

Polyamide Analysis on Agilent PL HFIPgel with Gel Permeation Chromatography

Application Note

Materials Testing and Research, Polymers

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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). The resulting calibration curves are shown in Figure 2. 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.

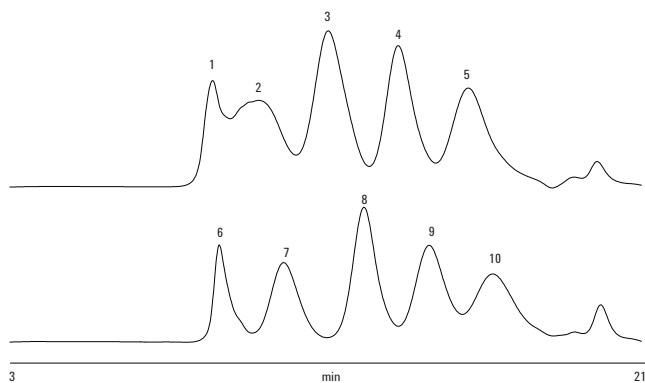


Figure 1. Chromatograms of two injections of PMMA standards on Agilent PL HFIP columns.

Key	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

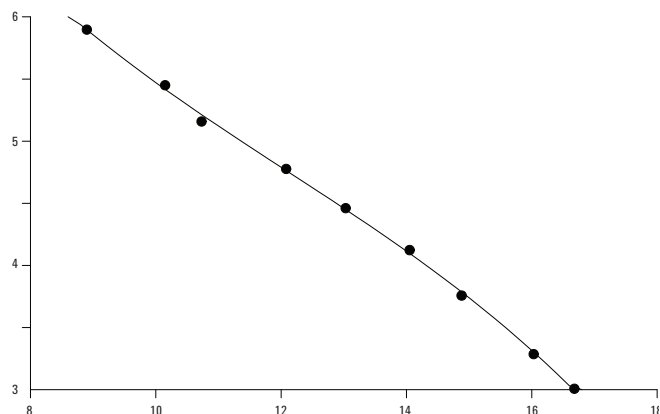


Figure 2. PMMA calibration curve (the highest molecular standard of Mp 1,900,000 g/mol is excluded on the PL HFIPgel columns).

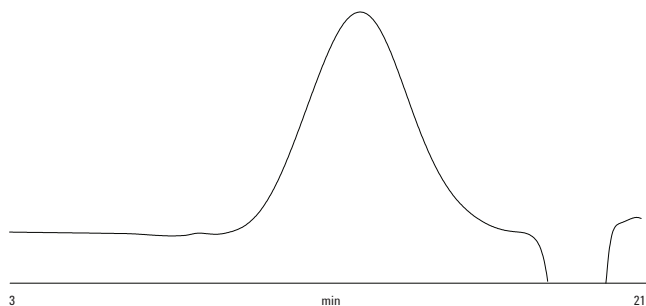


Figure 3. Chromatogram of a polyamide sample prepared at 0.2% (w/v) on an Agilent PL HFIPgel two-column set.

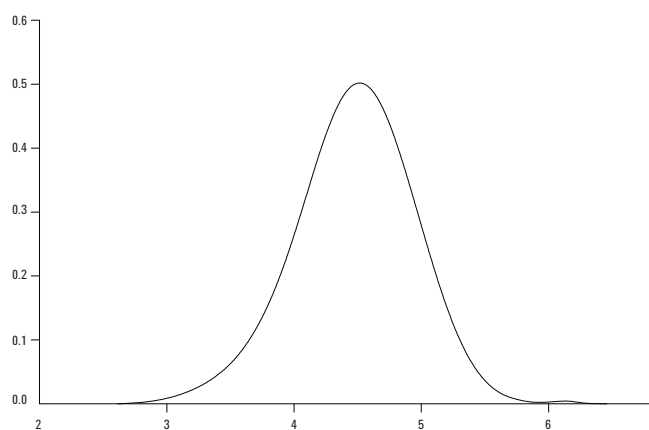


Figure 4. Molecular weight distribution of the polyamide based on the PMMA calibration.

Conditions

Columns	2 × PL HFIPgel, 300 × 7.5 mm (p/n PL1114-6900HFIP)
Eluent	HFIP + 0.2 M NaTFAc
Flow rate	1.0 mL/min
Inj vol	200 µL
Temp	40 °C
Detector	RI
System	Agilent PL-GPC 50

Conclusions

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.

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