONS 221207

```
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
```

MONS 221208

```
du,ptoutp,1
**    TR,PTOUTP,filename,output_filaname   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 summariza globals used·
**
**       1G = mass spactrometric 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,RPN
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 221209

```
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-6Cl3 (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
RU,XLU,,1                                                    MONS  221212
SV,9G,,IH
```

```
du,it#sul,I
**
**     .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,MC4,4G,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 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                                          MONS  221213
·RU,DR,7,X,5G:6G,,,11B,10421B
```

```
.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
```

MONS 221214

```
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 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,MC4,4G,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,CHARAG
:RU,ADD,0.04,T
·RU,RCH,CHARAC
·RU,SUB,T,0.04,T                                          MONS  221215
```

```
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
```

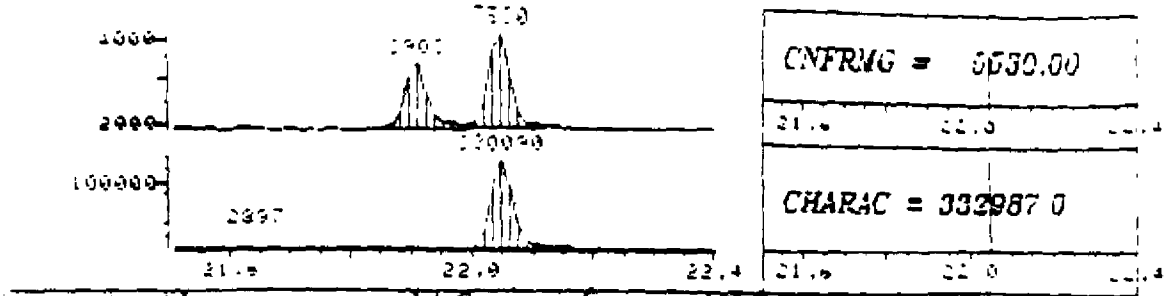
MONS 221216

(* data file header from /L4536

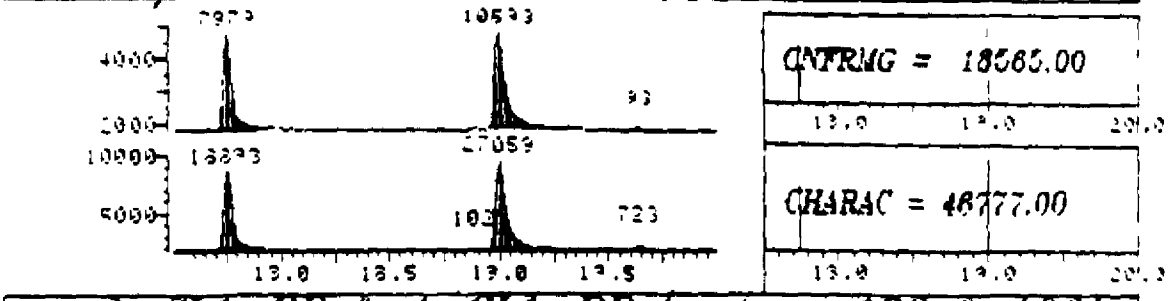
Sample 10L 150 150 010\10 U Operator MASON MS2 11/11 87 13 13
Misc G ML) + PCD-WINDOWN (0 5 UG ML) #43 BTL#43
Sys # 2 MS model 87 SW/HW rev IA ALS # ***
Method file PCSC25 Tuning file MIB602 No of extra records 4
