N95- 33279

19951 26858

ENVIRONMENTAL APPLICATIONS FOR ION MOBILITY SPECTROMETRY

Robert K. Ritchie and Andreas Rudolph
Barringer Research Limited
304 Carlingview Drive, Rexdale, ON, Canada M9W 5G2

ABSTRACT

The detection of environmentally important polychlorinated aromatics by ion mobility spectrometry (IMS) was investigated. Single polychlorinated biphenyl (PCB) isomers (congeners) having five or more chlorine atoms were reliably detected in isooctane solution at levels of 35 ng with a Barringer IONSCAN ion mobility spectrometer operating in negative mode; limits of detection (LOD) were extrapolated to be in the low ng region. Mixtures of up to four PCB congeners, showing characteristic multiple peaks, and complex commercial mixtures of PCBs (Aroclors) were also detected. Detection of Aroclors in transformer oil was suppressed by the presence of the antioxidant BHT (2,6-di-*t*-butyl-4-methylphenol) in the oil. The wood preservative pentachlorophenol (PCP) was easily detected in recycled wood shavings at levels of 52 ppm with the IONSCAN; the LOD was extrapolated to be in the low ppm region.

INTRODUCTION

The Barringer IONSCAN ion mobility spectrometer is a proven instrument for the detection of trace amounts of explosives and illicit drugs. Current research efforts at Barringer are aimed at new IONSCAN applications, including the analysis of environmentally important compounds. IONSCAN systems require minimal or no sample preparation, are easy to use, and are very selective towards high electron affinity compounds such as polychlorinated aromatics. This presentation describes initial research to develop the IONSCAN into a rapid and on-site PCB and PCP detector.

Polychlorinated Biphenyls

PCBs are non-flammable, non-conductive, chemically quite inert, and have high boiling points; these properties make the commercial Aroclor mixtures desirable for use as insulator fluids or oil additives in electrical utility equipment such as transformers and capacitors. Unfortunately they have also been identified as major environmental pollutants, because high application temperatures can generate dangerous levels of dioxins and chlorinated dibenzofurans in the insulating fluids. Programs to remove PCBs from service usually either replace the whole transformer assembly (in case of high PCB content), or exchange the PCB containing oil with clean fluids (in case of lower PCB content). The present cutoff level for transformer replacement vs. oil exchange in Ontario is 50 ppm PCB in the oil. Analysis methods able to detect PCBs at levels below 50 ppm are needed to select the appropriate PCB removal action. [1]

PCB analysis is usually carried out by expensive and time consuming GC or GC/MS techniques. Recently, immunoassay [2] and colorimetric techniques aimed at on-site analysis have been developed; however, these require several handling and analysis steps, and are prone to interferences from other chlorinated organics or inorganic chlorides.

Wood Preservatives

Growing environmental awareness has led to a different attitude towards preservative-treated wood. It constitutes hazardous waste at the end of its life cycle, and it cannot be burned or landfilled due to release of the potentially dangerous preservatives. Manufacturers and users of this wood, faced with "cradle-to-grave" responsibility for these products, are looking for disposal alternatives.[3] Recycling is one important possibility, and in the case of wooden utility poles can result in almost complete recovery of the preservative (mostly creosote or PCP).

The patented TWT Technologies recycling process first removes the outer preservative-containing layer from the pole, and the somewhat smaller pole can then be reused, possibly for less demanding applications. The outer layer is subjected to a precisely defined heat treatment, dependent on the preservative used, which reclaims the preservative by distillation, and also produces a clean wood shaving product.[4]

Operational requirements are that the preservatives be identified before processing, and that the clean wood shaving product be declared non-hazardous (<40 ppm PCP, possibly reducing to 5 ppm). Fast, low-cost alternative methods to GC/MS are required, and again IMS appears to be a promising technique.[5]

EXPERIMENTAL METHODS

Table 1 details the analysis conditions that were employed on a Barringer IONSCAN Model 350 for the analyses of PCBs and PCP.

Table 1

	PCB Analysis	PCP Analysis
Ion Mode	Negative *	Negative *
Chloride Reactant Addition	No	Yes
Drift Tube Temperature	115°C	120°C
Inlet Temperature	300°C	300°C
Desorber Temperature	300°C	300°C

* explosives mode

PCB standard solutions (35 μg/ml in isooctane) were obtained from AccuStandard. Of the 209 different congeners, the following were chosen for analysis: 2-Chlorobiphenyl (#1), 3,3'-Dichlorobiphenyl (#11), 2,4,5-Trichlorobiphenyl (#29), 2,2'4,4'-Tetrachlorobiphenyl (#47), 2,3',4,5',6-Pentachlorobiphenyl (#121), 2,2',3,3',6,6'-Hexachlorobiphenyl (#136), 2,2',3,4,5,5',6-Heptachlorobiphenyl (#185), 2,2',3,3',4,4',5,5'-Octachlorobiphenyl (#194), 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (#206), and Decachlorobiphenyl (#209). The numbers in parentheses represent the Ballschmiter-Zell numbering method.

Aroclor standard solutions in Volt-Esso 35 (a hydrocarbon transformer oil) and in isooctane were provided by Ontario Hydro and Barringer Laboratories, respectively. The following Aroclors were investigated: Aroclor 1254 (54% chlorine by weight, 75 ppm in Volt-Esso 35, and 35 μg/ml in isooctane) and Aroclor 1260 (60% chlorine by weight, 50 ppm in Volt-Esso 35, and 5 mg/ml in isooctane). BHT was available in-house.

1 μl aliquots of the PCB or Aroclor solutions in isooctane were applied to a standard

IONSCAN Teflon filter, and the solvent was allowed to evaporate (ca. 30 s) before IMS analysis. Aroclor analysis from transformer oil was carried out by applying ca. 10 µl of the oil solutions onto a standard Teflon filter, followed by desorption.

PCP samples supplied by TWT Technologies included a 5% solution in oil (a typical solution used in applying PCP to utility poles), and wood shavings containing 52 ppm PCP.

Barringer Ionscan System Manager Software Version 2.209 was used to acquire and handle the data.

RESULTS AND DISCUSSIONS

Polychlorinated Biphenyls

PCBs have been investigated by IMS by Karasek et al. as early as 1971.[6,7] He identified the species giving rise to the observed peaks as $(C_{12}H_{10-x}Cl_x)(H_2O)H^+$ for the positive ions and as $(C_{12}H_{10-x}Cl_x)^-$ for the negative ions. He also noted that the response of his instrument to the PCBs increased with degree of chlorine substitution in the negative mode and decreased in the positive mode, both due to increasing electron affinity with higher degree of chlorine substitution.

