Analysis of Mineral Oil by GC-FID

Method 1:

Instrumentation: Agilent 6890 GC with FID and PTV Inlet

Column: Varian Select Mineral Oil LVI, 15 m x 0.32 mm

Injection Mode: Programmable Temperature Vaporizer (PTV) Injector, split/splitless as follows:

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Split state</th>
<th>Split ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>ON</td>
<td>75</td>
</tr>
<tr>
<td>0.45</td>
<td>OFF</td>
<td>100</td>
</tr>
<tr>
<td>3.00</td>
<td>ON</td>
<td>150</td>
</tr>
</tbody>
</table>

Carrier Gases: Helium at variable pressure as follows:

<table>
<thead>
<tr>
<th>Rate (psi/min)</th>
<th>Step (psi)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>7.0</td>
<td>1.25</td>
</tr>
<tr>
<td>400.0</td>
<td>15.0</td>
<td>1.72</td>
</tr>
<tr>
<td>400.0</td>
<td>7.0</td>
<td>0.98</td>
</tr>
<tr>
<td>2.0</td>
<td>10.9</td>
<td>0.00</td>
</tr>
<tr>
<td>1.7</td>
<td>14.3</td>
<td>0.00</td>
</tr>
<tr>
<td>1.7</td>
<td>17.8</td>
<td>1.11</td>
</tr>
</tbody>
</table>

Total Time 12.24

Col./Oven Temp: 35 °C to 350 °C/min, as follows:

<table>
<thead>
<tr>
<th>Rate (psi/min)</th>
<th>Step (psi)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>35.0</td>
<td>4.00</td>
</tr>
<tr>
<td>60.0</td>
<td>150.0</td>
<td>0.00</td>
</tr>
<tr>
<td>50.0</td>
<td>250.0</td>
<td>0.00</td>
</tr>
<tr>
<td>30.0</td>
<td>350.0</td>
<td>1.50</td>
</tr>
</tbody>
</table>

Total Time 12.25

Retention Gap: 2.5 m x 0.53 mm

Liner: Varian 1079 LVI Liner

Injection volume: 70 µL

GC Detection: FID at 350 °C

Sample Preparation:
All samples were prepared by dissolving the alkanes and the reference sample in hexane.

Results:
The ratios C10/C20 peak area and C40/C20 peak area were measured. Both values were between 0.8 and 1.2. The ratios are much higher when using the Varian 1079 LVI Liner versus a regular liner.
Repeatability data of a Test Sample Mineral Oil Standard from the National Institute for Public Health and the Environment (RIVM) (CP741970) showed a 1.1 % RSD for the hydrocarbon ratios as below:
Run | Area/C10-C20 fraction | Area/C10-C20 fraction | HC Ratio |
--- | --- | --- | --- |
1 | 1201944.7 | 1860482.4 | 1.548 |
2 | 1208160.4 | 1867571.7 | 1.546 |
3 | 1205740 | 1846199.5 | 1.531 |
4 | 1212651.5 | 1826599.8 | 1.506 |
5 | 1194517 | 1850616.1 | 1.549 |
6 | 1190457.6 | 1854374.9 | 1.558 |
7 | 1189986 | 1851458.5 | 1.556 |
8 | 1193781.4 | 1852083.6 | 1.551 |
Average | 1199654.825 | 1851173.313 | 1.543 |
St. dev. | 8640 | 11926 | 0.0170 |
RSD (%) | 0.720 | 0.644 | 1.0988 |

Method 2:

Instrumentation: Gas Chromatograph with FID, Split/splitless Inlet

**Column:** Zebron™ ZB-5HT, GC Cap. Column 30 m x 0.32 mm x 0.10 µm

**Injection Mode:** Split 51.575:1, 1 µL @ 330°C

**Carrier Gases:** Constant Flow Helium, 1.9 mL/min

**Col./Oven Temp:** 150 °C for 2 min to 420 °C @ 20 °C / min for 4.5 min

**Inlet temperature:** 330 °C

**Injection volume:** 1 µL, split 51.575:1

**GC Detection:** FID at 430 °C

Sample Preparation: Mineral Oil @ 10% in iso-Octane

References:


http://www.phenomenex.com/Application/Detail/18198?returnURL