Source temp 200 Analyzer temp 0 Transfer line temp 0

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

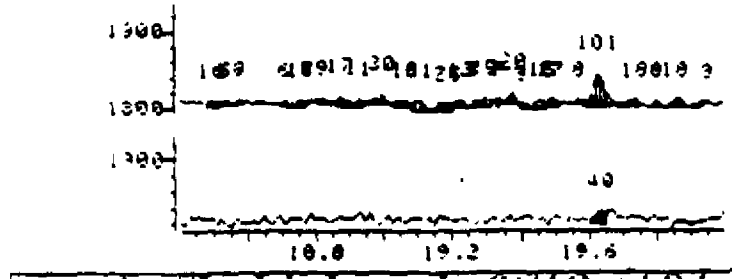
MONS 221217



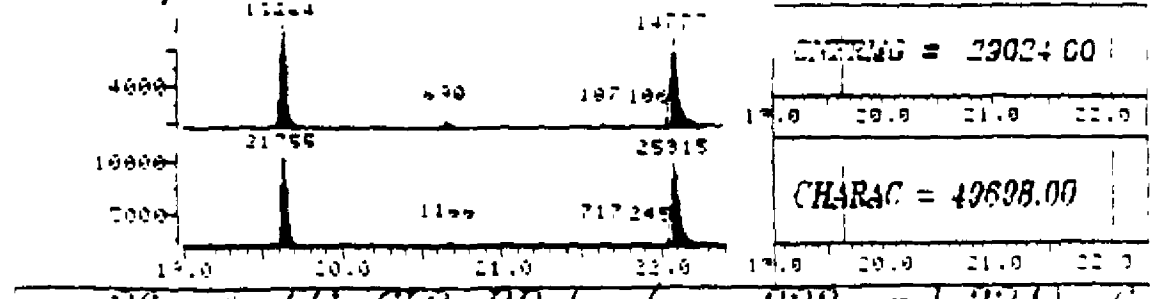
Anthracene-d10 (m/e = 188 and 190) #1
GC/MS FILE = 214536



3-CLI-BP & 4-CLI-BP (m/e = 188 & 190)
GC/MS FILE = 214536

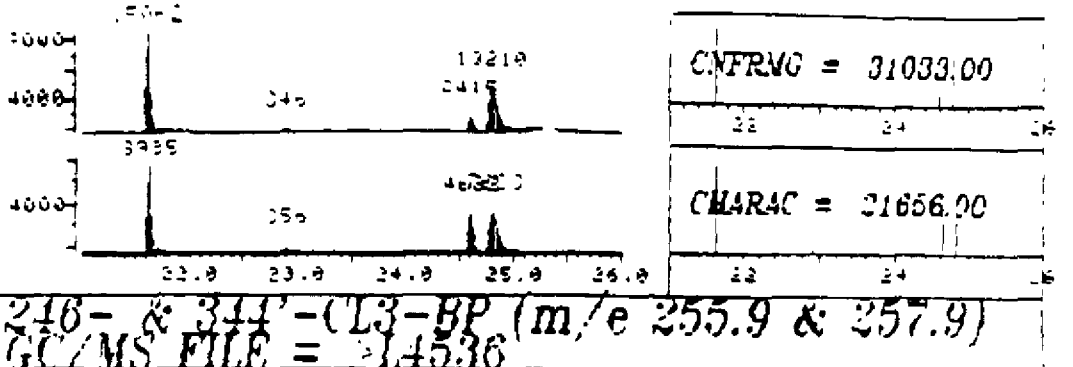


4-CL-biphenyl-6C13 (194 and 196) #5
GC/MS FILE = 214536

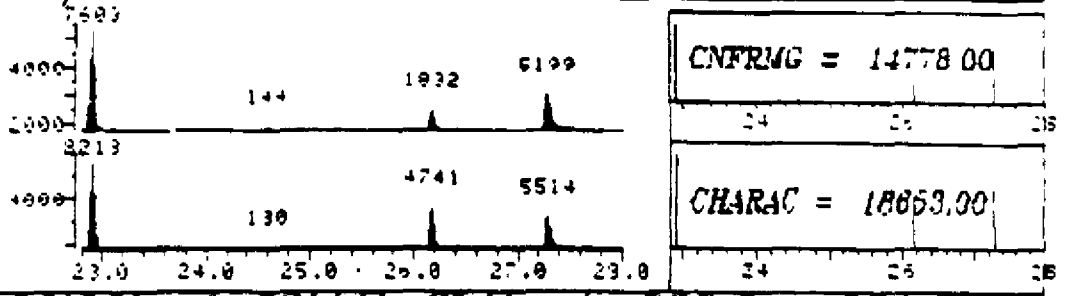


26- & 44-CL2-BP (m/e = 222 and 224) #6
GC/MS FILE = 214536

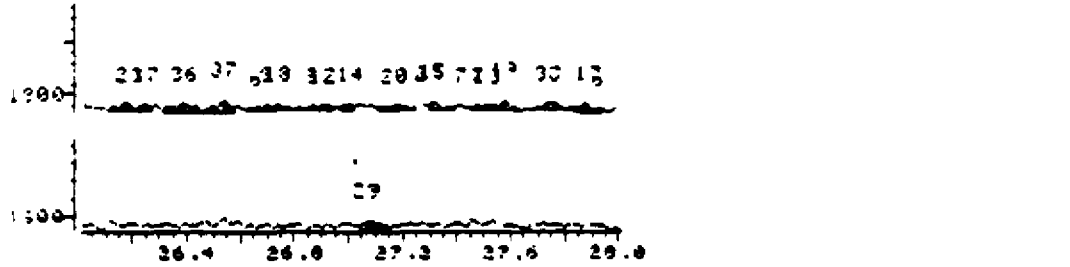
MONS 221218



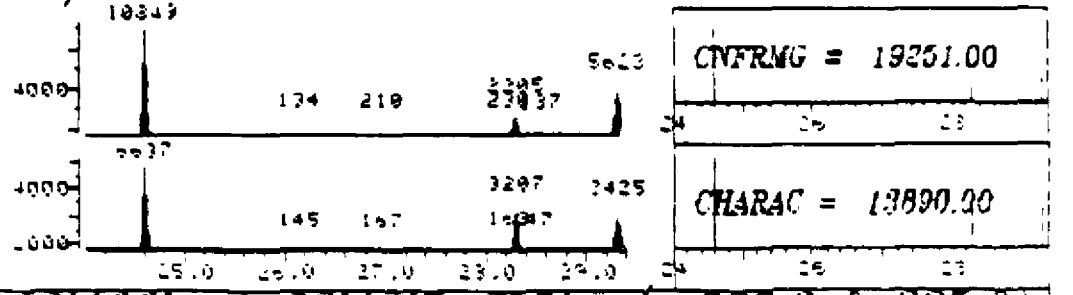
2,6- & 3,4'-CL3-BP (m/e 255.9 & 257.9)
GC/MS FILE = 14536



2,2',6,6'- & 3,3',4,4'-CL4 (m/e 289.8 & 291.8)
GC/MS FILE = 14536

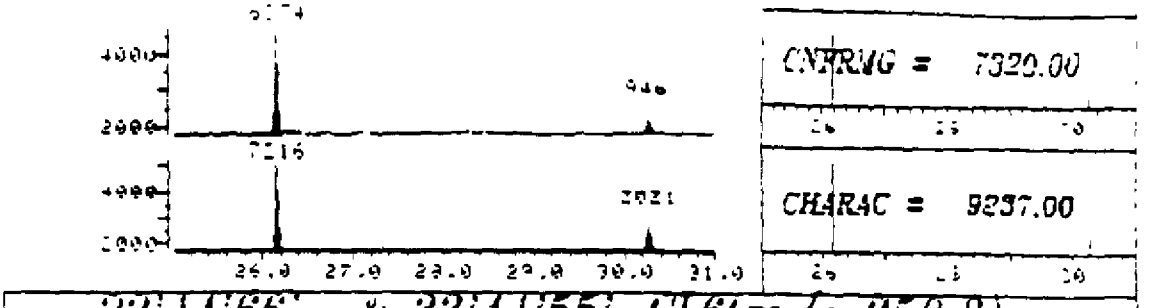


CL4-biphenyl-12C13 (301.8 and 303.8) #16
GC/MS FILE = 14536

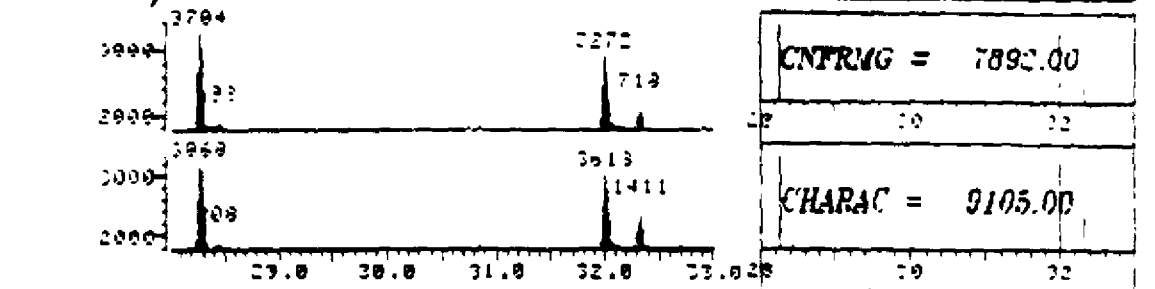


2,2',4,6,6'- & 3,3',4,4',5-CL5 (m/e 323.9 & 325.9)
GC/MS FILE = 14536

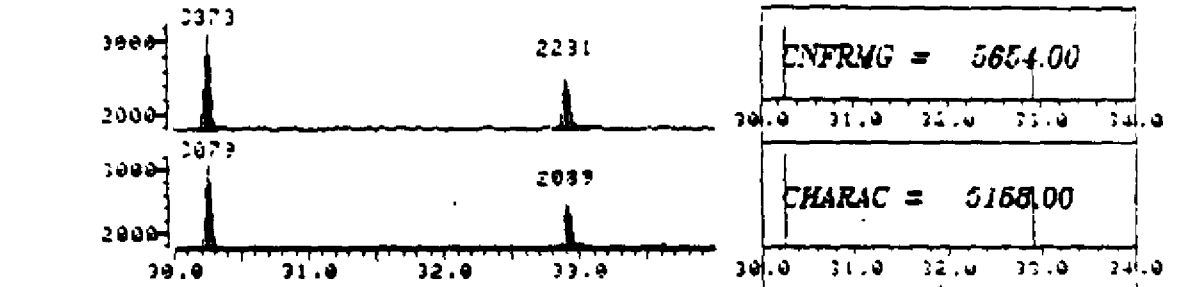
MONS 221219



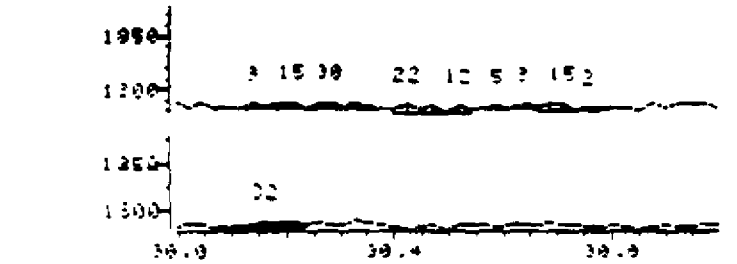
22'44'66'- & 33'44'55'-CL6(m/e 359.8)
GC/MS FILE = L4536



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

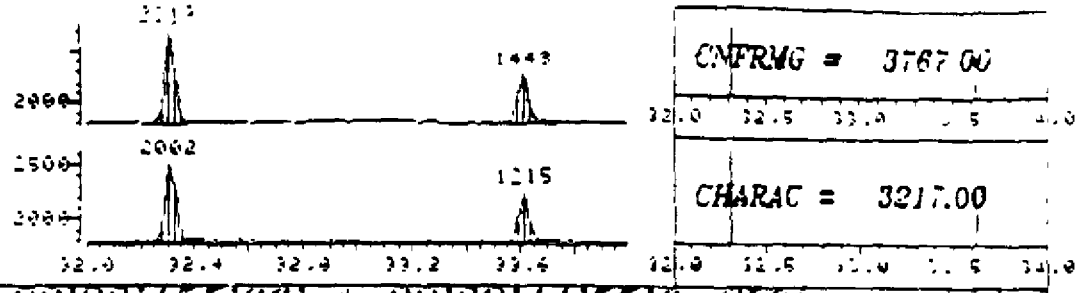


22'33'55'66'- & 233'44'55'6-CL8(m/e 427.7)
GC/MS FILE = L4536

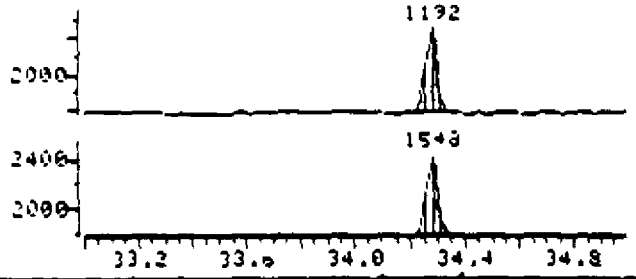


CL8-biphenyl-13C13 (439.7 and 441.7) =26
GC/MS FILE = L4536

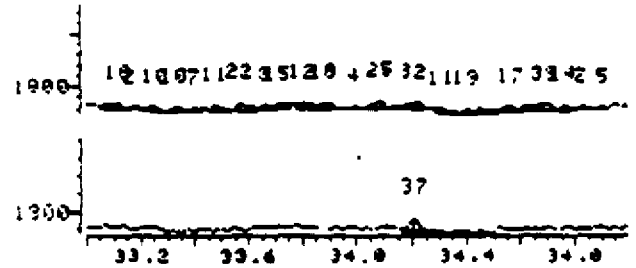
MONS 221220



22'33'55'66' & 22'33'44'55'6-CL9(m/e 461.6)
GC/MS FILE = >14536



CL10-biphenyl (m/e 497.6 and 499.6) #28
GC/MS FILE = >14536



CL10-biphenyl-12C13 (509.6 and 511.6) #29
GC/MS FILE = >14536

MONS 221221

```
20,WAIT,15
50,F24536,1
!!!
```

012						
013	560	144	00E+00	126	22	4
014						
015						
016	000071	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	0960	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 221223

Wellington
Laboratories

PO 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 221224

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
19.868	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
24.644	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',6,6',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
30.365	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

MONS 221225

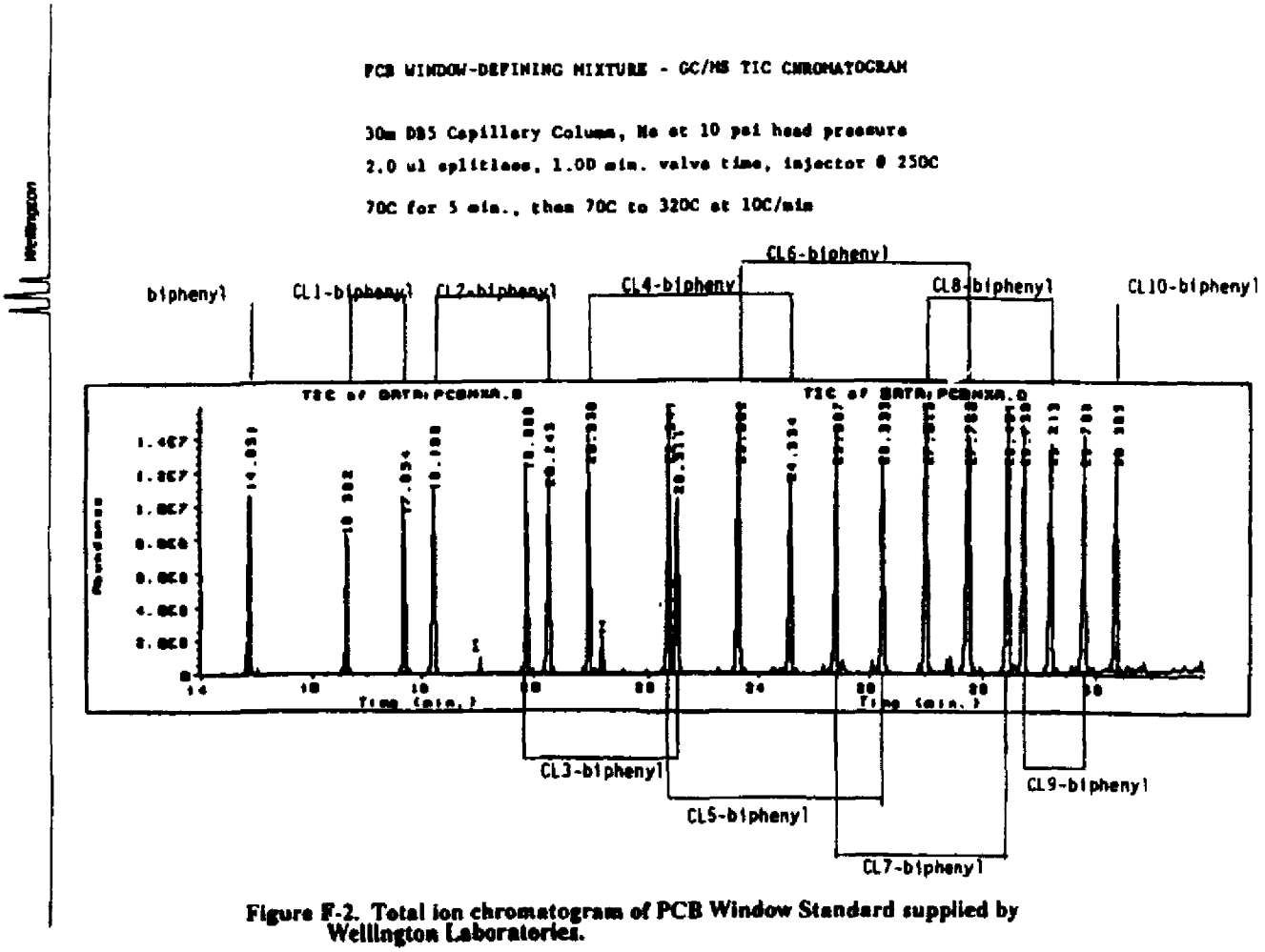


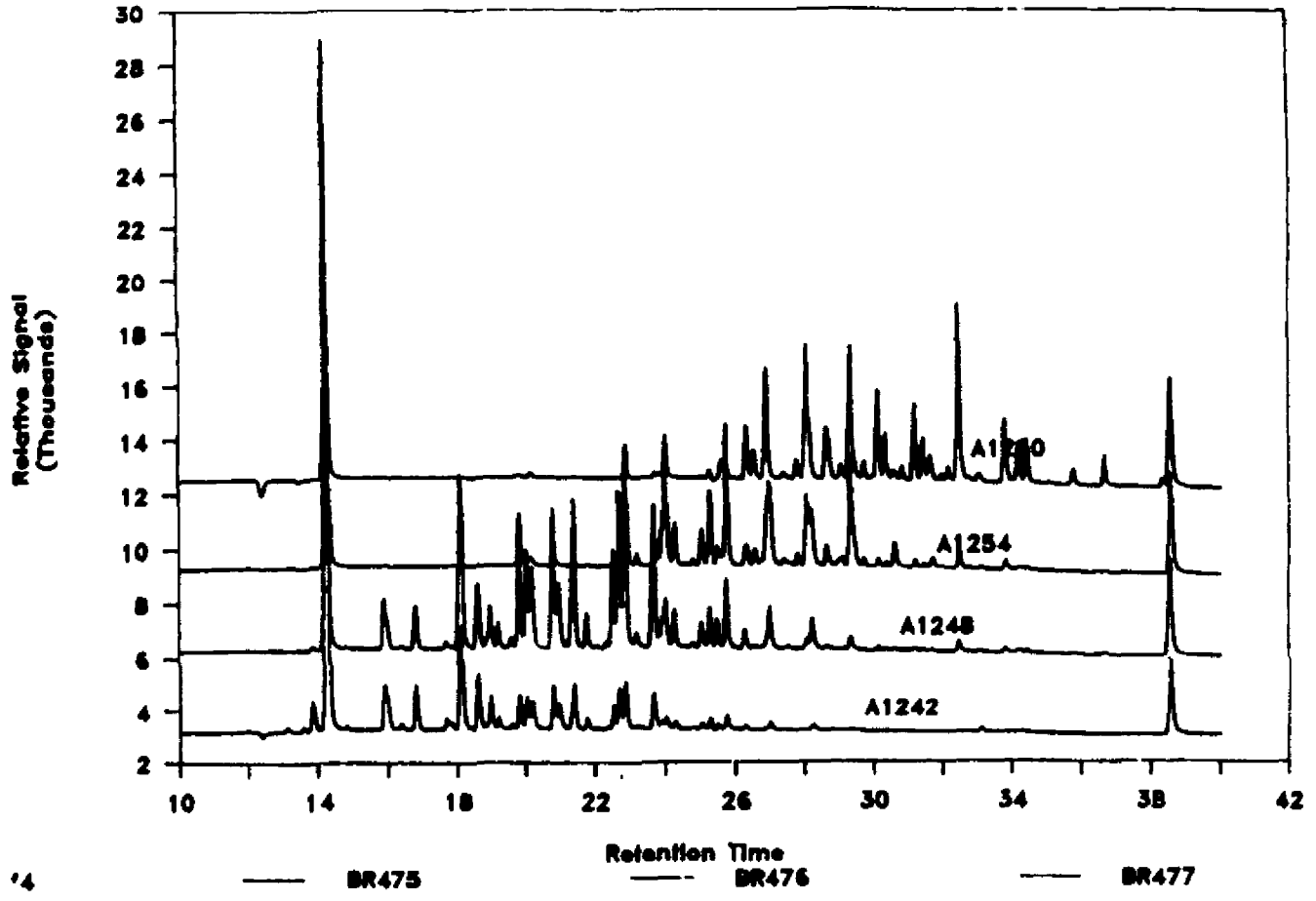
Figure F-2. Total ion chromatogram of PCB Window Standard supplied by Wellington Laboratories.