Under our analysis conditions, the PCBs with less than five chlorine atoms did not lead to an IONSCAN response, but the PCBs containing five or more chlorine atoms gave strong signal responses for 35 ng PCB. Typical plasmagrams and growth curves (the signal response plotted vs. desorption time) are shown in Figures 1 - 4. (Note: Since the negative mode in the IONSCAN is the explosives mode, all figures illustrations are titled Explosives Plasmagrams) The results are summarized in Table 2.

Table 2

PCB	K_o	Drift Time (ms)	Peak Area [a]	Max. Peak Height [b]	Estimated LOD (ng)
Penta-Cl	1.1813	15.265	288	320	<5 ng
Hexa-Cl	1.1572	15.566	49	70	#
Hepta-Cl	1.1004	16.373	396	371	<5 ng
Octa-Cl	1.0443	17.267	274	238	<5 ng
Nona-Cl	1.0182	17.734	389	244	<5 ng
Deca-Cl	0.9964	18.127	221	193	<5 ng

see text [a] in digital units * ms [b] in digital units

(1 digital unit corresponds to ca. 1.4 x 10^9 ions)

Unlike Karasek, we did not observe an increase of response with higher chlorine substitution. All PCBs investigated gave rise to single peak plasmagrams with a reasonable correlation between inverse reduced mobility ($1/K_o$) and molecular mass as shown in Figure 5. The growth curves show that the PCBs desorb relatively quickly under the conditions used, despite their high boiling points. The relatively poor response to the hexachlorobiphenyl is likely to be due to an impurity in this standard suppressing the ionization of the biphenyl. From Table 2 data, limits of detection (LOD) for the single PCB isomers are estimated to be in the low ng region.

Figures 6 - 8 depict plasmagrams of examples of binary, ternary, and quaternary mixtures of PCBs (35 ng for each individual compound) and show the expected multiple peaks. Unless not available due to partial peak overlap, the integrated and maximum peak heights of each component in the mixtures were calculated and compared to the equivalent data from the single isomer analysis; the data are listed in Table 3. There is not an appreciable degree of suppression of the responses if mixtures are analyzed except in mixtures containing the hexachlorobiphenyl where the impurity in that solution seems to suppress responses of the other isomers as well. The comparisons are also less reliable if there is peak overlap.

Figures 9 and 10 show Aroclor 1254 and Aroclor 1260, respectively, analyzed from isooctane solutions. Aroclors are highly complex mixtures of PCBs, and the plasmagrams show peaks that can be interpreted as sums of the individual responses. Based on the results of the single PCB isomer analyses, the observed peaks in the plasmagram of Aroclor 1254 can be assigned to pentachlorobiphenyls (peak 110), and hexachlorobiphenyls or possibly a mixture of hexa- and heptachlorobiphenyls (peak 111). In the analysis of Aroclor 1260, the peaks 120 - 123 are most likely derived from penta-, hexa-, hepta-, and octachlorobiphenyls, respectively. The distribution of the peaks reflect the different components in these Aroclors, with 1260 having a higher proportion of more substituted PCB isomers. It is thus possible to distinguish different Aroclors with an IONSCAN, without the need for a lengthy GC profiling analysis.

IMS analysis of blank (unspiked) Volt-Esso 35 transformer oil, shown in Figure 11, reveals the presence of a large peak (peak 100). Such a large peak must be due to an additive, since Volt-Esso 35 is a hydrocarbon oil, and hydrocarbons do not give good responses in IMS. Further investigation confirmed that the oil contains 0.03% of 2,6-di-*t*-butyl-4-methylphenol (BHT), a common antioxidant. The plasmagram of BHT is shown in Figure 12, confirming that this additive to Volt-Esso 35 is responsible for the large peak. Unfortunately presence of this compound in PCB containing oil suppresses the ionization of PCBs by an order of magnitude, as demonstrated in the plasmagram of Volt-Esso 35 spiked with 350 ng of heptachlorobiphenyl (Figure 13). IMS data on transformer oil samples are summarized in Table 4.

Figures 14 and 15 show plasmagrams of Aroclors 1254 and 1260, respectively, dissolved in Volt-Esso 35. Again it can easily be seen by comparison with Figures 9 and 10 that the presence of BHT suppresses any PCB response. Further research will be conducted into ways to eliminate the interference and suppression from BHT, e.g. possibly a quick sample filtration to selectively adsorb BHT on basic media.

Table 3

Mixture of PCBs (# of Cl)	Compounds	Int. Peak Area [a]	Max. Peak Height [b]	Individual Int. Peak Area [a]	Individual Max. Peak Height [b]
5+6	Penta-Cl	n.a.	83	288	320
	Hexa-Cl	n.a.	59	49	70
5+7	Penta-Cl	187	209	288	320
	Hepta-Cl	273	244	396	371
5+8	Penta-Cl	193	304	288	320
	Octa-Cl	331	204	274	238
5+9	Penta-Cl	176	278	288	320
	Nona-Cl	326	243	389	244
5+10	Penta-Cl	232	305	288	320
	Deca-Cl	284	202	221	193
6+7	Hexa-Cl	90	89	49	70
	Hepta-Cl	152	149	396	371
7+8	Hepta-Cl	211	284	396	371
	Octa-Cl	248	172	274	238
8+9	Octa-Cl	n.a.	111	274	238
	Nona-Cl	n.a.	133	389	244
9+10	Nona-Cl	n.a.	148	389	244
	Deca-Cl	n.a.	98	221	193
5+7+9	Penta-Cl	141	232	288	320
	Hepta-Cl	147	185	396	371
	Nona-Cl	204	165	389	244
7+8+10	Hepta-Cl	223	256	396	371
	Octa-Cl	252	139	274	238
	Deca-Cl	168	122	221	193
8+9+10	Octa-Cl	n.a.	103	274	238
	Nona-Cl	n.a.	70	389	244
	Deca-Cl	n.a.	53	221	193
5+7+8+10	Penta-Cl	119	208	288	320
	Hepta-Cl	166	166	396	371
	Octa-Cl	190	117	274	238
	Deca-Cl	136	107	221	193

