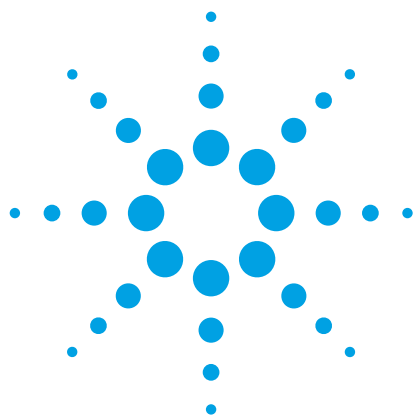




Polyvinylidene Fluoride Analysis on Agilent PLgel 10 μ m MIXED-B and GPC/SEC

Application Note

Materials Testing and Research, Polymers

Authors

Greg Saunders and Ben MacCreath
Agilent Technologies (UK) Ltd
Essex Rd
Church Stretton
SY6 6AX
UK

Introduction

Fluoropolymers are important materials with a wide range of uses from non-stick coatings to inert packaging materials. However, fluoropolymers can be difficult to analyze by gel permeation chromatography due to their low solubility in common solvents. This concern can be overcome by using dimethyl sulfoxide as solvent, Agilent PLgel MIXED-B columns, and operating the GPC system at elevated temperature with appropriate high temperature sample preparation.

Analysis of Polyvinylidene Fluoride

Three samples of polyvinylidene fluoride (PVDF) were analyzed in dimethyl sulfoxide at 95 °C using the Agilent PL-GPC 220 instrument with two Agilent PLgel 10 μ m MIXED-B, 300 $\times$ 7.5 mm columns. For this application, elevated temperatures are required to dissolve the samples and to reduce the viscosity of the solvent that would otherwise result in high back pressures at flow rates of 1.0 mL/min, and poor chromatography.

The samples were prepared at a nominal 3 mg/mL. An appropriate amount of each sample was weighed into a vial, the eluent added and the solution heated at 95 °C for 4 h in the Agilent PL-SP 260 sample preparation unit.

The solutions were then filtered and dispensed into the PL-GPC 220 autosampler vials. The samples were analyzed on the Agilent PL-GPC 220 with the autosampler carousel temperature at 95 °C and 40 °C in the hot and warm zones, respectively.



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The system was calibrated using Agilent narrow polydispersity polymethylmethacrylate (PMMA) standards. When using polar organic solvents, polystyrene standards may be soluble but they exhibit hydrophobic interactions with the column, resulting in non-meaningful molecular weight assignments.

Conditions

Samples	Polyvinylidene fluoride
Columns	2 × Agilent PLgel 10 µm MIXED-B, 300 × 7.5 mm (p/n PL1110-6100)
Eluent	Dimethyl sulfoxide
Flow rate	1.0 mL/min
Injection volume	100 µL
Temp	95 °C
System	PL-GPC 220

Figure 1 shows raw data chromatograms of the three samples. The positive peaks that elute after the samples are due to imbalances between the solvent in which the sample is prepared and the eluent.

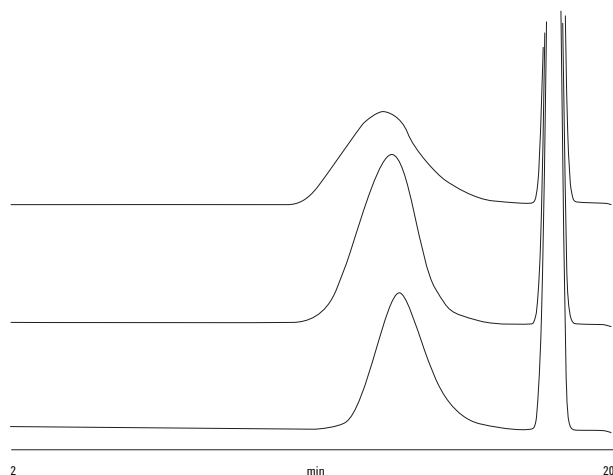


Figure 1. Three samples of polyvinylidene fluoride separated on an Agilent PLgel 10 µm MIXED-B two-column set.

Figure 2 shows overlaid molecular weight distributions of the three samples based on polymethylmethacrylate standards.

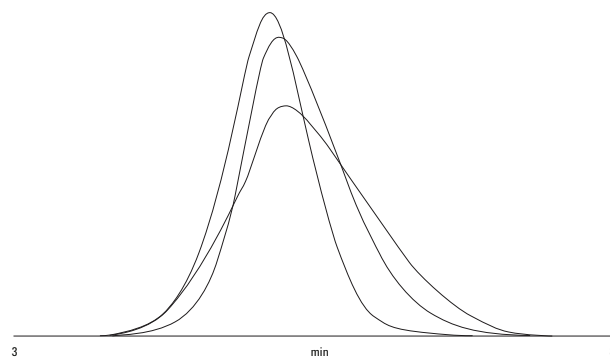


Figure 2. Overlaid molecular weight distributions of three samples of polyvinylidene fluoride based on polymethylmethacrylate standards.

Conclusions

Agilent PLgel columns can be used in a wide range of solvents, including polar organics, which facilitate the analysis of very polar samples such as polyvinylidene fluoride.

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