Capillary GC Analyses of Chlorinated Pesticides in Apples

Based on federal and state regulations for identifying and quantifying low levels of pesticides in food and environmental samples, we selected three capillary columns to screen for chlorinated pesticides. A nonpolar and two low/intermediate polarity phases were chosen to evaluate differences in component elution order and retention times. Low level screening analyses were performed effectively by using split/splitless injection and an electron capture detector (ECD). Each column separated the 18 pesticides in approximately 35 minutes. Example chromatograms are shown.

Key Words:
- pesticides
- aldrin
- endrin
- methoxychlor
- fruit

Federal and state regulations require that pesticides in food and environmental samples be identified and quantified at low levels. Based on these regulations, we selected three capillary GC columns to screen for pesticides at low levels. We chose a nonpolar phase, PTE™-5, and two low/intermediate polarity phases, SPB™-608 and SPB-1701, to illustrate the differences in component elution order and retention times for chlorinated pesticides.

Eighteen chlorinated pesticides (Table 1) were spiked into, then extracted from, apples purchased at a local grocery store. Extracts were prepared by weighing out 50 grams of fruit, blending it, and adding 100mL of acetonitrile. The fruit extract was reextracted in hexane, using a partitioning process (1). The untreated extracts contained no pesticides.

Samples of the spiked and unspiked extracts were injected onto each capillary column under the conditions listed in Figure A. Figure A shows chromatograms of the extracted pesticides from each column. The low/intermediate polarity SPB-608 and SPB-1701 columns selectively eluted the analytes, based on dipole-dipole and hydrogen bonding interactions between the solute and the stationary phase. Each column separated all of the analytes. The nonpolar PTE-5 column eluted the analytes by boiling point. One pair of analytes (endosulfan sulfate and 4,4'-DDT) coeluted from the nonpolar column. Analysis time for each column was 31-36 minutes. Elution order differences among the three columns are evident in Figure A. In a two-column analysis, these differences can be used to help confirm the identities of the analytes.

Table 2 lists the recovery values for the pesticides, determined using the SPB-608 column. Recovery of the spiked analytes ranged from 15% to 122%.

Based on these evaluations, we determined that the three stationary phases, PTE-5, SPB-608, and SPB-1701, exhibited differences in retention times, resolution, and elution order for 18 common chlorinated pesticides. Using these columns, screening analyses for low levels of these pesticides can be performed effectively, in approximately 35 minutes, with split/splitless injection and an electron capture detector (ECD).

Table 1. Chlorinated Pesticide Standards Mixture
(TCL Pesticides Mix, Cat. No. 4-8913, 2000µg/mL each component in toluene:hexane, 50:50)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aldrin</td>
<td>122</td>
</tr>
<tr>
<td>α-BHC</td>
<td>70</td>
</tr>
<tr>
<td>β-BHC</td>
<td>56</td>
</tr>
<tr>
<td>γ-BHC</td>
<td>35</td>
</tr>
<tr>
<td>δ-BHC</td>
<td>111</td>
</tr>
<tr>
<td>4,4'-DDD</td>
<td>53</td>
</tr>
<tr>
<td>4,4'-DDE</td>
<td>112</td>
</tr>
<tr>
<td>4,4'-DDT</td>
<td>29</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>89</td>
</tr>
<tr>
<td>Endosulfan I</td>
<td>101</td>
</tr>
<tr>
<td>Endosulfan II</td>
<td>71</td>
</tr>
<tr>
<td>Endosulfan sulfate</td>
<td>15</td>
</tr>
<tr>
<td>Endrin</td>
<td>98</td>
</tr>
<tr>
<td>Endrin aldehyde</td>
<td>88</td>
</tr>
<tr>
<td>Endrin ketone</td>
<td>—</td>
</tr>
<tr>
<td>Heptachlor</td>
<td>47</td>
</tr>
<tr>
<td>Heptachlor epoxide</td>
<td>91</td>
</tr>
<tr>
<td>Methoxychlor</td>
<td>41</td>
</tr>
</tbody>
</table>

Table 2. Recovery of Chlorinated Pesticides from Apples (SPB-608 Column)

Table 2 lists the recovery values for the pesticides, determined using the SPB-608 column. Recovery of the spiked analytes ranged from 15% to 122%.

Based on these evaluations, we determined that the three stationary phases, PTE-5, SPB-608, and SPB-1701, exhibited differences in retention times, resolution, and elution order for 18 common chlorinated pesticides. Using these columns, screening analyses for low levels of these pesticides can be performed effectively, in approximately 35 minutes, with split/splitless injection and an electron capture detector (ECD).
**Figure A. Chlorinated Pesticides from Apples**

Stationary Phases: PTE-5, SPB-608, SPB-1701

Column Dimensions: 30 m x 0.25 mm ID, 0.25 µm phase film

Catalog Nos.: 24135-U (PTE-5), 24103-U (SPB-608), 24113 (SPB-1701)

Oven: 100°C to 280°C at 6°C/min
Carrier: helium, 40 cm/sec.
Det.: ECD, 250°C
Sample: 1 µL of 0.1 µg/mL extract, split/spillless 45 sec, 250°C

1. Aldrin
2. α-BHC
3. β-BHC
4. γ-BHC
5. δ-BHC
6. 4,4'-DDD
7. 4,4'-DDE
8. DDT
9. Dieldrin
10. Endosulfan I
11. Endosulfan II
12. Endosulfan sulfate
13. Endrin
14. Endrin aldehyde
15. Endrin ketone
16. Heptachlor
17. Heptachlor epoxide
18. Methoxychlor

*Note: For a suitable extraction procedure, refer to AOAC Methods, 16th edition (Order from AOAC International, 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877-2504 USA, Tel.: +1-301-924-7077; Fax: +1-301-924-7089.)

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**Ordering Information:**

**Fused Silica Capillary Columns**
all 30m x 0.25mm ID, 0.25µm phase film

- PTE-5: 24135-U
- SPB-608: 24103-U
- SPB-1701: 24113

**Chlorinated Pesticides Mixture**
(TCL Pesticides Mix)
2000 µg/mL each analyte in Figure A in toluene:hexane, 50:50

**Cat. No.**
48913

**Reference**

For a suitable extraction procedure, refer to AOAC Methods, 16th edition (Order from AOAC International, 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877-2504 USA, Tel.: +1-301-924-7077; Fax: +1-301-924-7089.)

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