MONS 221226

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

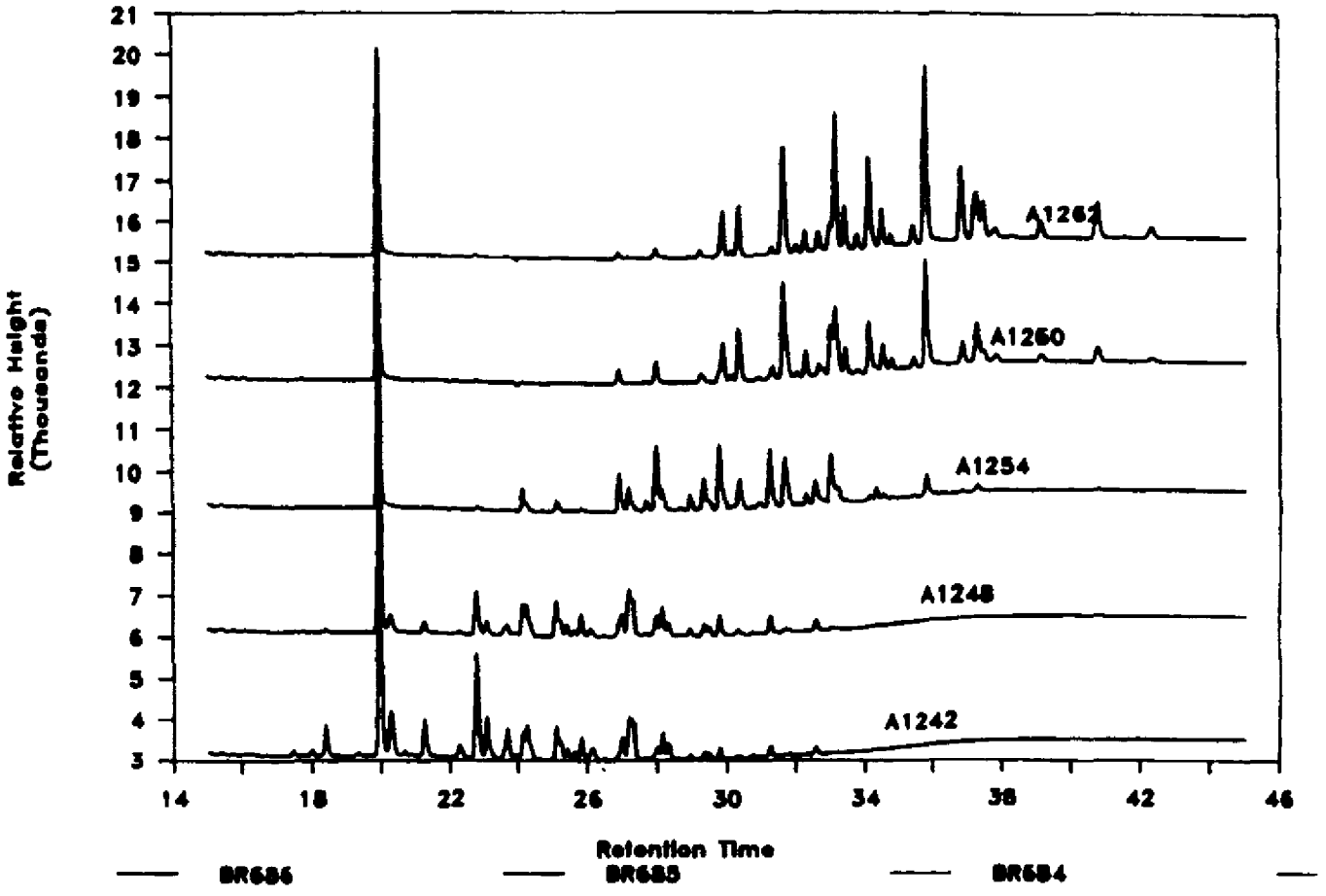
MONS 221227

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



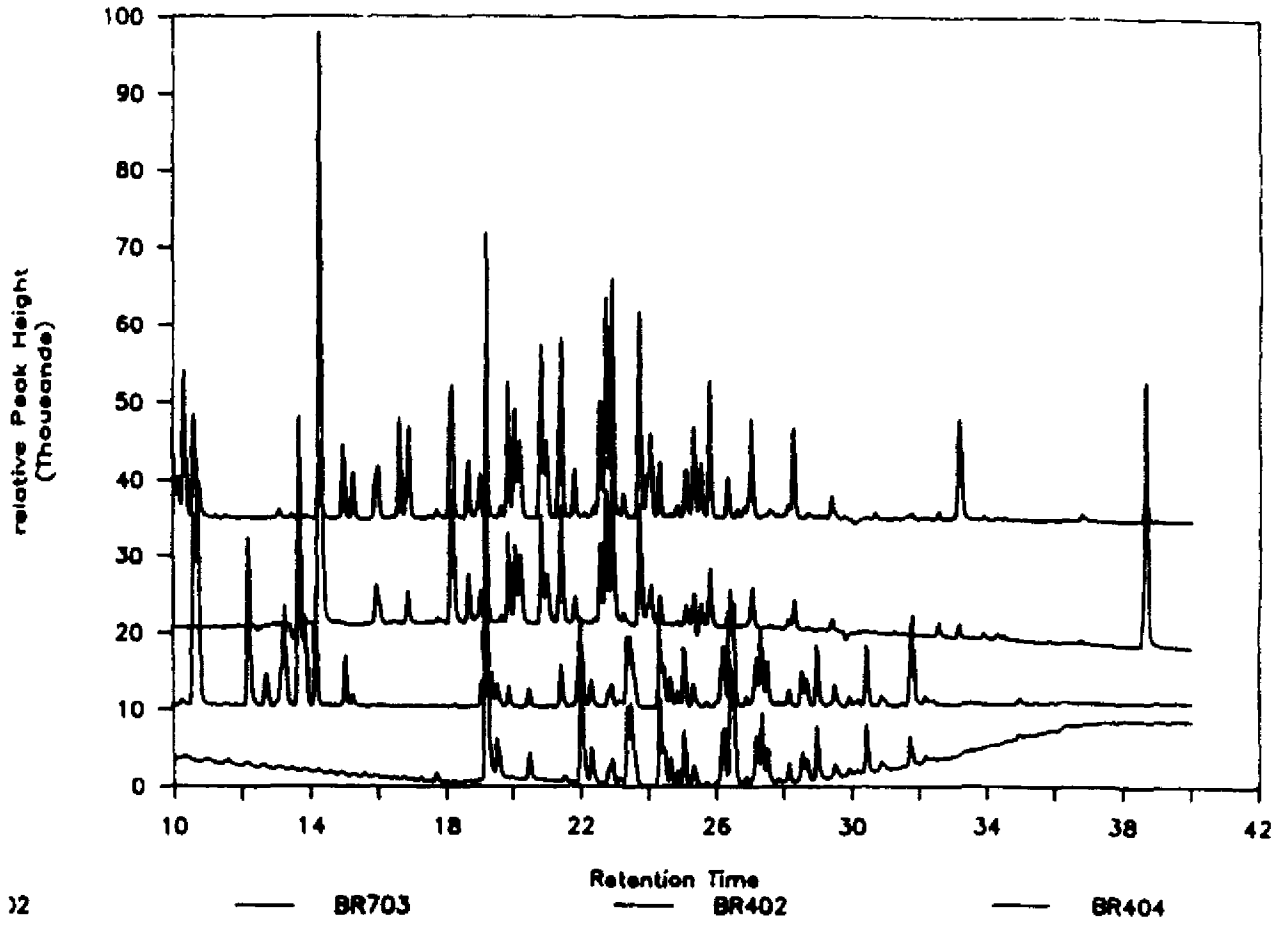
MONS 221228

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



MONS 221229

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



MONS 221230

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/UAC 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 effects the precision of the analysis is the sample extraction step. This step normally results in sample analysis precision on the order of + or - 30%.

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

(continued)

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

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

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

(continued)

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

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

Further Information

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

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

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

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

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

QAQCTE:MH

Analyte Extraction Terminology

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

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

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

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

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

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

(continued)

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

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

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

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

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

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

(continued)

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

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

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

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

(continued)

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

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

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

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

(continued)

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

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

Further Information

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

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

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

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

ANEXTE:MH

MONS 221239

Instrumental Analysis Terminology

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

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

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

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

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

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

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

(continued)

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

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

RRF = RR/(Analyte Concentration)

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

Analyte Extract Concentration = RR/RRF

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

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

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

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

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

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

External Standard Quantitation Method - Terminology which refers to a method of data analysis This method requires that a known amount of the analyte standard be analyzed under the same conditions as the sample extract. In order to determine the analyte concentracion-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

MONS 221242

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

RECEIVED
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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)

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

APPROVAL BY: Janet M Lee
J. M. Lee
Environmental Sciences Center
QC/QA and GLP Supervisor

APPROVAL DATE: March 4, 1988

REPT NO MSL-7431
COPY NO 17
AUTHORS: D. E. McKenzie and B. M. Hughes
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.

MONS 221245

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. Varnedo (G4WM)
C. E. McLaughlin (B3NF)
J. R. Craddock (A2NE)
P. E. Michael (A2NE)
R. G. Kaley (A2NE)
T. M. Bistline (E2NP)

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

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)

MONS 221246

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:

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

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 ultasonic 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.

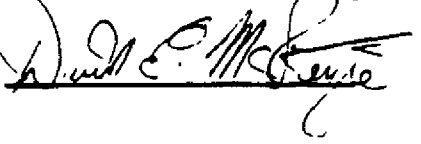
Appendix B. A Typical Soil and Oil Extraction Procedure.

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.

MONS 221251

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

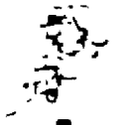
A PCB screening test for transformer oil.

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.)



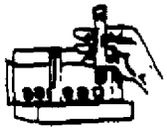
STEP 3. Remove the caps from both tubes and pour the buffer solution from Tube 2 (with green cap) into Tube 1. Replace the cap tightly on Tube 1 and shake vigorously for 10 seconds. Vent the tube by partially unscrewing the dispenser cap. Close securely and shake well for 10 seconds more. Vent the tube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



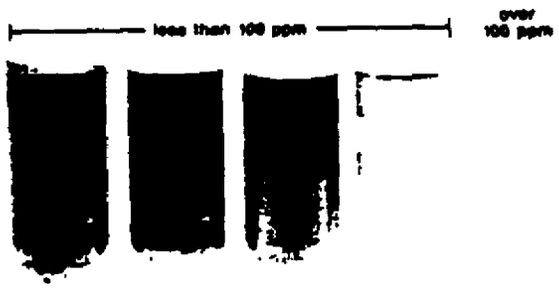
STEP 4. Stand Tube 1 upside-down on its cap and allow to settle for two minutes. If the oil layer is below the buffer layer, discontinue the test at this point as the oil is primarily pure PCB. (Askarel). See photograph on back of this sheet. If the oil layer is on top of the water layer, position Tube 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.



DEXSIL DEXSIL CORPORATION

MONS 221252

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

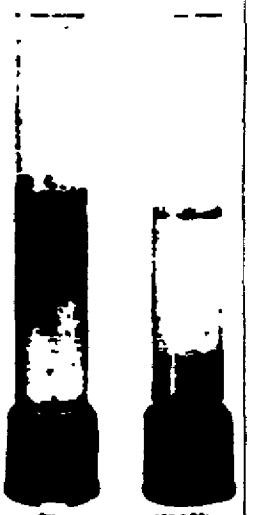
A precise amount of transformer oil is placed into Tube 1. The colorless ampule containing a catalyst is broken and thoroughly mixed with the oil. A second ampule containing metallic sodium is broken and the sodium activated by the catalyst strips chlorine from the PCBs forming sodium chloride. A buffer 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



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 950210

MONS 221253

Appendix B. A Typical Soil and Oil Extraction Procedure.