[a] in digital units * ms [b] in digital units

n.a. not available due to partial peak overlap

Table 4

Compound	35 ng in isooctane Peak Area [a]	Max. Peak Height [b]	35 ng in Volt-Esso Peak Area [a]	Max. Peak Height [b]	350 ng in Volt-Esso Peak Area [a]	Max. Peak Height [b]
Penta-Cl	288	320	#	#	#	#
Hexa-Cl	49	70	#	#	#	#
Hepta-Cl	396	371	39	62	382	645
Octa-Cl	274	238	&	&	&	&
Nona-Cl	389	244	&	&	&	&
Deca-Cl	221	193	27	49	166	233

[a] in digital units * ms   [b] in digital units

no detection due to direct overlap with BHT   & not carried out

Wood Preservatives

Figure 16 shows a plasmagram for the IMS analysis of a 5% PCP solution in oil, and Figure 17 depicts a plasmagram from wood chips containing 52 ppm PCP. Both demonstrate that PCP gives rise to strong single peak plasmagrams in the IONSCAN. The weaker adjacent peak 130 in Figure 17 is most likely a tetrachlorophenol, and a previously conducted GC/MS analysis of this sample had shown the presence of ca. 3 ppm of this substance.

Figure 17 shows that the LOD for PCP is much lower than 52 ppm; it is extrapolated to be in the neighbourhood of 1 ppm, making IMS ideally suited for the wood recycling application for which the PCP levels of product shavings should be less than 40 ppm, or possibly less than 5 ppm.

Further experiments with more samples will be conducted to enable calibration of the IONSCAN for PCP in the region between 0 and 50 ppm; also, tetrachlorophenols will be analyzed to confirm the identity of the extra peak in Figure 17.

CONCLUSIONS

It was shown that Barringer's IONSCAN is in principle useful in environmental analysis since it is able to detect PCBs and PCP at low levels; however, in the case of PCB analysis the presence of BHT in transformer oil presents a major problem that has to be solved before this application can be developed any further. On the other hand PCP analysis in wood recycling applications presents no problems, and the development of this application is continuing.

ACKNOWLEDGMENTS

The authors wish to thank Ron Massey of Ontario Hydro for providing the transformer oil standards, Andy Murray of Barringer Laboratories for samples of Aroclors in isooctane, and

Peter Fransham of TWT Technologies for the PCP and wood samples.

REFERENCES

1. Massey, R. Personal communication.

2. Harrison, R.O., Carlson, R.E., Weiss, A.J. Analysis of PCBs in Soil, Sediments, and Other Matrices by Enzyme Immunoassay. *Proceedings:* 1993 U.S. EPA/A&WMA International Symposium on Field Screening Methods for Hazardous Wastes and Toxic Chemicals, Las Vegas, Nevada, pp. 462-471.

3. Meil, J. Exploring the Resource - Timber Supplies & Life Cycle Analysis. Canadian Institute of Treated Wood: Treated Wood Life Cycle Workshop, Toronto, ON, June 1994.

4. Fransham, P. Chemical Extraction Processes. Canadian Institute of Treated Wood: Treated Wood Life Cycle Workshop, Toronto, ON, June 1994.

5. Lawrence, A.H. Rapid Characterization of Wood Species by Ion Mobility Spectrometry. *J. Pulp Pap. Sci.* **15**, J196-J199 (1989).

6. Karasek, F.W. Plasma Chromatography of the Polychlorinated Biphenyls. *Anal. Chem.* **43**, 1982-1986 (1971).

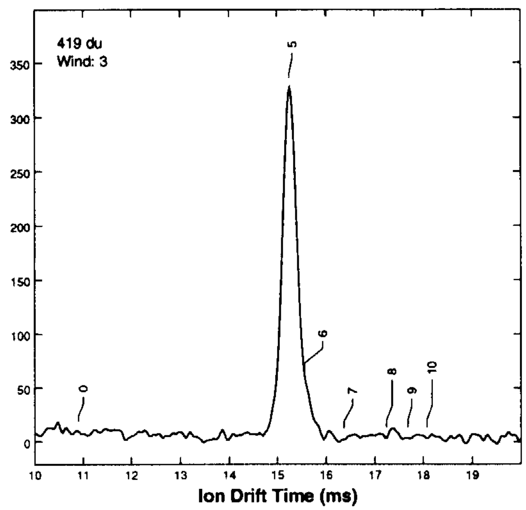
7. Karasek, F.W., Tatone, O.S., and Kane, D.M. Study of Electron Capture Behaviour of Substituted Aromatics by Plasma Chromatography. *Anal. Chem.* **45**, 1210-1214 (1973).

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: cl5bi-2 **Cls:** pcb **WTime:** 0.88 - 1.32 s
Sample: 1 ul (35 ng)

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.901	
5	Cl5-PCB	1.1813 *	15.245	*
6	Cl6-PCB	1.1572 *	15.562	*
7	Cl7-PCB	1.1004 *	16.365	*
8	Cl8-PCB	1.0443 *	17.245	*
9	Cl9-PCB	1.0182 *	17.687	*
10	Cl10-PCB	0.9964 *	18.074	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 14:04:16 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

Penta-Cl ALARM! Substances found: C15-PCB

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10901	215	N/A	N/A	
5	C15-PCB	1.1813	15253	320	+9	4	C15-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No: 350 Serial No: 8812
Comments:

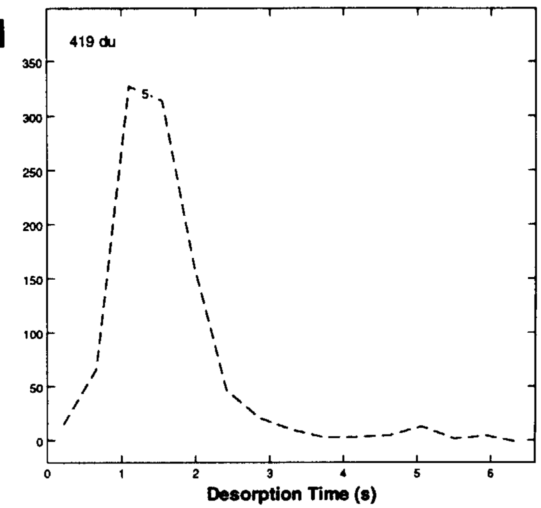
Figure 1
IMS analysis of 2,3',4,5',6-pentachlorobiphenyl

BARRINGER

IONSCAN™ Explosives Growth Curves

SampID: cl5bi-2 **Cls:** pcb
Sample: 1 ul (35 ng)

