

Analysis of Polybutadienes by GPC with Triple Detection

Application Note

Materials Testing & Research

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Introduction

Polybutadienes are a type of synthetic rubber, commonly used to make tires along with other car parts such as belts, hoses and gaskets, because of their resilience to temperature and wear. They also offer a high level of electrical resistivity. Polybutadiene was one of the first types of synthetic elastomer to be invented and now synthetic rubber has largely replaced natural rubber in a wide variety of industrial applications.

Triple detection size exclusion chromatography (SEC) employs a concentration detector, a viscometer and a light scattering detector to assess the molecular weight distribution and molecular structure of polymers without having to rely on column calibrations. This can be important when analyzing complex materials for which no structurally similar standards are available.

Verified for Agilent
1260 Infinity
GPC/SEC System



Agilent Technologies

Conditions

Sample: Polybutadiene
Columns: 2 × Agilent PLgel 5 µm MIXED-C,
7.5 × 300 mm (p/n PL1110-6500)
Inj Vol: 100 µL
Eluent: THF
Flow Rate: 1 mL/min
Detector: Agilent PL-GPC 50, RI, Agilent
PL-BV 400, Agilent dual angle LSD

Results and Discussion

A sample of polybutadiene was analyzed on a PL-GPC 50 Integrated GPC/SEC System running at 30 °C. The system was fitted with a refractive index detector, a PL-BV 400 four capillary bridge viscometer and a dual angle light scattering detector (collecting scattered light at low and high angle).

Two PLgel 5 µm MIXED-C columns were used for this analysis. The polybutadiene sample was prepared accurately at a nominal concentration of 2 mg/mL in tetrahydrofuran and injected into the system without further treatment. For the purpose of light scattering calculations, an average dn/dc was used for the sample.

Figure 1 shows an overlay of the triple detector chromatograms for the sample.

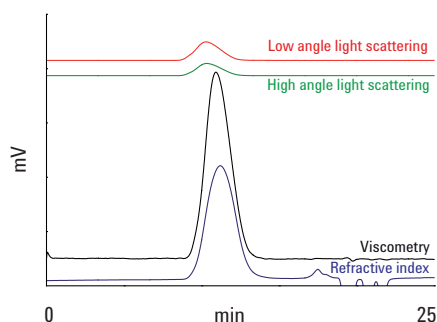


Figure 1. Triple detection of a polybutadiene

Figure 2 reveals the molecular weight distribution calculated for the sample of polybutadiene.

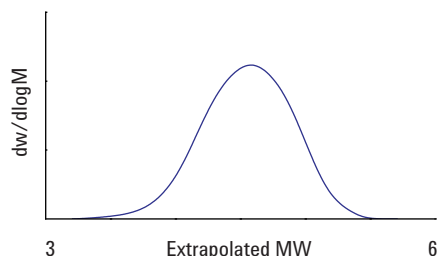


Figure 2. Molecular weight distribution of a polybutadiene

Mark-Houwink (log intrinsic viscosity versus log M) plots were generated from the viscometry and light scattering data (Figure 3). The curvature in the Mark-Houwink Plot may be a result of structural changes in the polymer as a function of molecular weight.

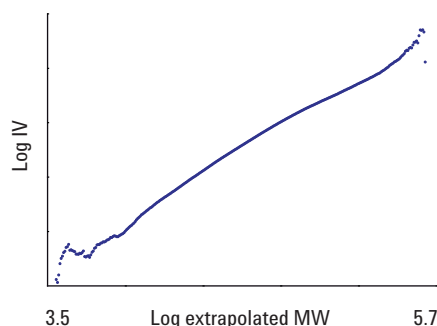


Figure 3. Mark-Houwink plot of a polybutadiene

Conclusion

The PL-GPC 50 Integrated GPC/SEC System is ideal for the analysis of structurally complex but commercially important materials by multi detector GPC. By using triple detection the molecular weight distribution and molecular structure of polymers can be ascertained without having to rely on column calibrations which may not be

possible because of a lack of suitable standards.

The enhanced PL-GPC 50 is a high resolution, cost effective integrated GPC system designed for operation from ambient to 50 °C. The standard system comprises a precision solvent delivery pump, a sample injection system, a high performance differential refractive index detector and a column oven, with fully integrated software control. For maximum flexibility and applicability, a choice of system enhancements is also available.

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