Application Note

Materials Testing and Research, Polymers

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Polyamide Analysis on Agilent PL HFIPgel with Gel Permeation Chromatography

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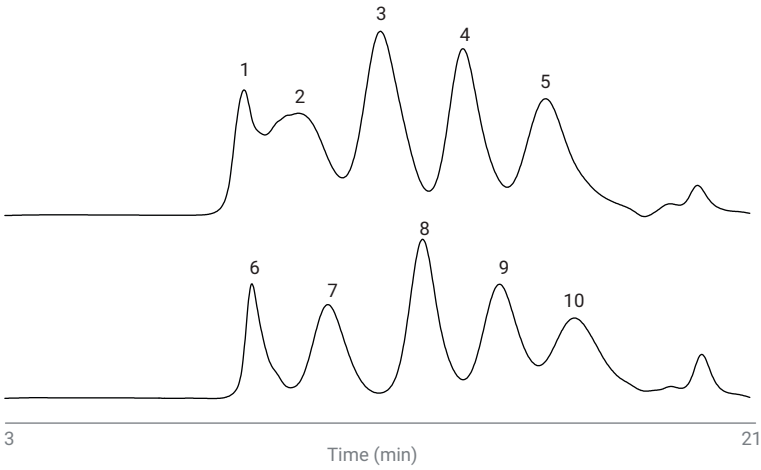
Introduction

Polyamides and polyesters are extremely important commercial polymers used in the manufacture of containers and clothing. Determining the molecular weight distributions of these materials is an important aspect of research and development, and quality control procedures.

Analysis of polyamide

Hexafluoroisopropanol (HFIP) has become a favored solvent for analysis of polyamides and polyesters by gel permeation chromatography (GPC). Unlike alternative solvents such as *m*-cresol or *o*-chlorophenol, HFIP is used at near ambient temperatures, and has a low refractive index compared to polyester and polyamides, making it appropriate for differential refractive index and light scattering detection. Unfortunately, due to the high solvent polarity of HFIP, conventional styrene/divinyl benzene-based columns generally give poor chromatography with dislocations, excessive curvature of calibrations, and poor low molecular weight resolution.

This Application Note illustrates the use of Agilent PL HFIPgel columns for the analysis of a polyamide sample. The GPC system was set up using two PL HFIPgel columns and a refractive index detector running in HFIP, with 0.02 M NaTFAc added to minimize aggregation of the polymer in solution. The system was calibrated with a series of Agilent PM-1 polymethylmethacrylate (PMMA) narrow standards (Figure 1). Figure 2 shows the resulting calibration curves. Figure 3 is a chromatogram of the polyamide prepared at 0.2% (w/v), and Figure 4 shows a molecular weight distribution of the polyamide based on the PMMA calibration.



No.	Mp (g/mol)
1.	1,900,000
2.	281,700
3.	59,800
4.	13,300
5.	1,936
6.	790,000
7.	144,000
8.	28,900
9.	5,720
10.	1,020

Figure 1. Chromatograms of two injections of PMMA standards on PL HFIP columns.

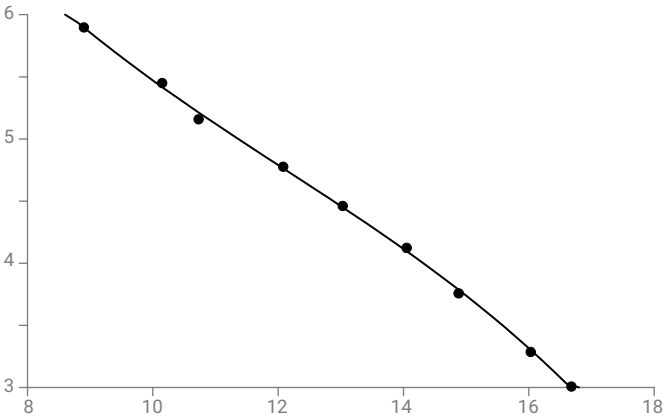


Figure 2. PMMA calibration curve (the highest molecular standard of Mp 1,900,000 g/mol is excluded on the PL HFIPgel columns).