Pk2	PeakID	K_o	DTime
0 *	Cal	1.6520 *	10.901
5	Cl5-PCB	1.1813 *	15.245
6	Cl6-PCB	1.1572 *	15.562
7	Cl7-PCB	1.1004 *	16.365
8	Cl8-PCB	1.0443 *	17.245
9	Cl9-PCB	1.0182 *	17.687
10	Cl10-PCB	0.9964 *	18.074



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 14:04:16 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

Penta-Cl ALARM! Substances found: C15-PCB

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10901	215	N/A	N/A	
5	C15-PCB	1.1813	15253	320	+9	4	C15-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No: 350 Serial No: 8812
Comments:

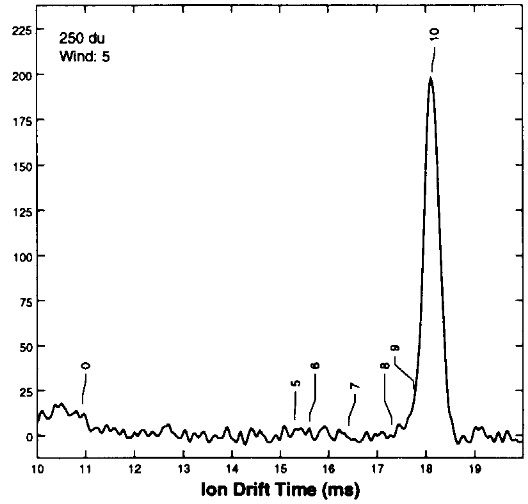
Figure 2
Growth curve of 2,3',4,5',6-pentachlorobiphenyl

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: cl10bi-2 **Cls:** pcb **WTime:** 1.76 - 2.20 s
Sample: 1 ul (35 ng)

Pk2	PeakID	K_o	DTime	
0 •	Cal	1.6520 •	10.933	
5	Cl5-PCB	1.1813 *	15.289	*
6	Cl6-PCB	1.1572 *	15.608	*
7	Cl7-PCB	1.1004 *	16.413	*
8	Cl8-PCB	1.0443 *	17.295	*
9	Cl9-PCB	1.0182 *	17.738	*
10	Cl10-PCB	0.9964 •	18.127	•



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 12:27:35 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

```
Deca-Cl  ALARM!  Substances found: Cl10-PCB
                         DTime   Max   Delta  # of  Detected Substances (Partly)
Chn  ChanID      Ko      (µs)    Amp   (µs)   Hits  Dependent upon this Channel
---  --------  ------   -----   -----  -----  ----  ----------------------------
 0   Cal       1.6520   10933    249    N/A   N/A
10   Cl10-PCB  0.9964   18121    193     -5     4   Cl10-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No: 8812
Comments:
```

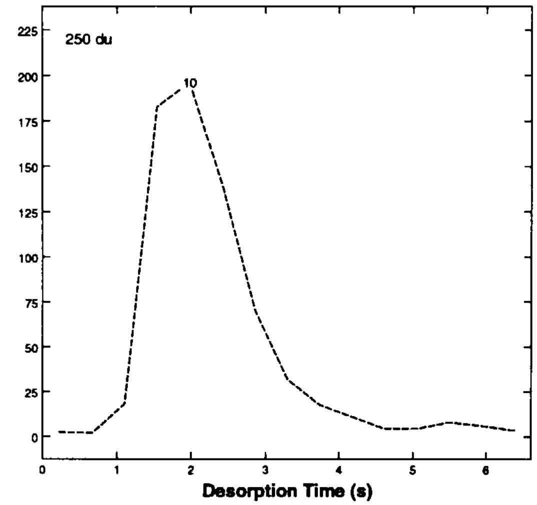
Figure 3
IMS analysis of decachlorobiphenyl

BARRINGER

IONSCAN™ Explosives Growth Curves

SampID: cl10bi-2 **Cls:** pcb
Sample: 1 ul (35 ng)

Pk2	PeakID	K_o	DTime	
0 •	Cal	1.6520 •	10.933	
5	Cl5-PCB	1.1813 *	15.289	
6	Cl6-PCB	1.1572 *	15.608	
7	Cl7-PCB	1.1004 *	16.413	
8	Cl8-PCB	1.0443 *	17.295	
9	Cl9-PCB	1.0182 *	17.738	
10	Cl10-PCB	0.9964 •	18.127	•



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 12:27:35 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

```
Deca-Cl  ALARM!  Substances found: Cl10-PCB
                         DTime   Max   Delta  # of  Detected Substances (Partly)
Chn  ChanID      Ko      (µs)    Amp   (µs)   Hits  Dependent upon this Channel
---  --------  ------   -----   -----  -----  ----  ----------------------------
 0   Cal       1.6520   10933    249    N/A   N/A
10   Cl10-PCB  0.9964   18121    193     -5     4   Cl10-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No: 8812
Comments:
```

Figure 4
Growth curve of decachlorobiphenyl

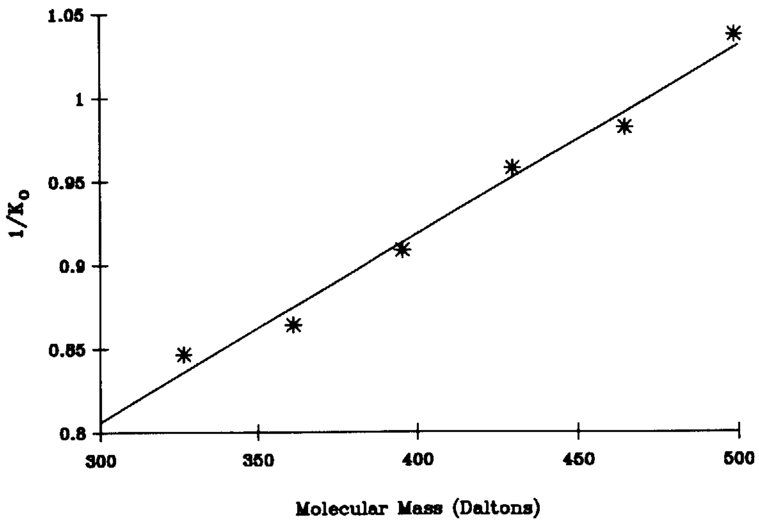


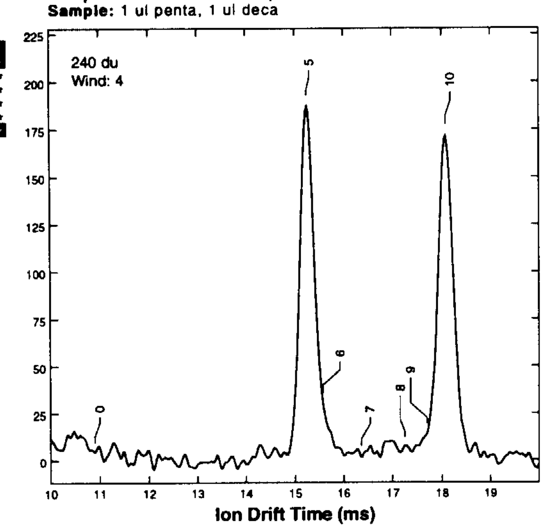
Figure 5
Correlation between inverse reduced mobility ($1/K_o$, in Vs/cm^2) and molecular mass of PCBs