MONS 221254

ESC Standard Extraction Method (SEM) — ESC Standard Extraction Method No PCEX02

TITLE PCBs in Non-EC Active Matrices

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, monochlorobiphanyl isomers through decachlorobiphenyl

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

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

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

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

LIMITATIONS: Method appliss 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

MONS 221255

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 221256

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). [ ] Indicate Kit type, below next to extract numbers.

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. [ ]

Test Negative. [ ] [ ] Test positive.

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

[ ] Add 500 ppm $Hg(NO_3)_2$ 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 ($Hg(NO_3)_2$ 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 221258

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

MONS 221259

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**

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

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

External Standard Method

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

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

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

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual

DATE OF METHOD CREATION: 10/25/83, 7/20/87 — Time Scamp - <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 CONTAIN- [ ]
ING 0 04 ug/mL CL6-BENZENE .
IN A 1.6 mL AUTOSAMPLER .
VIAL .

PLACE VIAL IN AUTOSAMPLER [ ]
TRAY OF HP-5880 .
.
RECORD ELECTRON CAPTURE [ ] . . . .
AND GAS CHROMATOGRAPHIC
OPERATING PARAMETERS AND
3357-LAS FILE INFORMATION
IN INSTRUMENT LOGBOOK.

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

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

INTERNAL STANDARD CALCULATION

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

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

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 avarage concentration by (Extract Volume)/(Sample Amount).

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

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

MONS 221261

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

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, anthrecene-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 charecteristic 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'-pencachlorobiphenyl
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'-nonechlorobiphenyl
decachlorobiphenyl

(continued)

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

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

Characteristic and Confirming Masses Monitored

	Characteristic (AMU)	Confirming (AMU)
anthracene-d10	188	190
4-chlorobiphenyl-6C13	194	196
3,3'4,4'-tetrachlorobiphenyl-12C13	301.8	303.8
2,2'3,3',5,5',6,6'-octachlorobiphenyl-12C13	439.7	441.7
decachlorobiphenyl-12C13	509.6	511 6
monochlorobiphenyls	188	190
dichlorobiphenyls	222	224
trichlorobiphenyls	255.9	257 9
tetrachlorobiphenyls	289 8	291 8
pentachlorobiphenyls	323.9	325 9
hexachlorobiphenyls	359.8	361 8
heptachlorobiphenyls	393 8	395 8
octachlorobiphenyls	427.7	429 7
nonachlorobiphenyls	461.6	463 6
decachlorobiphenyl	497.6	499 6

FLOW SHEET MSFS25

FOR ESC ANALYSIS METHOD MSAN25

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

..... LOG NO ....

MS LOGBOOK PAGE NUMBER: .....

EXTRACT COLOR AND DESCRIPTION .....

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

PLACE IN A 1.6 mL AUTO- . THE INTERNAL STANDARD SOLUTION
SAMPLER VIAL THE SAMPLE EX- . USUALLY CONTAINS 10 UG/mL ANTHRACENE-D10
TRACT OR STANDARD SOLUTION . IN CH2CL2 OR BENZENE
CONTAINING PCB CONGENERS .
WITH 150 uL INTERNAL [ ]
STANDARD SOLUTION. .

PLACE VIAL IN AUTOSAMPLER [ ] CALCULATE CHARACTERISTIC ION AREAS OF
TRAY OF HP-5985/87. . ANALYTES OF INTEREST AND ANTHRACENE-D10
. INTERNAL STANDARD
RECORD MASS SPECTROMETRIC [ ] . . . . . [ ]
AND GAS CHROMATOGRAPHIC .
OPERATING PARAMETERS IN .
INSTRUMENT LOGBOOK.

.

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

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

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

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

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

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

MONS 221266

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

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

(continued)

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

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

Further Information

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

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

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

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

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

QAQCTE:MH

Analyte Extraction Terminology

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

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

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

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

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

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

(continued)

Matrix - Terminology used to reference the major components of a sample In most cases, major components are not of direct interest except as they affect the types of extraction steps and other isolation procedures which must be used to isolate the analyte from the sample matrix. For the majority of environmental samples which ESC is asked to analyze, the matrix is normally water or soil However, many environmental matrices contain relatively high levels of organic compounds which may interfer with the instrumental analysis of the analyte of interest. Therefore, depending upon the analyte of interest, the required level of detection, and the amounts and types of other organic materials which may interfer with the instrumental analysis, extraction methods must be developed which are very specific for a given set of samples. In order to deal with almost a limitless set of matrices, analytes, extraction methods and instrumental methods, an organized approach to eample extraction, followed by instrumental anslysis, 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 ths 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 tha analyte of intsrest during the axtraction and clean-up steps Examples of this type of surrogstes are deutarated compounds which may be added to a matrix in which any chromatographable materials are to be identifisd 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 end extract enalysis what analytes may be present, surrogetes must be chosen that could not possibly be present in the semple. In eddition, since only liquid/liquid or liquid/solid extraction would be involved, with no clean-up (since clean-up would rsmove compounds in which the requestar is interested) any hydrophobic deuterated compound could serve es a surrogate Normelly, phenol-d5, 2-CL-phenol-d4, CL2-benzene-d4, pyrene-dl0 and chrysene-dl2 are used es Surrogate Stendards for the analysis of a wide veriety 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 enalysis of Aroclor formulations. Since mass spectrometric detection is not used, surrogatss which contain unique isotopes cannot be distinguished from their native counterparts. Therefore, other compounds are used as Surrogats Standards since they heve been observed to act exactly ee the polychlorinated biphenyls (PCBs) in the Aroclor formulations. Any halogenated compound can be used which acts exactly as PCBs in the extraction and cleen-up step. ESC normally uses CL5-benzene and 2,4,6-CL3-biphenyl for this purpose.

Native Analytes - Terminology which references the enalytes 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 leval 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.

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

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

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

ANEXTE:MH

MONS 221275

Instrumental Analysis Terminology

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

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

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

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

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

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

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

(continued)

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

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

RRF = RR/(Analyte Concentration)

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

Analyte Extract Concentration = RR/RRF

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

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

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

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

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

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

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

(continued)

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

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

Analyte Extract Concentration = (Analyte Area)/RF

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

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

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

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

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

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

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

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

(continued)

Further Information

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

INANTE:MH

MONS 221279

Mortality of PCB Workers at the Monsanto Plant in Sauget, Illinois

Judith A. Zack and David C. Musch
December 14, 1979

MONS 221280

Summary

An historical prospective mortality study was conducted on workers exposed to polychlorinated biphenyls at the Monsanto Company plant in Sauget, Illinois. Eighty-nine workers exposed to PCBs for a minimum of six months during the period 1945 to 1965 were followed for their mortality experience. Observed deaths were not statistically different from expected deaths for any of the cancer sites examined. No deaths were observed from cancer of the liver or pancreas or from malignant melanoma. No excess was seen in deaths from liver cancer. A statistically significant excess of circulatory disease, exclusive of arteriosclerotic heart disease, was seen in white males.

Background

The term "polychlorinated biphenyls" (PCBs) describes a group of mixed chlorinated biphenyls which vary in consistency from light free-flowing liquids to crystalline and non-crystalline solids depending on the degree of chlorination. PCBs have been in commerical use since 1929. Their electrical properties, fire resistance, and inertness have made them popular for many applications. They have been used particularly in the production of electric capacitors and transformers and in the insulation of electric cables and wires.

Reports of adverse health effects in man and carcinogenic effects in certain animals species prompted the National Institute of Occupational Safety and Health (NIOSH) to request that epidemiological studies be carried out on PCB-exposed worker populations. In a preliminary study of workers exposed to PCBs at the Mobil Oil Company plant in Paulsboro, New Jersey, Bahn reported an excess of deaths from malignant melanoma and pancreatic cancer among research and production employees.[1] At the same time, Roush reported the findings of a preliminary mortality study of PCB workers at the Monsanto Company plant in Sauget, Illinois (G. Roush, written communication, September 1976). No cases of malignant melanoma or pancreatic cancer were found.

The accidental poisonings with PCBs in Yusho, Japan provide information on the acute toxic effects of PCBs for man. PCB poisoning occurred following the ingestion of Kanemi rice oil contaminated with PCBs. The general symptoms of Yusho disease included neuroendocrine disturbances, abnormal lipid metabolism, disturbances of the respiratory system, acneform eruption, pigmentation, and ocular changes.[2] Yusho disease, however, has also been attributed to the ingestion of polychlorinated dibenzofurans (PCDFs) present in the rice oil as PCB contaminant. The presence of PCDFs obscurs the relationship of PCBs to the clinical picture of Yusho disease.

In 1976, the Monroe County Board of Health conducted biochemical tests among Bloomington, Indiana residants exposed to PCBs. Serum levels of gamma glutamyl transpeptidaise and plasma triglyceride levels were found to show a direct relationship to serum PCB levels.[3]

In a recent study, Warshaw et al. evaluated pulmonary function in 243 workers exposed to PCBs in the manufacture of capacitors.[4] Thirty-four of these workers (14%) were found to have abnormal forced vital capacity measurement. Of these thirty-four, twenty-seven demonstrated a restricted pattern of impairment with virtually no radiographic change. The relationship between restrictive spirometric impairment and duration of PCB exposure was not addressed in this study.

