Analysis of Bromostyrene by GPC Triple Detection using the Agilent 390-MDS Multi Detector Suite

Application Note

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Introduction

The structure of many common types of polymer may be modified by either post-polymerization reaction or by the use of modified monomers, producing materials with new and novel properties. Understanding and assessing the effect of these modified structures on the behavior of polymers in various applications is crucial to developing novel uses for such materials. Polystyrene is one of the most commonly seen polymers, used primarily for the manufacture of packing materials. Modified forms of polystyrene may be produced by the use of novel styrene monomers.

Gel permeation chromatography (GPC) is a well-known technique for assessing the molecular weight distribution of polymers, a property that influences many physical characteristics. Importantly, increasing the sophistication of the GPC experiment by the use of additional detectors allows the structure and solution properties of polymers to be assessed, of interest when studying the affect of structural modifications on a polymer. Triple detection employing a light scattering detector and a viscometer in combination with a refractive index detector may be used to determine accurate molecular weights for polymers for which narrow standards are not available, and to assess their solvation properties. This application note describes the analysis of a sample of polystyrene and a modified polybromostyrene material by triple detection.

Verified for Agilent
1260 Infinity
GPC/SEC System

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Methods and Materials

Conditions

Samples:	Polystyrene and polybromostyrene
Columns:	2 x Agilent PLgel 5 µm MIXED-C, 300 x 7.5 mm (p/n PL1110-6500)
Injection Volume:	100 µL
Eluent:	THF (stabilized)
Flow Rate:	Agilent 290-LC PIM at 1.0 mL/min
Detector Train:	390-MDS incorporating Agilent 390 Dual angle light scattering, Viscometer and DRI options
Detector Temp:	All detectors set at 40 °C

Polystyrene and polybromostyrene are both soluble in tetrahydrofuran, which is an excellent solvent for GPC and was therefore chosen for this analysis. The 390-MDS was chosen as part of the system in its powerful triple detection set up, as this would allow the most detailed analysis of the polymer samples.

Results and Discussion

Figure 1 shows an example overlaid multi-detector chromatogram for the sample of polybromostyrene. Each of the polymers gave strong signals in all the detectors that were fairly broad, indicating the high polydispersity of the materials.

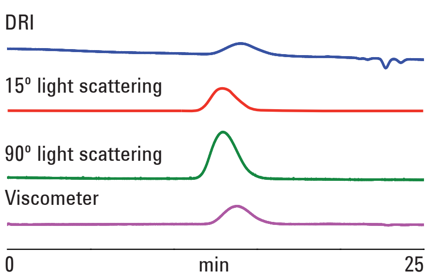

Figure 1. Overlaid multi-detector chromatogram for an example of polybromostyrene

Figure 2 shows an overlay of the molecular weight distributions of the two samples under investigation. As can be seen, the polybromostyrene was considerably higher in molecular weight than the polystyrene sample.

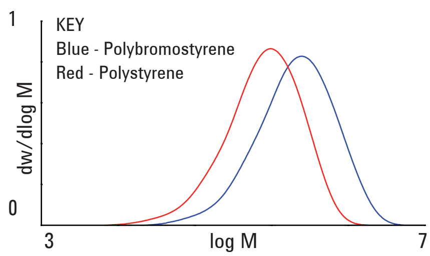

Figure 2. Overlaid triple detector molecular weight distributions of the two samples

Figure 3 shows the overlaid Mark-Houwink plot of log intrinsic viscosity as a function of molecular weight for the two samples. Compared to the polystyrene, the polybromostyrene shows a marked shift of the Mark-Houwink plot to lower intrinsic viscosity values at any given molecular weight. This indicates that polybromostyrene is smaller in solution than polystyrene across the molecular weight range, a result of changes in the level of interaction between the polymer coils and the solvent. The plot parallels that of polystyrene, indicating that the structure is identical across the range of molecular weight, as expected when a modified monomer unit is used to create the new polymer.

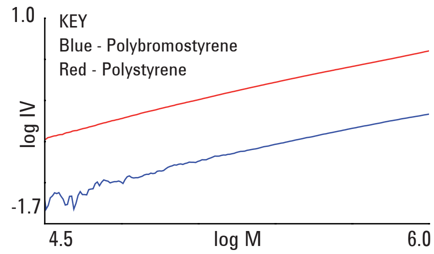

Figure 3. Overlaid Mark-Houwink plots for two samples

Conclusion

The data in this application note illustrate how multi-detector GPC employing the 390-MDS can be used to clearly see structural differences between polystyrene and a related polymer with a modified structure.

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This information is subject to change without notice.

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