BARRINGER

IONSCAN™ Explosives Plasmagram

Pk2	PeakID	K_0		DTime	
0 •	Cal	1.6520	•	10.901	
5	Cl5-PCB	1.1813	•	15.215	•
6	Cl6-PCB	1.1572	*	15.562	*
7	Cl7-PCB	1.1004	*	16.365	*
8	Cl8-PCB	1.0443	*	17.245	*
9	Cl9-PCB	1.0182	*	17.687	*
10	Cl10-PCB	0.9961	•	18.071	•

SampID: 2mixbi-5 **Cls:** pcb **WTime:** 1.32 - 1.76 s
Sample: 1 ul penta, 1 ul deca



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 14:26:35 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

```
Penta-Cl ALARM!  Substances found: C15-PCB
Deca-Cl  ALARM!  Substances found: C110-PCB
                                DTime   Max   Delta  # of  Detected Substances (Partly)
Chn   ChanID       Ko           (µs)    Amp   (µs)   Hits  Dependent upon this Channel
---   --------     -------      -----   ----  -----  ----  ---------------------------
 0    Cal          1.6520       10901   212    N/A   N/A
 5    C15-PCB      1.1813       15256   305    +12     4   C15-PCB
10    C110-PCB     0.9964       18073   202    +0      5   C110-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No:  8812
Comments:
35 ng of 2,3',4,5',6-Pentachlorobiphenyl plus
35 ng of Decachlorobiphenyl
```

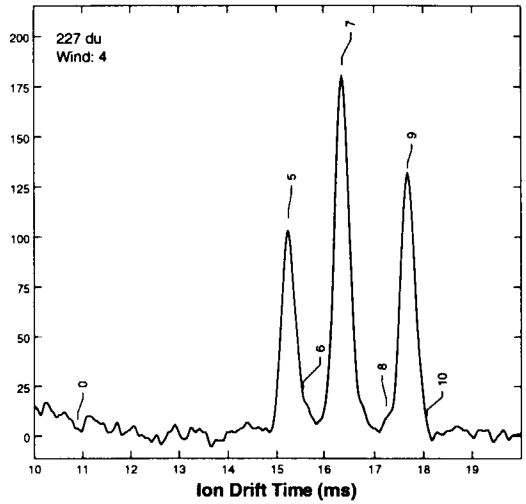
Figure 6
IMS analysis of a mixture of 2,3',4,5',6-pentachlorobiphenyl and decachlorobiphenyl

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: 3mixbi-1 **Cls:** pcb **WTime:** 1.32 - 1.76 s
Sample: 1 ul penta, 1 ul hepta, 1 ul nona

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.899	
5	Cl5-PCB	1.1813 *	15.242	*
6	Cl6-PCB	1.1572 *	15.559	*
7	Cl7-PCB	1.1004 *	16.362	*
8	Cl8-PCB	1.0443 *	17.241	*
9	Cl9-PCB	1.0182 *	17.683	*
10	Cl10-PCB	0.9964 *	18.070	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 14:50:34 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS ***** FAIL ***** No. of IONSCAN windows: 15

```
Penta-Cl ALARM!  Substances found: C15-PCB
Hepta-Cl ALARM!  Substances found: C17-PCB
Nona-Cl  ALARM!  Substances found: C19-PCB
                          DTime   Max   Delta  # of  Detected Substances (Partly)
Chn  ChanID      Ko       (µs)    Amp   (µs)   Hits  Dependent upon this Channel
---  --------  -------  -----   -----  -----  ----  ----------------------------
 0   Cal       1.6520   10899    214    N/A    N/A
 5   C15-PCB   1.1813   15248    232    +7       2  C15-PCB
 7   C17-PCB   1.1004   16365    185    +3       4  C17-PCB
 9   C19-PCB   1.0182   17687    165    +4       4  C19-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No: 8812
Comments:
35 ng of 2,3',4,5',6-Pentachlorobiphenyl plus
35 ng of 2,2',3,4,5,5',6-Heptachlorobiphenyl plus
35 ng of 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl
```

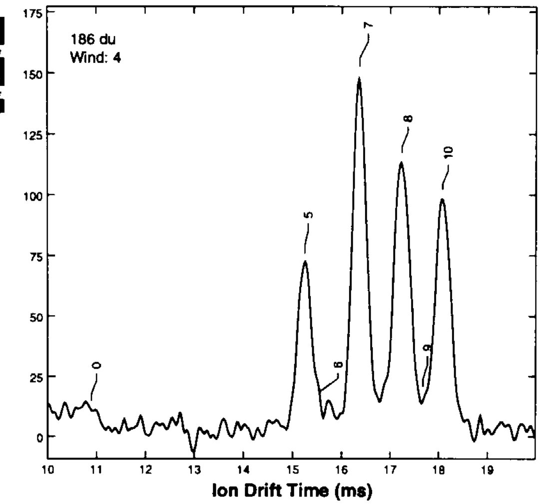
Figure 7
IMS analysis of a mixture of 2,3',4,5',6-pentachlorobiphenyl, 2,2',3,4,5,5',6-heptachlorobiphenyl, and 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: 4mixbi-1 **Cls:** pcb **WTime:** 1.32 - 1.76 s
Sample: 1 ul each of penta, hepta, octa, deca

