Repeatability in High Temperature Polypropylene Analysis Using Agilent PLgel MIXED-B

Technical Overview

Introduction

A gel permeation chromatography system comprising Agilent PLgel MIXED-B columns and the Agilent PL-GPC 220 integrated, high temperature instrument is ideally suited to the analysis of polypropylene. The ability of the system is demonstrated in a repeatability study using six polypropylene injections.

A commercial sample of PP was prepared at 1.5 mg/mL using the PL-SP 260 sample preparation system with a dissolution temperature of 160 °C and a dissolution time of two hours. Six aliquots of the master batch solution were dispensed into the PL-GPC 220 autosampler vials and placed in the carousel where the hot zone temperature was 160 °C and the warm zone 80 °C.

Figure 1 shows an overlay of the raw data chromatograms obtained for the six consecutive injections of the sample.

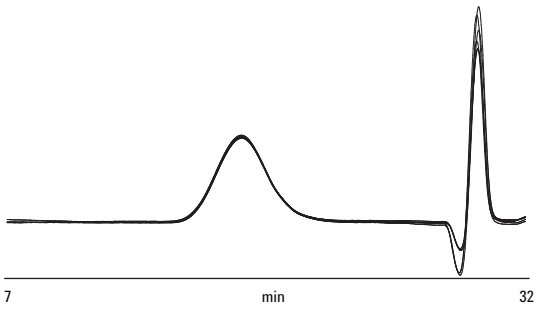

Figure 1. An overlay of the raw data chromatograms obtained for six consecutive polypropylene injections.

The data were analyzed against a polystyrene standards calibration using the following Mark-Houwink parameters to obtain the polypropylene equivalent molecular weight averages shown in Table 1.

Polystyrene in TCB[1] $K = 12.1 \times 10^{-5}$ $\alpha = 0.707$

Polypropylene in TCB[2] $K = 19.0 \times 10^{-5}$ $\alpha = 0.725$

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Table 1. Calculated Molecular Weights for Six Injections of Polypropylene and Calculated % Variation

Injection number	Mp	Mn	Mw
1	127,132	65,086	185,795
2	131,893	65,089	185,236
3	128,673	66,802	186,202
4	132,062	67,417	188,048
5	131,625	69,320	188,679
6	130,227	69,677	186,188
Mean	130,202	67,232	186,691
Standard deviation	1,693	1,815	1,239
% Variation	0.13	2.70	0.66

Conditions

Samples	Polypropylene
Columns	3 × Agilent PLgel 10 µm MIXED-B, 300 × 7.5 mm (p/n PL1110-6100)
Eluent	TCB + 0.0125% BHT
Flow rate	1.0 mL/min
Injection volume	200 µL
Temperature	160 °C
System	Agilent PL-GPC 220

Figure 2 shows an overlay of the molecular weight distribution calculated for six consecutive injections of the polypropylene sample

References

1. H. Col and D. K. Giddings, J. Polym. Sci., (A2) 8 (1970) 89.
2. T. G. Scholte *et al.*, J. Appl. Polym. Sci., 29 (1984) 3763.

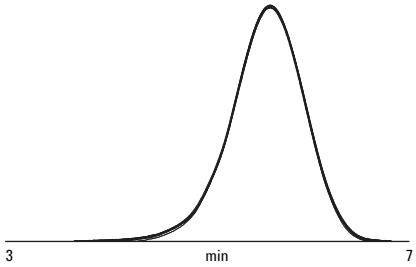

Figure 2. Overlay of the molecular weight distribution calculated for six consecutive injections of polypropylene that illustrates the excellent repeatability obtained with the Agilent PL-GPC 220 using Agilent PLgel 10 µm MIXED-B columns.

GPC/SEC Columns and Calibrants from Agilent

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