Capillary GC Analyses of Triazine 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 triazine pesticides. A nonpolar and two low/intermediate polar 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 a thermal specific detector (TSD). Each column separated the triazines in less than 30 minutes. Example chromatograms are shown.

Key Words:
- triazines
- atrazine
- simazine
- 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 triazine 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 at least partially separated all of the analytes (prometon and simazine were incompletely separated by the SPB-1701 column). The nonpolar PTE-5 column eluted the analytes by boiling point. One pair of analytes (simazine and atrazine) coeluted from the nonpolar column. Analysis time for each column was approximately 28 minutes.

Table 2 lists the recovery values for the triazine pesticides, determined using the SPB-608 column. Recovery of the spiked analytes ranged from 103% to 122%. Limits of detection for the standards ranged from 0.1 ppm to 10.0 ppm.

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 six common triazine pesticides. Using these columns, screening analyses for low levels of triazine pesticides can be performed effectively, in less than 30 minutes, with split/splitless injection and a thermal specific detector (TSD).

Table 1. Triazine Pesticide Standards Mixture (1000 µg/mL each component in methanol)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Recovery (%)</th>
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<tbody>
<tr>
<td>Atrazine</td>
<td>120</td>
</tr>
<tr>
<td>Prometon</td>
<td>110</td>
</tr>
<tr>
<td>Prometryn</td>
<td>103</td>
</tr>
<tr>
<td>Simazine</td>
<td>113</td>
</tr>
<tr>
<td>Simetryn</td>
<td>122</td>
</tr>
<tr>
<td>Terbutryn</td>
<td>109</td>
</tr>
</tbody>
</table>

Triazine 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 100 mL of acetonitrile. The fruit extract was reextracted in hexane, using a partitioning process (1). The untreated extracts contained no pesticides.
**Figure A. Triazine Pesticides from Apples**

Stationary Phases: PTE-5, SPB-608, SPB-1701  
Column Dimensions: 30m x 0.25mm ID, 0.25µm phase film  
Catalog Nos.: 24135-U (PTE-5), 24103-U (SPB-608), 24113 (SPB-1701)  
Oven: 80°C (1 min) to 280°C at 6°C/min  
Carrier: helium, 40cm/sec  
Det.: thermal specific, 250°C  
Sample: 1µL of 10µg/mL extract, split/splitless 45 sec, 200°C

1. Atrazine  
2. Prometon  
3. Prometryn  
4. Simazine  
5. Simetryn  
6. Terbutryn

<table>
<thead>
<tr>
<th>Description</th>
<th>Cat. No.</th>
</tr>
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<tbody>
<tr>
<td>Fused Silica Capillary Columns</td>
<td></td>
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<tr>
<td>all 30m x 0.25mm ID, 0.25µm phase film</td>
<td></td>
</tr>
<tr>
<td>PTE-5</td>
<td>24135-U</td>
</tr>
<tr>
<td>SPB-608</td>
<td>24103-U</td>
</tr>
<tr>
<td>SPB-1701</td>
<td>24113</td>
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</tbody>
</table>

**Triazine Pesticide Standards**  
neat, 100mg  
Atrazine | 49085  
Prometon | 49086  
Prometryn | 49087  
Simazine | 49089  
Simetryn | 49090-U  
Terbutryn | 49091

**Reference**  

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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