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.898	
5	Cl5-PCB	1.1813 *	15.240	*
6	Cl6-PCB	1.1572 *	15.558	*
7	Cl7-PCB	1.1004 *	16.361	*
8	Cl8-PCB	1.0443 *	17.240	*
9	Cl9-PCB	1.0182 *	17.682	*
10	Cl10-PCB	0.9964 *	18.069	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 15:12:12 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS ***** FAIL ***** No. of IONSCAN windows: 15

```
Penta-Cl ALARM!  Substances found: C15-PCB
Hepta-Cl ALARM!  Substances found: C17-PCB
Octa-Cl  ALARM!  Substances found: C18-PCB
Deca-Cl  ALARM!  Substances found: C110-PCB
                          DTime   Max   Delta  # of  Detected Substances (Partly)
Chn  ChanID      Ko       (µs)    Amp   (µs)   Hits  Dependent upon this Channel
---  --------  -------  -----   -----  -----  ----  ----------------------------
 0   Cal       1.6520   10898    218    N/A    N/A
 5   C15-PCB   1.1813   15258    208    +18      3  C15-PCB
 7   C17-PCB   1.1004   16361    166    +1       4  C17-PCB
 8   C18-PCB   1.0443   17239    117    +0       4  C18-PCB
10   C110-PCB  0.9964   18080    107    +12      4  C110-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No: 8812
Comments:
35 ng of 2,3',4,5',6-Pentachlorobiphenyl plus
35 ng of 2,2',3,4,5,5',6-Heptachlorobiphenyl plus
35 ng of 2,2',3,3',4,4',5,5'-Octachlorobiphenyl plus
35 ng of Decachlorobiphenyl
```

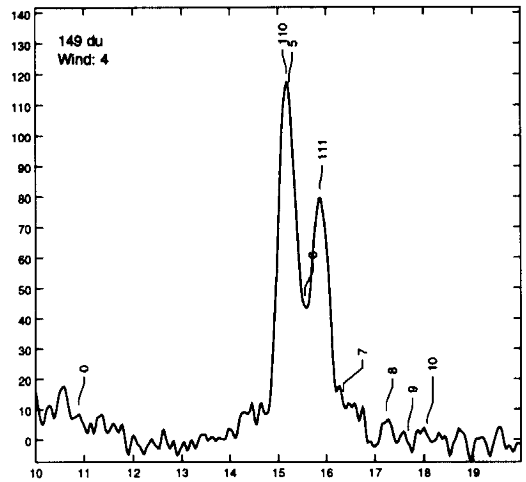
Figure 8
IMS analysis of a mixture of 2,3',4,5',6-pentachlorobiphenyl, 2,2',3,4,5,5',6-heptachlorobiphenyl, 2,2',3,3',4,4',5,5'-octachlorobiphenyl, and decachlorobiphenyl

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: 1254-1 **Cls:** pcb **WTime:** 1.32 - 1.76 s
Sample: 35 ng Aroclor 1254 in isooctane

Pk2	PeakID	K_0	DTime	
[illegible]	[illegible]	[illegible]	[illegible]	
5	Cl5-PCB	1.1813*	15.233	*
6	Cl6-PCB	1.1572*	15.551	*
7	Cl7-PCB	1.1004*	16.353	*
8	Cl8-PCB	1.0443*	17.232	*
9	Cl9-PCB	1.0182*	17.674	*
10	Cl10-PCB	0.9964*	18.060	*
110		1.1855*	15.179	*
111		1.1338*	15.872	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 15:30:41 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** PASS *** No. of IONSCAN windows: 15

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10893	222	N/A	N/A	

```
Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No: 8812
Comments:
```

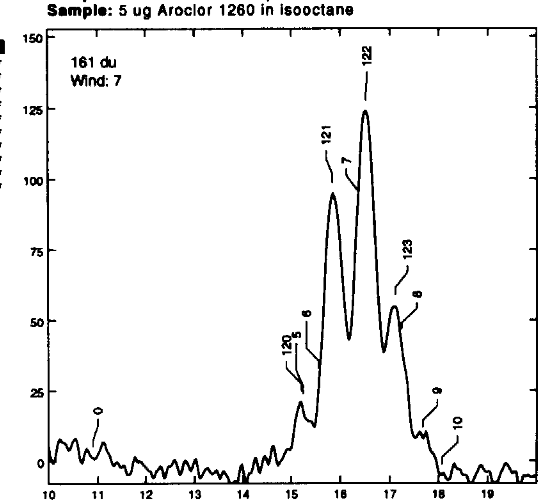
Figure 9
IMS analysis of Aroclor 1254 in isooctane

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: 1260-1 **Cls:** pcb **WTime:** 2.64 - 3.08 s
Sample: 5 ug Aroclor 1260 in isooctane

Pk2	PeakID	K_0	DTime	
[illegible]	[illegible]	[illegible]	[illegible]	
5	Cl5-PCB	1.1813*	15.243	*
6	Cl6-PCB	1.1572*	15.561	*
7	Cl7-PCB	1.1004*	16.364	*
8	Cl8-PCB	1.0443*	17.243	*
9	Cl9-PCB	1.0182*	17.685	*
10	Cl10-PCB	0.9964*	18.072	*
120		1.1843*	15.205	*
121		1.1364*	15.846	*
122		1.0905*	16.513	*
123		1.0529*	17.103	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 15:35:10 07/29/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** PASS *** No. of IONSCAN windows: 15

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10900	222	N/A	N/A	

```
Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350    Serial No: 8812
Comments:
```

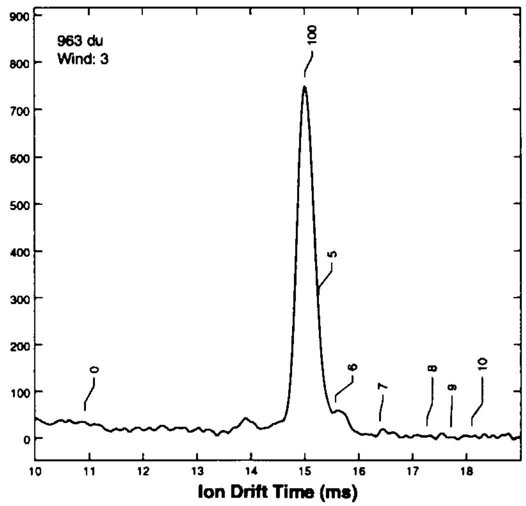
Figure 10
IMS analysis of Aroclor 1260 in isooctane

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: blank1 **Cls:** oil **WTime:** 0.88 - 1.32 s
Sample: Blank transformer oil

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.915	
5	Cl5-PCB	1.1813 *	15.264	*
6	Cl6-PCB	1.1572 *	15.582	*
7	Cl7-PCB	1.1004 *	16.386	*
8	Cl8-PCB	1.0443 *	17.267	*
9	Cl9-PCB	1.0182 *	17.709	*
10	Cl10-PCB	0.9964 *	18.097	*
100	BHT	1.2013 *	15.010	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 16:22:49 09/29/94
Desc: Model 350-8867 Neg Mode; 113,298,300 degC; 351,302,647 cc/min;

