ENVIRONMENTAL APPLICATIONS FOR ION MOBILITY SPECTROMETRY

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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, immunoassay² 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. 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.

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.

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

<table>
<thead>
<tr>
<th>Ion Mode</th>
<th>PCB Analysis</th>
<th>PCP Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloride Reactant Addition</td>
<td>Negative *</td>
<td>Negative *</td>
</tr>
<tr>
<td>Drift Tube Temperature</td>
<td>115°C</td>
<td>120°C</td>
</tr>
<tr>
<td>Inlet Temperature</td>
<td>300°C</td>
<td>300°C</td>
</tr>
<tr>
<td>Desorber Temperature</td>
<td>300°C</td>
<td>300°C</td>
</tr>
</tbody>
</table>

* 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'-Hexachlorobiphenyl (#185), 2,2',3,3',6,7,8,8'-Tetrachlorobiphenyl (#194), 2,2',3,4,4',5,5'-Octachlorobiphenyl (#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. He identified the species giving rise to the observed peaks as \((\text{C}_{12}\text{H}_{10}\text{Cl}_n\text{H}_2\text{O})\text{H}^+\) for the positive ions and as \((\text{C}_{12}\text{H}_{10}\text{Cl}_n\text{Cl})\) 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

<table>
<thead>
<tr>
<th>PCB</th>
<th>(K_o)</th>
<th>Drift Time (ms)</th>
<th>Peak Area (^a)</th>
<th>Max. Peak Height (^b)</th>
<th>Estimated LOD (ng)</th>
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</thead>
<tbody>
<tr>
<td>Penta-Cl</td>
<td>1.1813</td>
<td>15.265</td>
<td>288</td>
<td>320</td>
<td>&lt;5 ng</td>
</tr>
<tr>
<td>Hexa-Cl</td>
<td>1.1572</td>
<td>15.566</td>
<td>49</td>
<td>70</td>
<td>#</td>
</tr>
<tr>
<td>Hepta-Cl</td>
<td>1.1004</td>
<td>16.373</td>
<td>396</td>
<td>371</td>
<td>&lt;5 ng</td>
</tr>
<tr>
<td>Octa-Cl</td>
<td>1.0443</td>
<td>17.267</td>
<td>274</td>
<td>238</td>
<td>&lt;5 ng</td>
</tr>
<tr>
<td>Nona-Cl</td>
<td>1.0182</td>
<td>17.734</td>
<td>389</td>
<td>244</td>
<td>&lt;5 ng</td>
</tr>
<tr>
<td>Deca-Cl</td>
<td>0.9964</td>
<td>18.127</td>
<td>221</td>
<td>193</td>
<td>&lt;5 ng</td>
</tr>
</tbody>
</table>

\(^a\) in digital units \(^b\) in digital units

(1 digital unit corresponds to ca. \(1.4 \times 10^9\) 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-i-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>
<thead>
<tr>
<th>Mixture of PCBs (# of Cl)</th>
<th>Compounds</th>
<th>Int. Peak Area *</th>
<th>Max. Peak Height b</th>
<th>Individual Int. Peak Area *</th>
<th>Individual Max. Peak Height b</th>
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<tr>
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<tr>
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<tr>
<td>5+10</td>
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<td>Deca-Cl</td>
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<td>107</td>
<td>221</td>
<td>193</td>
</tr>
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</table>

* in digital units  * ms  b in digital units

n.a. not available due to partial peak overlap
Table 4

<table>
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<tr>
<th>Compound</th>
<th>35 ng in isooctane</th>
<th>35 ng in Volt-Esso</th>
<th>350 ng in Volt-Esso</th>
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<tr>
<td></td>
<td>Peak Area *</td>
<td>Max. Peak Height b</td>
<td>Peak Area *</td>
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<tr>
<td>Octa-Cl</td>
<td>274</td>
<td>238</td>
<td>&amp;</td>
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<td>&amp;</td>
</tr>
<tr>
<td>Deca-Cl</td>
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<td>193</td>
<td>27</td>
</tr>
</tbody>
</table>

* in digital units  
* in digital units  
* 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
REFERENCES

1. Massey, R. Personal communication.


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

Figure 2
Growth curve of 2,3',4,5',6-pentachlorobiphenyl
IONSCAN™ Explosives Plasmagram

Sample ID: c10bi-2  Class: pcb  WTime: 1.76 - 2.20 s
Sample: 1 ul (35 ng)

Peak  Kd  DTime
1  3.812  2.804  250 du
Wnd: 5

Ion Drift Time (ms)

Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cpm/min;

RESULTS OF IONSCAN ANALYSIS

Deca-C1 ALARM! Substances found: C110-PCB

Chn  ChnID  Kd  Dime  Max Delta  # of Detected Substances (Partly)  Depth (cm)  Time (min)  Heat (°C)
0  Cal  1.6520  1093  193  4  C110-PCB

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

Figure 3
IMS analysis of decachlorobiphenyl

IONSCAN™ Explosives Growth Curves

Sample ID: c10bi-2  Class: pcb  WTime: 1.76 - 2.20 s
Sample: 1 ul (35 ng)

Peak  Kd  DTime
5  GS-PCB  1.1813  15.289
6  CB-PCB  1.1572  16.808
7  CO-PCB  1.0464  17.325
8  CI-PCB  1.0465  17.229
9  C110-PCB  1.0465  17.229

Desorption Time (s)

Desc: Model 350-8812 Neg Mode; 115,298,300 degC; 351,302,643 cpm/min;

RESULTS OF IONSCAN ANALYSIS

Deca-C1 ALARM! Substances found: C110-PCB

Chn  ChnID  Kd  Dime  Max Delta  # of Detected Substances (Partly)  Depth (cm)  Time (min)  Heat (°C)
0  Cal  1.6520  1093  193  4  C110-PCB

Directory Pathname: g:\research\pcbinemode
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/cm²) and molecular mass of PCBs

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

Figure 8
IMS analysis of a mixture of 2,3',4,5',6-pentachlorobiphenyl, 2,2',3,4,5,5'-hexachlorobiphenyl, 2,2',3,3',4,4',5,5'-octachlorobiphenyl, and decachlorobiphenyl
**Figure 9**
IMS analysis of Aroclor 1254 in isooctane

**Figure 10**
IMS analysis of Aroclor 1260 in isooctane
**IONS CAN™ Explosives Plasmagram**

**Sample:** blank 1  
**Clas:** oil  
**Sample:** Blank transformer oil  
**WTime:** 0.88 - 1.32 s

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<tr>
<th>PeakID</th>
<th>K&lt;br&gt;</th>
<th>DTime</th>
<th>Max</th>
<th>Delta</th>
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<td>1200</td>
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<td>1200</td>
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<td>15.010</td>
<td>800</td>
<td>300</td>
<td>1200</td>
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</table>

**RESULTS OF IONS CAN ANALYSIS**

**No. of IONS CAN windows:** 15

1. **Chn:** 1  
2. **ChanID:** 1  
3. **Ko:** 0  
4. **DTime:** 1.6520 *  
5. **Max:** 10915  
6. **Delta:** 356  
7. **No:** N/A  
8. **Detected Substances (Partly):** N/A

**Directory Pathname:** g:\research\pcb\negmode

**IONS CAN Model No:** 350  
**Serial No:** 8867

**Comments:**

- From Ontario Hydro

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**IONS CAN™ Explosives Plasmagram**

**Sample:** BHT 1  
**Clas:** oil  
**Sample:** Butylated hydroxytoluene  
**WTime:** 0.88 - 1.32 s

<table>
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<th>PeakID</th>
<th>K&lt;br&gt;</th>
<th>DTime</th>
<th>Max</th>
<th>Delta</th>
<th>of Detected Substances (Partly)</th>
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<td>15.301</td>
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<td>1200</td>
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**RESULTS OF IONS CAN ANALYSIS**

**No. of IONS CAN windows:** 15

1. **Chn:** 1  
2. **ChanID:** 1  
3. **Ko:** 0  
4. **DTime:** 1.6520 *  
5. **Max:** 10941  
6. **Delta:** 421  
7. **No:** N/A  
8. **Detected Substances (Partly):** N/A

**Directory Pathname:** c:\ism\pcb

**IONS CAN Model No:** 350  
**Serial No:** 8867

**Comments:**

- 2,6-Di-tert-butyl-4-methylphenol, antioxidant

---

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

**Figure 12**
IMS analysis of BHT (2,6-di-tert-butyl-4-methylphenol)
Figure 13
IMS analysis of 2,2'-3,4,5,5',6-heptachlorobiphenyl in Volt-Esso 35

Figure 14
IMS analysis of Aroclor 1254 in Volt-Esso 35
**Figure 15**

IMS analysis of Aroclor 1260 in Volt-Essco 35

**Figure 16**

IMS analysis of pentachlorophenol in oil
**Figure 17**

IMS analysis of wood shavings containing 52 ppm PCP