Extensive studies of PCB toxicity for animals have been conducted and have been throughly reviewed.[5] In a two-year chronic oral toxicity test of PCBs, no carcinogenic effects were observed in the rats and beagles tested. Hyperplasia, hypertrophy, extensive fibrosis, and several benign tumors were observed in the livers of the rats, but there was no evidence of malignancy of any of the test animals.[6]

Other investigators have reported liver tissue effects ranging from hyperplasia to well-differentiated hepatocellular carcinomas in rats or mice fed with various PCB-containing diets. Kimbrough fed rats PCBs and observed lipid accumulation, hypertrophy, and adenofibromas, but no maligancies in the livers of the test animals.[7] In a later

experiment, the same researcher reported the induction of hepatocellular carcinomas and neoplastic nodules of the liver.[8]

The literature on PCB toxicity points to several organ sites in man that could potentially incur toxic effects of exposure to PCBs. The present study represents a completed analysis of the preliminary study of Monsanto PCB workers reported in 1976. This study will examine the mortality experience of PCB workers with particular attention given to overall cancer, liver and pancreatic cancer, and malignant melanoma.

Population and Methods

This study focused on workers exposed to polychlorinated biphenyls at the Monsanto plant in Sauget, Illinois. PCBs were manufactured at this plant during the period 1936 to 1977. All male employees from the hourly roll who worked in the PCB department for at least six months between January 1, 1945, and December 31, 1965, were selected for study.

During the period of study, the PCB process remained relatively unchanged The process, in brief, involved the batch chlorination of biphenyls in the presence of iron and iron chloride catalysts. The crude mixture produced in the chlorinators was aerated, distilled, earth treated, filtered and transferred to storage tanks. No industrial hygiene data were available to quantify the PCB exposure levels. Through interviews with plant personnel, it was determined that exposure levels to PCB did not vary considerably within the PCB department. Therefore, all workers in the PCB department were considered to have common exposure level. Other exposures in this department included hydrochloric acid, tri- and tetrachlorobenzene, biphenyl, and chlorine gas.

The PCB worker cohort was identified from the plant's computerized work history system. Eighty-nine male hourly employees met the criteria for inclusion in the study. The vital statue of each member was determined as of December 31, 1977, using standard followup techniques. A death certificate was obtained for each person found to be deceased.

The underlying cause of death was coded to the Eighth Revision of the International Classification of Disease, Adapted[8] by an experienced independent nosologist.

The data were analyzed by the modified life table method using the Monson program.[9] Standardized mortality ratios were calculated for nineteen selected cause-of-death categories. Mortality comparisons were made with the general U.S. population. Statistical significance was determined at the 5% level assuming a Poisson distribution.

Results

Eighty-eight of the eighty-nine members of the study cohort were traced. Fifty-eight were verified living and thirty were verified deceased by death certificate. The results of this vital status tracing are shown in Table 1.

The person-years of observation contributed by the eighty-nine member study cohort are shown in Table 2. A total of 1800.1 person-years were observed at all ages. Most of these person-yeers were observed in the middle-age range with 1333.1 person-years (74%) observed for ages 35-60.

Table 3 categorizes duration of exposure to PCBs by vital status. For both living and deceased workers, the majority were exposed less than three years. The next frequent exposure class was for five or more years of exposure. The least number of workers fell in the three to five years of exposure category. Overall, the average length of exposure for living and deceased workers was similar with deceased workers having 3.7 years of exposure and living workers having slightly less at 3.2 years.

The mortality experience for all males is summarized in Table 4. The mortality of white and nonwhite males is examined separately in Tables 5 and 6. Observed and expected deaths and SMR's are shown for each of the nineteen cause-of-death categories examined.

For all males, there were 30 deaths observed and 22.88 expected (Table 4) The overall SMR was 131. For malignant neoplasms, 8 deaths were observed with 4.46 expected. The SMR for all malignant neoplasm was 179. No deaths were observed from liver or pancreatic cancer or from malignant melanoma. The SMR for lung cancer was high at 278. The only statistically significant difference seen in this table is seen for diseases of the circulatory system, exclusive of arteriosclerotic heart disease. Nine deaths were observed with 3.98 expected, yielding a SMR of 226.

Table 5 shows the overall SMR remains high for white males at 133. There were 4 deaths from cancer with 2.70 expected. None were from liver or pancreatic cancer or from malignant melanoma. The high SMR's seen in Table 4 for lung cancer and the residual diseases of the circulatory syetem appear to be explained by the high SMR's for these causes in white males. There were 3 deaths observed from lung cancer with 0.94 expected, yielding a SMR of 319. For the category of circulatory disease, exclusive of arteriosclerotic heart disease, the SMR was 526 with 7 observed and 1.83 expected deaths. This difference was the only one of statistical significance in this table.

For nonwhite males, there were no statistically significant differences seen in Table 6. The overall SMR was also high at 128. Four deaths from cancer were observed with 1.76 expected, but none from liver or pancreatic cancer or from malignant melanoma.

Case histories for the eight cancer deaths are summarized in Table 7.

Discussion

The results of this study do not corroborate an excess risk of pancreati cancer or malignant melonoma among workers exposed to PCBs as reported in the study of Mobil workers. Nor do the results uphold translating to man the outcome of animal tests, in which hepatomas have been associated with long-term PCB exposure. In this study, no deaths were ob-

served from pancreatic or liver cancer or from malignant melanoma.

The SMR for all causes was elevated from what one would expect in an industrial cohort. The high SMR for white males is, for the most part, explained by the excess in deaths from circulatory diseases exclusive of arteriosclerotic heart diseases. This cause-of-death category includes deaths from rheumatic heart disease, cerebrovascular disease, and other forms of heart disease, all of which are unlikely to be related to exposure in the workplace. It is urged that a larger study to be undertaken of the total Krummrich plant population to evaluate the overall risk of death from all causes and from cardiovascular diseases.

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Table 1

Polychlorinated Biphenyl Workers -
Results of Tracing as of 12/31/77

Total to be traced	89
Status Known	88
Verified Alive	58
Verified Dead	30
Death Certificate Found	30
Death Certificate Not Found	0
Status Unknown	1

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Table 2

Person-Years of Observation Contributed by Workers Exposed to PCB's by Five Year Age Groups

Age in Years	Person-Years of Observation
20-24	11.4
25-29	63.8
30-34	142.6
35-39	222.5
40-44	283.2
45-49	325.3
50-54	294.5
55-59	207.6
60-64	134.0
65+	115.2
Total - All Workers	1800.1

Table 3

Duration of Exposure to PCB's for Workers by Vital Status

Years of Exposure	Living N=58	Deceased N=30	Unknown N=1	Total N=89
< 1 yr.	20	5	1	26
1-3 yrs.	20	12	0	32
3-5 yrs.	6	4	0	10
> 5 yrs.	12	9	0	21
Average Length of Exposure	3.2 yrs.	3.7 yrs.	0.7 yrs.	3.3 yrs.

*Lifetime exposure prior to 1-1-66.