RESULTS OF IONSCAN ANALYSIS *** PASS *** No. of IONSCAN windows: 15

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10915	356	N/A	N/A	

```
Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No:  350   Serial No: 8867
Comments:
from Ontario Hydro
```

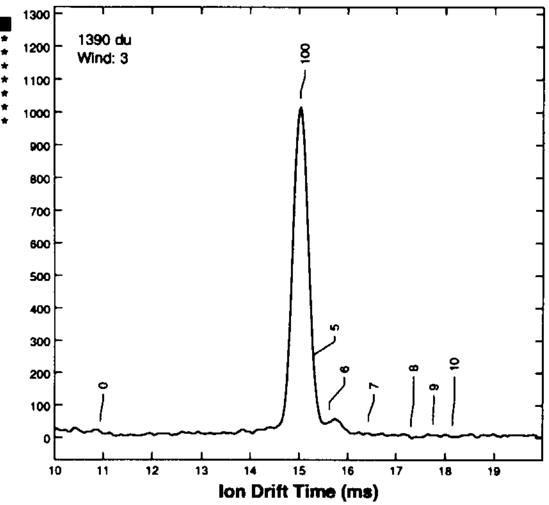
Figure 11
IMS analysis of blank Volt-Esso 35 transformer oil

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: BHT1 **Cls:** oil **WTime:** 0.88 - 1.32 s
Sample: Butylated hydroxytoluene

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.911	
5	Cl5-PCB	1.1813 *	15.301	*
6	Cl6-PCB	1.1572 *	15.619	*
7	Cl7-PCB	1.1004 *	16.425	*
8	Cl8-PCB	1.0443 *	17.308	*
9	Cl9-PCB	1.0182 *	17.751	*
10	Cl10-PCB	0.9964 *	18.140	*
100	BHT	1.2034 *	15.020	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 15:35:05 09/30/94
Desc: Model 350-8867 Neg Mode; 115,298,300 degC; 351,302,647 cc/min;

RESULTS OF IONSCAN ANALYSIS *** PASS *** No. of IONSCAN windows: 15

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10941	421	N/A	N/A	

```
Directory Pathname: c:\ism\pcb
IONSCAN Model No:  350   Serial No: 8867
Comments:
2,6-Di-tert.butyl-4-methylphenol, antioxidant
```

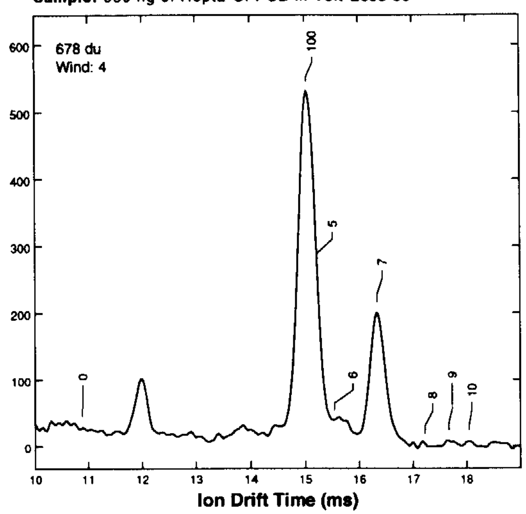
Figure 12
IMS analysis of BHT (2,6-di-*t*-butyl-4-methylphenol)

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: blank_7 **Cls:** oil **WTime:** 1.32 - 1.76 s
Sample: 350 ng of Hepta-Cl PCB in Volt-Esso 35

Pk2	PeakID	K_o	DTime	
0 •	Cal	1.6520 •	10.886	
5	Cl5-PCB	1.1813 *	15.224	
6	Cl6-PCB	1.1572 *	15.541	
7	Cl7-PCB	1.1004 •	16.343	*
8	Cl8-PCB	1.0443 *	17.221	
9	Cl9-PCB	1.0182 *	17.662	
10	Cl10-PCB	0.9964 *	18.049	
100	BHT	1.1963 *	15.033	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 17:00:49 09/29/94
Desc: Model 350-8867 Neg Mode; 115,298,300 degC; 351,302,647 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

Hepta-Cl ALARM! Substances found: Cl7-PCB

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10886	404	N/A	N/A	
7	Cl7-PCB	1.1004	16350	645	+8	5	Cl7-PCB

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No: 350 Serial No: 8867
Comments:

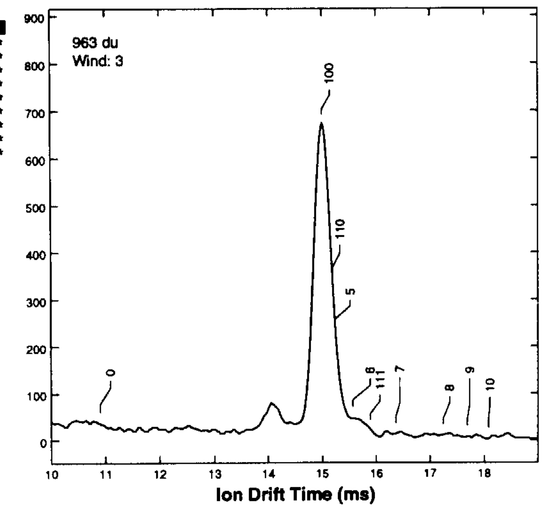
Figure 13
IMS analysis of 2,2',3,4,5,5',6-heptachlorobiphenyl in Volt-Esso 35

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: 1254-1 **Cls:** oil **WTime:** 0.88 - 1.32 s
Sample: 75 ppm Aroclor 1254 in Volt-Esso 35

Pk2	PeakID	K_o	DTime	
0 •	Cal	1.6520 •	10.902	
5	Cl5-PCB	1.1813 *	15.246	*
6	Cl6-PCB	1.1572 *	15.564	*
7	Cl7-PCB	1.1004 *	16.367	*
8	Cl8-PCB	1.0443 *	17.246	*
9	Cl9-PCB	1.0182 *	17.688	*
10	Cl10-PCB	0.9964 *	18.075	*
100	BHT	1.2007 *	15.000	*
110		1.1855 *	15.192	*
111		1.1338 *	15.885	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 16:25:42 09/29/94
Desc: Model 350-8867 Neg Mode; 114,298,300 degC; 351,302,647 cc/min;

