3-MCPD and glycidyl esters in edible oils are contaminants that are formed through refining processes. Several of these substances have been classified as possible human carcinogens. Methods, which are similar to one another, have been developed by ISO, AOCS, and DGf for analyzing these contaminants. While these methods cover extraction and derivatization techniques in detail, very little attention is paid to the GC-MS methods. With emerging automated systems, it is important to simplify and speed up the instrument method by optimizing the parameters, to include evaluating split injection.

### Optimization of GC-MS method

<table>
<thead>
<tr>
<th>Initial temp. (°C)</th>
<th>Splitless</th>
<th>RT1 (min)</th>
<th>RT2 (min)</th>
<th>Width1</th>
<th>Width2</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>8.03</td>
<td>8.07</td>
<td>0.030</td>
<td>0.035</td>
<td>0.762</td>
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</tr>
<tr>
<td>100</td>
<td>7.62</td>
<td>7.66</td>
<td>0.022</td>
<td>0.034</td>
<td>0.864</td>
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<tr>
<td>110</td>
<td>6.82</td>
<td>6.86</td>
<td>0.023</td>
<td>0.022</td>
<td>1.075</td>
<td></td>
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<tr>
<td>115</td>
<td>6.42</td>
<td>6.46</td>
<td>0.025</td>
<td>0.025</td>
<td>0.944</td>
<td></td>
</tr>
<tr>
<td>120</td>
<td>6.04</td>
<td>6.07</td>
<td>0.027</td>
<td>0.027</td>
<td>0.830</td>
<td></td>
</tr>
</tbody>
</table>

### Pro EZGC Model on Rxi-17Sil MS 30×0.25x0.25

**Pro EZGC**

Pro EZGC is a free GC modeler that allows for fast and simple optimization our analysis. Compounds currently present in the system: 3-MCPD and 3-MCPD-d5 derivatized with PBA.

- 2-MCPD and 2-MCPD-d5 derivatized with PBA.
- Glycidyl and glycidyl-d5 as 3-MBPD and 3-MBPD-d5 (resp.) derivatized with PBA.
- Phenyl boronic acid (PBA).

**MCPD and GE analysis**

**Rxi-175Sil MS 30mx0.25x0.25**

**Evaluation on GC-MS/MS**

- LOD = 0.02 mg/kg
- S/N = 5
- 12 μg/Kg

**Changing the GC-MS Method**

**Original method:**
- Temp program: 85°C (0.5), 6°C/min to 150°C, 12°C/min to 180°C, 25°C/min to 280°C (7); total time: 24.8 min
- Splitless time: 0.5-1 min

**New, optimized method:**
- Temp program: 120°C (0.5), 12°C/min to 180°C, 25°C/min to 330°C (5); total time: 16.5 min
- Split: 10:1

**Conclusions**

- An optimized GC-MS method led to improved peak shapes without detrimental effect on resolution.
- Manually optimized temperature program saved 8 minutes per analysis. EZGC model can save up to 20 minutes.
- Switching to split injection had no negative effect on limits of detection.
- Using regular split/splitless injector had no effect on the performance. However, using guard column is recommended.