Table 4

Observed and Expected Deaths, All Males

I.C.D. No. (Eighth Rev.)	Cause of Death	Observed	Expected	SMR
	All causes of death	30	22.88	131
140-209	All malignant neoplasms	8	4.46	179
140-149	Buccal cavity and pharynx	0	0.16	-
150-159	Digestive organs and peritoneum	1	1.33	75
155,156	Liver	0	0.10	-
-	All other digestive organs	1	1.23	81
160-163	Respiratory system	4	1.53	261
162,163	Lung	4	1.44	278
-	All other respiratory organs	0	0.09	-
185-189	Genitourinary organs	1	0.51	196
200-209	Lymphatic and hematopoietic tissue	1	0.40	250
-	Other sites	1	0.53	189
390-458	Diseases of the circulatory system	16	11.17	143
410-413	Arteriosclerotic heart disease	7	7.19	97
-	All other diseases of the circulatory system	9	3.98	226*
460-519	Diseases of the respiratory system	1	1.27	79
520-577	Diseases of the digestive system	2	1.20	167
-	All other diseases	2	2.40	83
800-990	External causes of death	1	2.38	42

Number of persons observed = 89

* $p < .05$

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Table 5

Observed and Expected Deaths, White Males

I.C.D. No. (Eighth Rev.)	Cause of Death	Observed	Expected	SMR
	All causes of death	10	13.53	133
140-209	All malignant neoplasms	4	2.70	148
140-149	Buccal cavity and pharynx	0	0.09	-
150-159	Digestive organs and peritoneum	0	0.75	-
155,156	Liver	0	0.05	-
-	All other digestive organs	0	0.70	-
160-163	Respiratory system	3	0.94	319
162,163	Lung	3	0.89	337
-	All other respiratory organs	0	0.05	-
185-189	Genitourinary organs	0	0.20	-
200-209	Lymphatic and haematopoietic tissue	0	0.20	-
-	Other sites	1	0.36	278
390-458	Diseases of the circulatory system	11	6.82	161
410-413	Arteriosclerotic heart disease	4	4.99	80
-	All other diseases of the circulatory system	7	1.83	526*
460-519	Diseases of the respiratory system	0	0.73	-
520-577	Diseases of the digestive system	1	0.75	133
-	All other diseases	2	1.10	182
800-998	External causes of death	0	1.43	-

Number of persons observed = 60

*p < .05

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Table 6

Observed and Expected Deaths, Nonwhite Males

I.C.D. No. (Eighth Rev.)	Cause of Death	Observed	Expected	SMR
	All causes of death	12	9.35	128
140-209	All malignant neoplasms	4	1.76	227
140-149	Buccal cavity and pharynx	0	0.07	-
150-159	Digestive organs and peritoneum	1	0.58	172
155,156	Liver	0	0.05	-
-	All other digestive organs	1	0.53	189
160-163	Respiratory system	1	0.59	169
162,163	Lung	1	0.55	182
-	All other respiratory organs	0	0.04	-
185-189	Genitourinary organs	1	0.23	435
200-209	Lymphatic and hematopoietic tissue	1	0.12	833
-	Other sites	0	0.17	-
390-458	Diseases of the circulatory system	5	4.35	115
410-413	Arteriosclerotic heart disease	3	2.20	136
-	All other diseases of the circulatory system	2	2.15	93
460-519	Diseases of the respiratory system	1	0.54	185
520-577	Diseases of the digestive system	1	0.45	222
-	All other diseases	0	1.30	-
800-998	External causes of death	1	0.95	105

Number of persons observed = 29

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Table 7

Case Summaries of Cancer Deaths Among PCB Workers

Race	Year of Birth	Year of First Exposure	Year of Death	Duration Ever Exposed to PCB's	Death Certificate Statement of Cause of Death
Nonwhite	1909	1942	1959	1 yr., 1 mo.	Carcinoma of colon (153.0)
Nonwhite	1913	1954	1965	7 yrs., 1 mo.	Carcinomatosis - primary site kidney (189.0)
Nonwhite	1909	1951	1968	2 yrs., 4 mos.	Carcinoma of the lung (162.1)
White	1893	1943	1963	2 yrs., 10 mos.	Carcinoma right lung (162.1)
White	1912	1951	1968	2 yrs., 4 mos.	Cancer of lung (162.1)
White	1916	1951	1977	2 yrs., 9 mos.	Carcinoma of lung (162.1)
Nonwhite	1907	1945	1964	8 mos.	Multiple myeloma (203.0)
White	1906	1940	1957	6 yrs., 7 mos.	Metastatic carcinoma - carcinomatosis (199.0)

A Retrospective Mortality Study of Polychlorinated Biphenyl Manufacturing Workers[1]

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Abstract

A retrospective cohort mortality study of workers exposed to polychlorinated biphenyls (PCBs) during production of the compounds is reported. A total of 89 workers exposed to PCBs for a minimum of six months during the period from 1945 to 1965 were followed through 1977, and their vital status was determined. Comparison of this PCB-exposed cohort's mortality experience with age-, race-, and cause-specific mortality rates of the U.S. male population was performed. Observed deaths were not statistically greater than expected for any of the cancer sites examined. No deaths were observed for cancer of the liver or malignant melanoma. A statistically significant excess of circulatory disease deaths, exclusive of arteriosclerotic heart disease, was observed in white males.

The term "polychlorinated biphenyls" (PCBs) describes a group of mixed chlorinated biphenyls which vary in consistency from light, free-flowing liquids to crystalline and non-crystalline solids, depending on the degree of chlorination. PCBs have been in commercial use since 1929. Their dielectric properties, resistance to oxidation, acids, bases, and other chemicals, and thermal stability have made them useful for many applications, particularly in the production of capacitors and transformers, and in the insulation of electric cables and wires.

Reports of adverse health effects in man initiated from several incidents of accidental heavy exposure to PCBs. A process change in a chemical plant introduced an unspecified PCB compound (Arochlor) into the work environment, resulting in exposure of 14 workers to estimated breathing zone PCB levels of 0.1 mg/m^3.[1] Chloracne developed in one-half of the exposed workers, and liver function tests showed a borderline abnormality in one of the seven affected workers. After 13 months of follow-up, the chloracne had disappeared, but the liver function abnormality, although improved, remained.

Accidental ingestion of rice oil contaminated with PCBs has resulted in several outbreaks of common symptomatology, reported from Japan ("Yusho" disease)[2] and Taiwan ("Yu-Cheng" disease).[3] The most common acute symptoms observed were hyperpigmentation and acne-like lesions, discharge from the eyes, neuroendocrine disturbances, emesis, and diarrhea. There was a dose-response relationship between the amount of oil ingested and the proportion of persons reporting symptoms. After six years, many of the patients still reported such symptoms as headache, stomach pain, numbness of the extremities, arthralgia, and respiratory symptoms.[4] Although massive ingestion of PCBs occurred in these incidents, analysis of the cooking oil in the Yusho episode showed that

polychlorinated dibenzofurans[5] and polychlorinated quaterphenyls[6] were co-contaminants. Analysis of blood samples from patients in both incidents, as well as liver and adipose tissue from Yusho patients, showed the presence of these compounds along with PCBs.[7, 8] These co-contaminants obscure the relationship of PCBs to the symptoms resulting from ingestion of the contaminated rice oils.

The Yusho incident served as a stimulus to a large number of toxicological studies dealing with the health effects of PCBs. Chronic toxicity studies of PCBs in mice and rats have shown hepatic effects ranging from hyperplasia to well-differentiated hepatocellular carcinomas following the ingestion of various PCB-containing diets.[9, 10] Studies of human exposure to PCBs have shown that body burdens are directly related to the level of exposure to PCBs.[11, 13] Morbidity studies have focused on the effects of PCBs on the skin, liver, blood chemistry, and other systems.[13, 16] Chloracne, dermatitis, mild liver function abnormalities, and increased triglycerides levels were reported with some consistency from these studies.

Mortality studies of occupationally-exposed individuals have not yielded consistent results. A preliminary report[17] addressed an increase in deaths due to malignant melanoma (2 obs. vs. 0.04 exp.), which occurred in a group of 51 research and development and 41 refinery workers at a New Jersey petrochemical plant. Another author commented on problems in defining exposures in this study.[18] In a study of 2567 capacitor plant workers exposed to PCBs,[19] excesses of liver and rectal cancer deaths, which were not statistically significant, were reported. Although few deaths were available for analysis by duration of exposure and latency, there was no trend by these measures for liver cancer deaths, and a slight increase by latency but not by length of

exposure for rectal cancer deaths. Another study of 1310 workers from a capacitor-manufacturing plant with at least six months employment between 1946 and 1970 found statistically significant excess deaths from all cancers (in males) and all causes (in females).[20] Suggestive, but not statistically significant, excesses were noted for deaths due to digestive system cancers (in males) and malignancies of lymphatic and hematopoietic tissue (in males and females). No deaths were attributed to liver cancer or malignant melanoma. Due to the relatively young age of this worker cohort and the resultant small number of deaths (n = 27), site-specific excesses were based on two or three deaths; therefore, further study of this cohort was proposed by the authors.

At about the time of the initial report dealing with the mortality experience of an occupationally-exposed worker cohort, a preliminary review of the mortality experience of Monsanto workers involved in the production of PCBs was carried out. The present study represents a completed analysis of this worker cohort's mortality, with particular attention given to overall cancer, liver and pancreatic cancer, and malignant melanoma.

Materials and Methods

During the period from 1936 to 1977, PCBs were manufactured at Monsanto Company's Sauget, Illinois plant. Throughout this period, the process of manufacture remained relatively unchanged. This process, in brief, involved the batch chlorination of biphenyls in the presence of iron and iron chloride catalysts. The crude mixture produced in the chlorinators was then aerated, distilled, earth treated, filtered, and transferred to storage tanks before shipping. While no industrial hygiene monitoring data were available to quantify the PCB-exposure levels during the stuoy time, interviews with plant personnel established the fact that PCB exposure levels did not vary considerably within the PCB department. Therefore, all workers in this department

were considered to have a common exposure to PCBs, as well as other chemicals used in the department, including hydrochloric acid, tri- and tetrachlorobenzene, biphenyl, and chlorine gas.

All male employees from the hourly roll who worked in the PCB department for at least six months between January 1, 1945 and December 31, 1965 were selected for the study. By use of the plant's computerized work history system, union records, and manual work history verification, 89 male, hourly employees met the criteria for inclusion in the study. The vital status of 98.9% (88) of the cohort was determined as of December 31, 1977, using standard follow-up techniques. Table 1 summarizes the results of the vital status tracing. A death certificate was obtained for each person found to be deceased, and coding of the underlying cause of death was performed by an experienced nosologist, using the International Classification of Diseases, Adapted, Eighth Revision.[21]

Using a computer program developed for such an application,[22] the observed number of deaths among PCB workers was compared with those expected based on age-, race-, and cause-specific mortality rates of U.S. males. To compute expected numbers, person-years of observation were allocated to five-year age and calendar time categories and multiplied by corresponding race- and cause-specific death rates for U.S. males. The resulting quantities were summed over all ages and years to obtain the total expected numbers. Ratios of observed to expected number of deaths were expressed as standardized mortality ratios (SMRs). Significance testing of the SMRs was performed at the 5% level, assuming that the observed number of deaths follow a Poisson distribution.[23]

Results

Of the 88 PCB workers for whom vital status tracing was successful, 58 were verified living and 30 were verified deceased. Death certificates were

obtained for each of the decedents. The person-years of observation contributed by the entire worker cohort are shown in Table 2. A total of 1800.1 person-years were observed at all ages, with the majority observed in the middle-age range (ages 35-60 yrs. contributed 74%, or 1333.1 person-years to the total). Cross-classification of PCB exposure duration with vital status (Table 3) shows that the majority of both living and deceased workers were exposed less than three years. The average length of exposure to PCBs for living and deceased workers was similar, with deceased workers having slightly more exposure time (3.7 yrs. vs. 3.2 yrs., respectively).

