

Resin Analysis with Agilent PLgel MIXED Columns and Gel Permeation Chromatography

Application Note

Materials Testing and Research, Polymers

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Introduction

The composition of oligomers in pre-polymers is of great commercial importance, as is the determination of residual monomer in the quality control of polymers. These low molecular weight samples are routinely characterized by gel permeation chromatography (GPC). Ideally, separation of discrete components is required in order to identify and quantify specific components of interest. In order to achieve this, small-particle-size packings are used to produce high resolution separations.



Resin Characterization

The Agilent PLgel 3 μ m MIXED-E column, with an exclusion limit of 30,000 molecular weight, has a guaranteed minimum efficiency of 80,000 plates/meter. Typical resin application chromatograms are shown in Figures 1 and 2.

Conditions for Figure 1

Sample	Isocyanate
Columns	2 \times Agilent PLgel 3 μ m MIXED-E, 7.5 \times 300 mm (p/n PL1110-6300)
Eluent	THF (stabilized)
Flow rate	1.0 mL/min
Detector	RI
System	Agilent PL-GPC 50

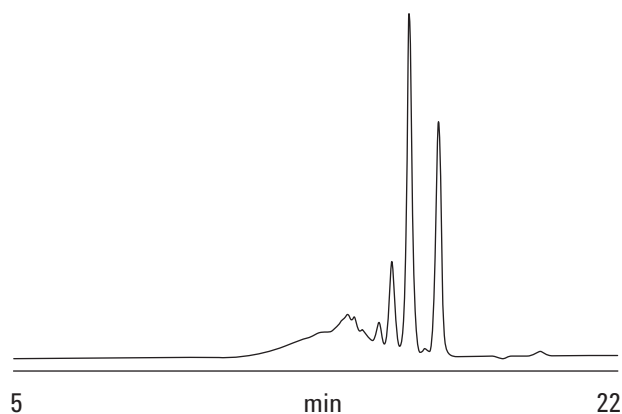


Figure 1. An isocyanate resin analyzed on an Agilent PLgel 3 μ m MIXED-E two-column set.

Conditions for Figure 2

Sample	Polyol
Columns	2 \times Agilent PLgel 3 μ m MIXED-E, 7.5 \times 300 mm
Eluent	THF (stabilized)
Flow rate	1.0 mL/min
Detector	UV, 240 nm
System	PL-GPC 50

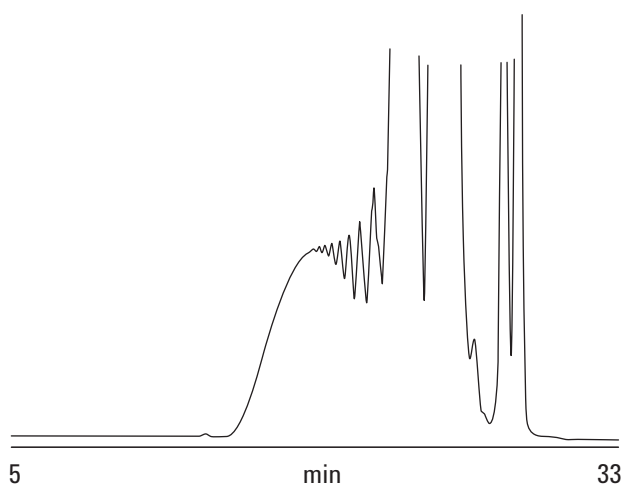


Figure 2. A polyol separated on Agilent PLgel 3 μ m MIXED-E columns.

For higher molecular weight resins, the determination of molecular weight distribution is a primary objective in GPC analysis, and columns with a broader resolving range are required. The Agilent PLgel 5 μ m MIXED-D column has been specifically designed for such applications where material above 400,000 molecular weight is unlikely to be present. Figure 3 illustrates a typical application chromatogram.

Conditions for Figure 3

Sample	Paint resin
Columns	2 × Agilent PLgel 5 μm MIXED-D, 7.5 × 300 mm (p/n PL1110-6504)
Eluent	THF (stabilized)
Flow rate	1.0 mL/min
Detector	RI
System	PL-GPC 50

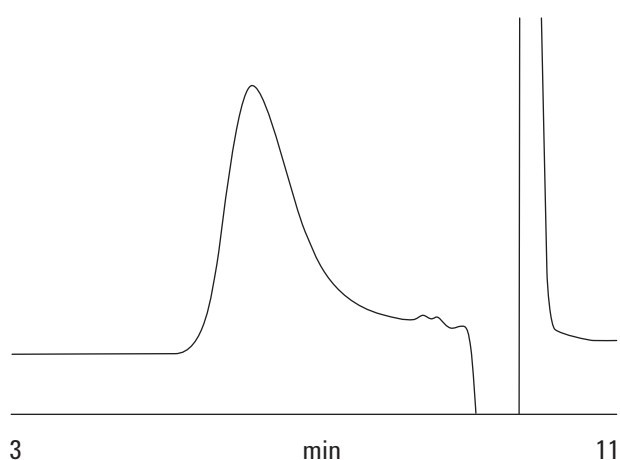


Figure 3. A medium molecular weight paint resin analyzed on an Agilent PLgel 5 μm MIXED-D two-column set.

The Agilent PLgel 10 μm MIXED-B column is designed to span a wide range of molecular weight, up to 10 million with a linear calibration curve. It is particularly useful for molecular weight distributions where slightly higher than average molecular weights are encountered. Alkyd resins typically fall into this category, as illustrated in Figure 4.

Conditions for Figure 4

Sample	Alkyd resin
Columns	2 × Agilent PLgel 10 μm MIXED-B, 7.5 × 300 mm (p/n PL1110-6100)
Eluent	THF (stabilized)
Flow rate	1.0 mL/min
Detector	RI
System	Agilent PL-GPC 50

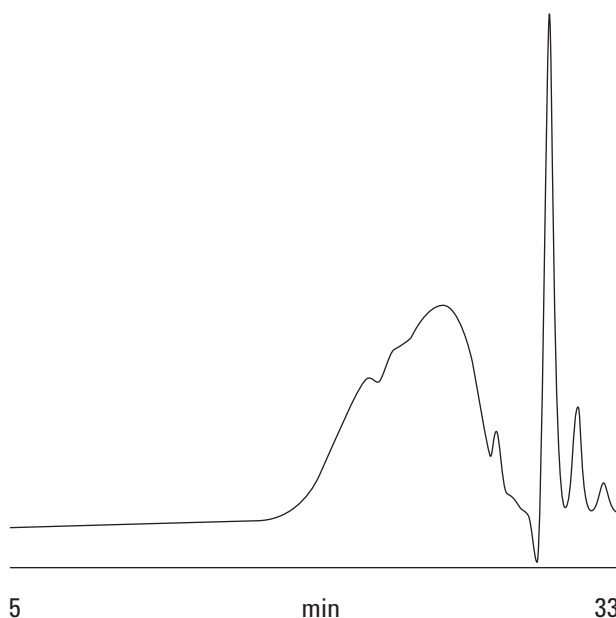


Figure 4. A higher molecular weight alkyd resin analyzed on an Agilent PLgel 10 μm MIXED-B two-column set.

Conclusions

Pre-polymer oligomer composition is important for the commercial use of polymers, as is an assessment of residual monomers for polymer quality control to ascertain their end-use suitability. Gel permeation chromatography is routinely used to characterize low molecular weight samples. To achieve high resolution separations, small-particle-size packings, such as those in Agilent PLgel columns, are used to separate, identify, and quantify discrete components of interest.

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