RESULTS OF IONSCAN ANALYSIS *** PASS *** No. of IONSCAN windows: 15

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10902	384	N/A	N/A	

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No: 350 Serial No: 8867
Comments:
Peaks 110 and 111 refer to the Aroclor 1254 peaks in Figure 9

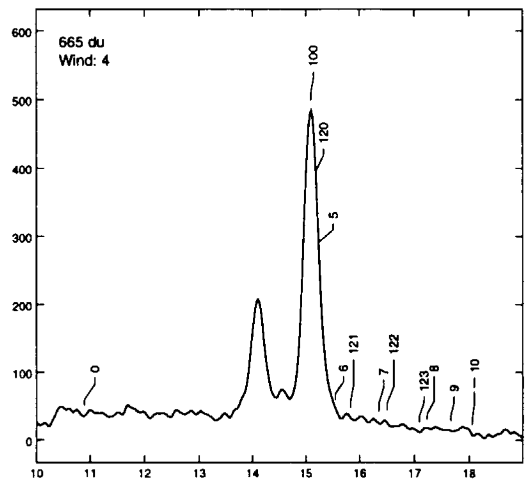
Figure 14
IMS analysis of Aroclor 1254 in Volt-Esso 35

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: 1260-1 **Cls:** oil **WTime:** 1.32 - 1.76 s
Sample: 50 ppm Aroclor 1260 in Volt-Esso 35

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.892	*
5	Cl5-PCB	1.1813 *	15.232	*
6	Cl6-PCB	1.1572 *	15.549	*
7	Cl7-PCB	1.1004 *	16.352	*
8	Cl8-PCB	1.0443 *	17.230	*
9	Cl9-PCB	1.0182 *	17.672	*
10	Cl10-PCB	0.9964 *	18.059	*
100	BHT	1.1922 *	15.093	*
120		1.1843 *	15.193	*
121		1.1364 *	15.834	*
122		1.0905 *	16.500	*
123		1.0529 *	17.090	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 16:28:19 09/29/94
Desc: Model 350-8867 Neg Mode; 115,298,300 degC; 351,302,647 cc/min;

RESULTS OF IONSCAN ANALYSIS *** PASS *** No. of IONSCAN windows: 15

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10892	402	N/A	N/A	

Directory Pathname: g:\research\pcb\negmode
IONSCAN Model No: 350 Serial No: 8867
Comments:
Peaks 120 - 123 refer to the Aroclor 1260 peaks in Figure 10

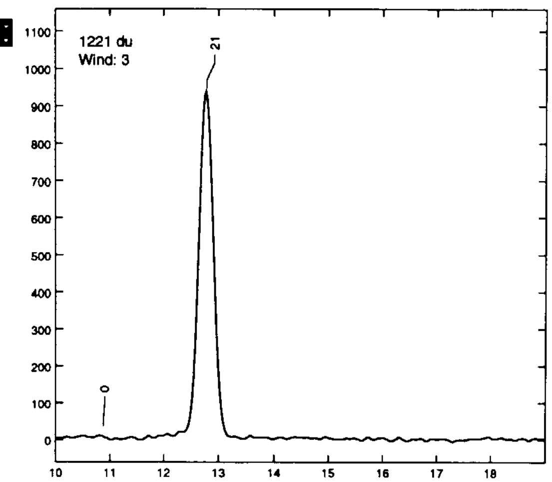
Figure 15
IMS analysis of Aroclor 1260 in Volt-Esso 35

BARRINGER

IONSCAN™ Explosives Plasmagram

SampID: twt_3 **Cls:** pcp **WTime:** 0.88 - 1.32 s
Sample: Liquid PCP solution in oil

Pk2	PeakID	K_o	DTime	
0 *	Cal	1.6520 *	10.878	*
21	PCPhenol	1.4053 *	12.788	*



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25µs **T:** 22ms **Alg:** 1 **Time:** 10:50:45 07/18/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS *** FAIL *** No. of IONSCAN windows: 15

PCPhenol ALARM! Substances found: PCPhenol

Chn	ChanID	Ko	DTime (µs)	Max Amp	Delta (µs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10878	345	N/A	N/A	
21	PCPhenol	1.4053	12777	1032	-10	10	PCPhenol

Directory Pathname: g:\research\wood
IONSCAN Model No: 350 Serial No: 8812
Comments:

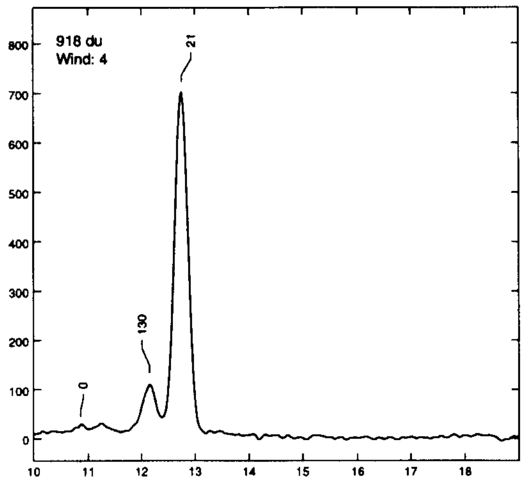
Figure 16
IMS analysis of pentachlorophenol in oil

BARRINGER

IONSCAN™ Explosives Plasmagram

Pk2	PeakID	K_0	DTime	
0 *	Cal	1.6520 *	10.852	*
21	PCPhenol	1.4053 *	12.75[illegible]	*
130	?	1.4760 *	12.146	*

SampID: twt_5 **Cls:** pcp **WTime:** 1.32 - 1.76 s
Sample: Processed wood shavings (52 ppm PCP)



Wnds: 15 **Scans:** 20 **Pts:** 856 **Per:** 25μs **T:** 22ms **Alg:** 1 **Time:** 12:16:39 07/18/94
Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cc/min;

RESULTS OF IONSCAN ANALYSIS ***** FAIL ***** No. of IONSCAN windows: 15

PCPhenol ALARM! Substances found: PCPhenol

Chn	ChanID	Ko	DTime (μs)	Max Amp	Delta (μs)	# of Hits	Detected Substances (Partly) Dependent upon this Channel
0	Cal	1.6520	10852	270	N/A	N/A	
21	PCPhenol	1.4053	12744	769	-13	15	PCPhenol

Directory Pathname: g:\research\wood
IONSCAN Model No: 350 Serial No: 8812
Comments:

Figure 17
IMS analysis of wood shavings containing 52 ppm PCP