Cause-specific SMRs are listed in Tables 4 and 5 for the 60 white and 29 nonwhite male members of the PCB study cohort. Deaths due to malignant neoplasms, as indicated in Table 4, accounted for 27% (8) of the total number of deaths. No cancer deaths were attributed to malignant neoplasms of the liver or pancreas, or malignant melanoma. While many of the cancer-specific SMRs exceed 100, none are statistically significant. In white males, lung cancer accounted for 75% of the deaths due to malignant neoplasms, while no single cause predominates in nonwhite males. Table 5 presents a brief summary of the eight cancer deaths in the PCB worker cohort.

Among the non-malignant causes of death (Table 6), diseases of the circulatory system accounted for almost half of the deaths. For the category of circulatory system disease, exclusive of arteriosclerotic heart disease, the SMRs for white males and for the total cohort were significantly greater than 100. The excess in deaths due to this category is accounted for solely by the experience of the white males, since nonwhite males showed an SMR of 93. No other causes of death in Table 6 showed observed deaths which were statistically

greater than expected. The overall SMR for malignant and non-malignant causes was 131 (30 observed, 22.88 expected deaths). Little difference was found in this overall SMR by race (for whites, 133; for nonwhites, 128).

Discussion

This investigation dealt with the mortality experience of a cohort of 89 male workers involved for a least six months in the production of PCBs between January 1, 1945 and December 31, 1965. The only cause of mortality which showed a statistically significant excess in observed deaths was circulatory diseases exclusive of arteriosclerotic heart disease. This excess was apparent only for white males. Circulatory diseases in this category included deaths from rheumatic heart disease, cerebrovascular disease, and other forms of heart disease.

If this excess cause of death is not an artifact, one would postulate that there may also be a relation between risk factors of these circulatory diseases and PCB exposure. Blood pressure, a risk factor (when elevated) of coronary heart disease (CHD) and cerebrovascular disease, has been related to blood PCB levels in two studies, with conflicting results. One study[24] found that serum PCB levels made a statistically significant contribution to explaining the variability of diastolic (but not systolic) blood pressure measurements in multiple regression analyses of data from a community exposed to DDT and PCBs, while another study[25] found no such association. Studies of lipids, which have been associated with CHD and (with less consistency) with cerebrovascular disease, have considered total cholesterol, high density lipoprotein (HDL), and triglyceride levels in relation to blood PCB levels. While no consistent association has been shown between blood PCBs and total cholesterol or HDL levels, most,[13, 16] but not all,[24] studies have shown increased triglyceride

levels with increased blood PCB levels. Given the lack of support for an association between PCBs exposure and non-ASHD circulatory system deaths from previously reported mortality studies, and the inconclusive relation between cardiovascular risk factors and blood PCBs, this excess cause of death must be viewed as a preliminary finding, in need of support from other studies.

While previous mortality studies as well as toxicological studies have implicated a variety of cancers - digestive system, liver, pancreas, malignant melanoma - as possible results of exposure to PCBs, the present study provides no support to such findings. In the Monsanto worker cohort, no deaths were attributed to liver or pancreatic cancer, nor were any malignant melanomas found. The overall excess in cancer deaths, while not statistically significant (8 observed, 4.46 expected, SMR = 179), is mainly a result of respiratory cancer deaths in workers for whom smoking habits were not ascertained.

Although the present study's results do not support a carcinogenic effect associated with PCB exposure, there are significant limitations to the results. A principal limitation is the size of the worker cohort, which prevents the detection of increases in risk to deaths from rare diseases. A much larger cohort followed for a longer period would be necessary to detect a two-fold increase, for example, in observed liver cancer deaths. In order to allow for a latency period, the study cohort was limited to those working at least six months until December, 1965. Since PCB production continued until 1977, further follow-up of the mortality of Monsanto's PCB-exposed workers will have larger numbers, as well as longer follow-up time.

Another limitation common to retrospective studies of occupational cohorts is lack of knowledge of exposures from other work-related activities. While personal habits, such as smoking and drinking, can sometimes be roughly ascertained if co-workers or family members are available for interview, previous

exposures, or concurrent occupational exposures during follow-up, are often impossible to assess. The connection of an effect to a previous PCB exposure must first consider other potential effect-related exposures.

A useful comparison group in occupational mortality studies is an unexposed industrial cohort, from the same plant, if possible. Ascertainment of mortality of such a cohort allows for comparison of SMRs between exposed and unexposed cohorts, which reduces the possibility that an elevated cause of death in the exposed cohort is a result of a generally greater than expected cause-specific mortality in that area or industry. Such a comparison group would have been useful in evaluating the elevated mortality due to non-ASHD circulatory system diseases found in this study.

Given the size of the study cohort and the other limitations described above, the results of this analysis of PCB-exposed workers' mortality experience are not definitive, but rather contribute to the health-effects research on PCBs. A future study of this cohort could evaluate the mortality of workers involved from the initial through the final year of PCB production, and thus have more power to detect any possible hazards of PCB exposure.

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Table 1

Follow-up Status of Polychlorinated Biphenyl Workers as of December 31, 1977

Follow-up Status	Number
Status known	88
Verified alive	58
Verified deceased	30
Death certificate found	30
Death certificate not found	0
Status Unknown	1
Total	89

Table 2

Person-years of Observation Contributed by Polychlorinated Biphenyl Workers by Age

Age in Years	Person-years of Observation
20-24	11.4
25-29	63.8
30-34	142.6
35-39	222.5
40-44	283.2
45-49	325.3
50-54	294.5
55-59	207.6
60-64	134.0
65+	115.2
Total	1800.1

Table 3

Duration of Exposure to Polychlorinated Biphenyls by Vital Status

Years of Exposure*	Vital Status: Living (n = 58)	Deceased (N = 30)	Unknown (n = 1)	Total (n = 89)
<1	20	5	1	26
1-3	20	12	0	32
3-5	6	4	0	10
>5	12	9	0	21
Average Length of Exposure	3.2 yrs.	3.7 yrs.	0.7 yrs.	3.3 yrs.

*Lifetime exposure prior to 1-1-66

Table 4

Observed and Expected Deaths Due to Malignant Neoplasms by Race, PCB Worker Cohort

I.C.D. No. (Eighth Rev.)	Cause of Death	White Males			Nonwhite Males			Total		
		O	E	SMR*	O	E	SMR*	O	E	SMR*
140-209	All Malignant neoplasms	4	2.70	148	4	1.76	227	8	4.46	179
140-149	Buccal cavity & pharynx	0	0.09	-	0	0.87	-	0	0.16	-
150-159	Digestive organs & peritoneum	0	0.75	-	1	0.58	172	1	1.33	75
155, 156	Liver	0	0.05	-	0	0.05	-	0	0.10	-
150-154, 157-159	Other digestive organs	0	0.70	-	1	0.53	189	1	1.23	81
160-163	Respiratory system	3	0.94	319	1	0.59	169	4	1.53	261
162, 163	Lung	3	0.89	337	1	0.55	182	4	1.44	278
160, 161	Other respiratory organs	0	0.05	-	0	0.04	-	0	0.09	-
185-189	Genitourinary organs	0	0.28	-	1	0.23	435	1	0.51	196
200-209	Lymphatic & hematopoietic tissue	0	0.28	-	1	0.12	833	1	0.40	250
164-184, 190-199	All other sites	1	0.36	278	0	0.17	-	1	0.53	189

*No SMR was statistically significant (all p-values greater than 0.05)

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Table 5

Case Summaries of Cancer Deaths Among
Polychlorinated Biphenyl Workers

Race	Year of Birth	Year of First Exposure	Year of Death	PCB Exposure Duration (mo.)	Death Certificate Cause of Death (ICO8)
Nonwhite	1909	1942	1959	13	Carcinoma of colon (153.8)
Nonwhite	1913	1954	1965	85	Carcinomatosis-Primary site, kidney (189.0)
Nonwhite	1909	1951	1968	28	Carcinoma of lung (162.1)
White	1893	1943	1963	34	Carcinoma of right lung (162.1)
White	1912	1951	1968	28	Carcinoma of lung (162.1)
White	1916	1951	1977	33	Carcinoma of lung (162.1)
Nonwhite	1907	1945	1964	8	Multiple myeloma (203.0)
White	1906	1940	1957	79	Metastatic carcinoma-carcinomatosis (199.0)

Table 6

Observed and Expected Deaths Due to Non-malignant Causes by Race, PCB Worker Cohort

I.C.D. No. (Eighth Rev.)	Cause of Death	White Males O	E	SMR	Nonwhite Males O	E	SMR	Total O	E	SMR
390-458	Diseases of the circulatory system	11	6.82	161	5	4.35	115	16	11.17	143
410-413	Arteriosclerotic heart disease	4	4.99	80	3	2.20	136	7	7.19	97
390-409, 414-458	All other circulatory system	7	1.83	526*	2	2.15	93	9	3.98	226*
460-519	Diseases of the respiratory system	0	0.73	-	1	0.54	185	1	1.27	79
520-577	Diseases of the digestive system	1	0.75	133	1	0.45	222	2	1.20	167
800-998	External causes of death	0	1.43	-	1	0.96	105	1	2.38	42
	All other non-malignant causes	2	1.10	182	0	1.30	-	2	2.40	83
Total	All causes, malignant & non-malignant	18	13.53	133	12	9.35	128	30	22.88	131

*$p < 0.05$

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