Polysiloxane Analysis on Agilent PLgel 5 µm MIXED-D using GPC/SEC with RI and ELS Detection

Application Note

Materials Testing and Research, Polymers

Authors
Greg Saunders and Ben MacCreath
Agilent Technologies (UK) Ltd.,
Essex Road
Church Stretton
SY6 6AX
UK

Introduction
Polysiloxanes are important commercial polymers used in applications such as lubricating oils, defoaming agents and sealants. Analysis of these materials by gel permeation chromatography (GPC) is an important part of process and quality control procedures. Although polysiloxane is soluble in tetrahydrofuran (THF), it is isorefractive with this solvent. As a result, refractive index (RI) detection cannot be used with THF.

Conditions
<table>
<thead>
<tr>
<th>Sample</th>
<th>Polysiloxane, 0.2% 9w/v</th>
</tr>
</thead>
<tbody>
<tr>
<td>Column</td>
<td>3 × Agilent PLgel 5 µm MIXED-D, 300 mm × 7.5 mm (p/n PL1110-6504)</td>
</tr>
<tr>
<td>Eluent</td>
<td>Toluene THF</td>
</tr>
<tr>
<td>Flow rate</td>
<td>1.0 mL/min</td>
</tr>
<tr>
<td>Inj vol</td>
<td>100 µL</td>
</tr>
<tr>
<td>Detector</td>
<td>Agilent ELSD (neb = 40 °C, evap = 80 °C, gas = 1.0 SLM)</td>
</tr>
</tbody>
</table>
Analysis of Polysiloxane

There are two options for the analysis of polysiloxane - change the solvent and use RI detection or use THF with an Agilent evaporative light scattering detector (ELSD). ELS detection does not rely on the refractive index of the polymer and provides a positive response proportional to sample concentration in all solvents. Another advantage of ELS over RI detection is that it does not generate system peaks due to solvent imbalance. The solvent is evaporated in the detector, which gives the Agilent ELSD excellent baseline stability.

Figure 1 shows chromatograms of a polysiloxane obtained in toluene with RI detection and THF with ELS detection. The ELS chromatogram has no system peaks and a flat, stable baseline. The RI chromatogram shows large system peaks and a drifting baseline. The ELS also shows a very large response to the polymer compared to the RI; in this application, the ELS sensitivity is about 250 times higher than the RI detector (RI full scale output is 1 V whereas the ELS full scale output is 10 V).

Conclusions

In the case of polysiloxanes, the Agilent ELS detector provides more information about the sample. The improved detector response and baseline stability compared to RI allow a much greater accuracy of analysis of the molecular weight distribution of the polymer to be achieved.

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem.