Analysis of Polydimethyl Siloxane by GPC Viscometry with the Agilent 390-MDS Multi Detector Suite

Application Note

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Introduction

Polydimethyl siloxane (PDMS) is a non-toxic, non-flammable, silicon-based polymeric material noted for its unusual rheological behavior. Composed of polymer chains with the formula $(CH_3)_3SiO[SiO(CH_3)_2]nSi(CH_3)_3$, PDMS is a viscoelastic material which, with long flow times or at low temperature, behaves like a liquid. PDMS is produced in a range of grades from liquids through to rubbery semi-solids, depending on the molecular weight of the constituent chains. PDMS is widely used in silicone caulks, lubricants, damping fluids and heat transfer fluids, as well as breast and knuckle implants. It is also a food additive with the E number E900, where it is employed as an anti-foaming and anti-caking agent.

Owing to the importance of the viscometric properties of the material in many final applications, a viscometer was included in the detector array, as well as a standard refractive index detector. This combination of detectors also permitted analysis by the universal calibration method, giving accurate molecular weights that were not reliant on the chemistry of the standards used for calibration (in this case, polystyrene). Although PDMS is soluble in tetrahydrofuran (THF), it is also isorefractive with this solvent and so THF is not suitable for the analysis. Toluene is preferable. The 390-MDS was chosen for this analysis as it can be used to perform multi-detector GPC in any solvent.

Verified for Agilent
1260 Infinity
GPC/SEC System

Methods and Materials

Conditions

Columns:	2 x Agilent PolyPore, 300 x 7.5 mm (p/n PL1113-6500)
Eluent:	Toluene
Flow Rate:	1.0 mL/min
Injection Volume:	100 µL
Detector Train:	390-MDS incorporating Viscometer and DRI
Detector Temp:	All detectors set at 60 °C

Results and Discussion

Figure 1 shows example overlaid refractive index and viscometer chromatograms for a sample of PDMS and Figure 2 is the overlaid molecular weight distributions for two different PDMS grades. Figure 3 shows the Mark-Houwink plots calculated for the two samples.

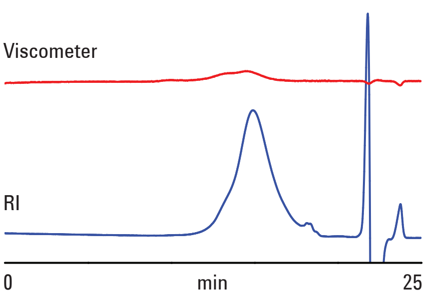

Figure 1. Overlaid multi-detector chromatograms for polydimethyl siloxane

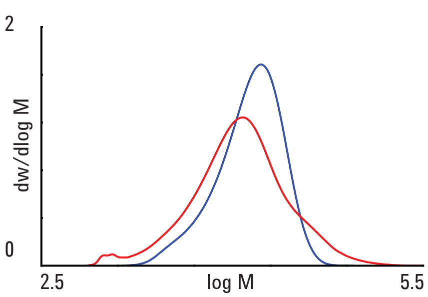

Figure 2. Overlaid multi detector molecular weight distributions from two detectors for two different samples of polydimethyl siloxane

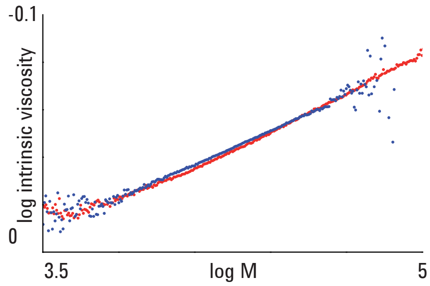

Figure 3. Overlaid Mark-Houwink plots for two samples of polydimethyl siloxane

Although quite different in molecular weight, the Mark-Houwink plots show that the two materials are structurally very similar, indicating that their viscoelastic behavior as a function of molecular weight would be comparable.

Conclusion

Multi-detector GPC revealed that samples of polydimethyl siloxane were structurally similar despite having very different molecular weights. PolyPore columns and 390-MDS multi-detection provide a powerful tool for elucidating the potential behavior of viscoelastic polymers, such as polydimethyl siloxane.

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