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

Application Note

Materials Testing and Research, Polymers

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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

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

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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).
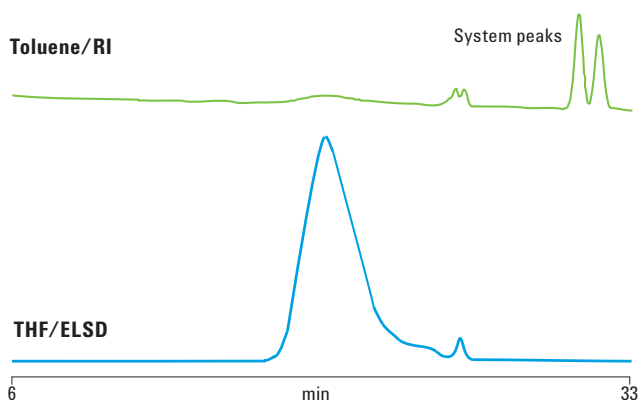

Figure 1   Analysis of a polysiloxane showing the improved data quality from evaporative light scattering detection compared to refractive index detection.

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.

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