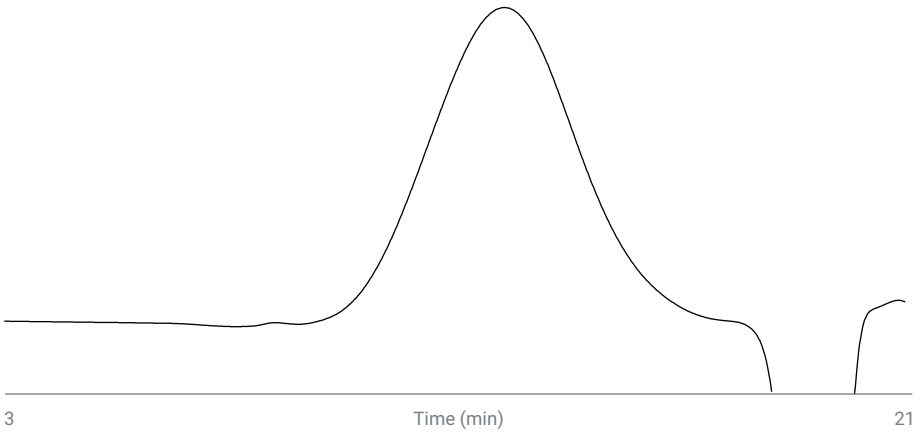


Figure 3. Chromatogram of a polyamide sample prepared at 0.2 % (w/v) on a PL HFIPgel two-column set.

Conditions

Parameter	Value
Columns	2 × PL HFIPgel, 300 × 7.5 mm (p/n PL1114-6900HFIP)
Eluent	HFIP + 0.2 M NaTFAc
Flow Rate	1.0 mL/min
Injection Volume	200 µL
Temperature	40 °C
Detector	RI

Conclusion

A sample of polyamide was successfully analyzed on a two-column set of Agilent PL HFIPgel columns. These columns use a novel dispersion polymerization process, giving near-uniform bead size and characteristics. This technology avoids the excessive calibration curvature, dislocations, and poor low molecular weight resolution associated with conventional columns that use styrene/divinyl benzene when using HFIP as solvent.

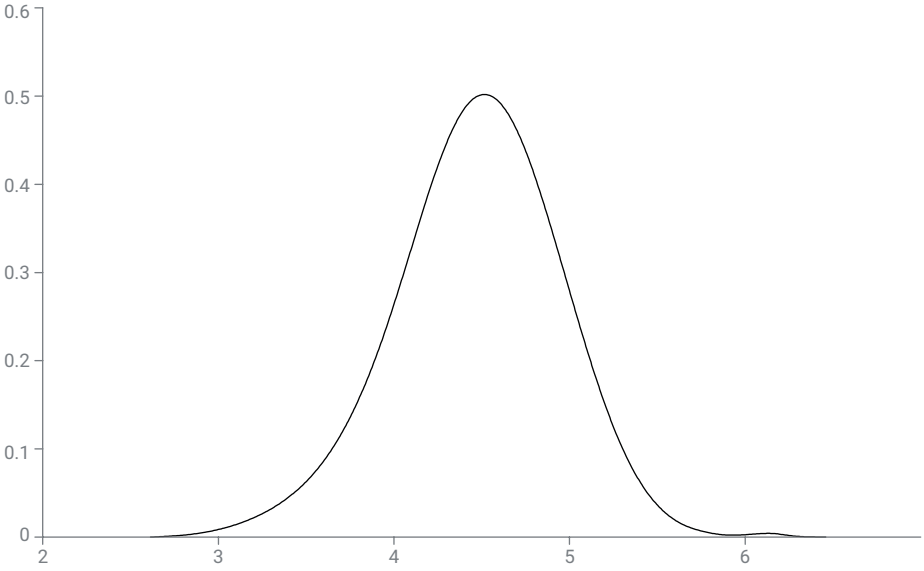


Figure 4. Molecular weight distribution of the polyamide based on the PMMA calibration.

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