Repeatability in High Temperature Polyethylene 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 polyethylene. The ability of the system is demonstrated in a repeatability study using eight polyethylene injections.

A commercial sample of high density polyethylene (HDPE) was prepared at 2 mg/mL using the Agilent PL-SP 260 sample preparation system with a dissolution temperature of 160 °C and a dissolution time of two hours. Eight aliquots of the master batch solution were dispensed into the Agilent PL-GPC 220 autosampler vials and placed in the autosampler carousel of the PL-GPC 220 where the hot zone temperature was 160 °C and the warm zone was 80 °C.

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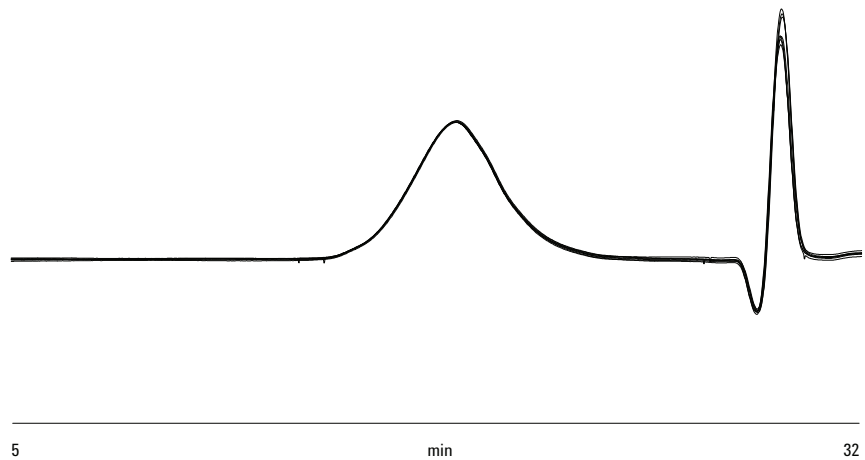

5   min   32

Figure 1.   An overlay of the raw data chromatograms obtained for eight consecutive polyethylene injections.

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The data were analyzed against a polystyrene standards calibration using the following Mark-Houwink parameters to obtain the polyethylene equivalent molecular weight averages. The molecular weight averages, peaks areas and a statistical analysis of the results for the eight injections are shown in Table 1.

Polystyrene in TCB[1] K = 12.1 x 10^{-5} α = 0.707
Polyethylene in TCB[2] K = 40.6 x 10^{-5} α = 0.725

Table 1.   Calculated Molecular Weights for Eight Injections of Polyethylene and Calculated % Variation

Injection number	Mp	Mn	Mw
1	17,289	76,818	333,851
2	16,988	77,434	335,496
3	17,428	77,514	332,616
4	17,521	77,052	335,635
5	17,348	76,520	334,212
6	17,487	77,728	333,511
7	16,898	77,578	335,642
8	17,457	77,288	334,923
Mean	17,302	77,241	334,485
Std dev	220	387	1,048
% Variation	1.3	0.5	0.3

Conditions

Sample	High density polyethylene
Columns	3 × Agilent PLgel 10 µm MIXED-B, 7.5 × 300 mm (p/n PL1110-6100)
Eluent	TCB + 0.0125% BHT
Flow rate	1.0 mL/min
Inj vol	200 µL
Temp	160 °C
System	Agilent PL-GPC 220

Figure 2 is an overlay of the molecular weight distribution calculated for the eight consecutive injections of the HDPE sample, and illustrates the excellent repeatability obtained with the PL-GPC 220 using Agilent PLgel 10 µm MIXED-B columns.
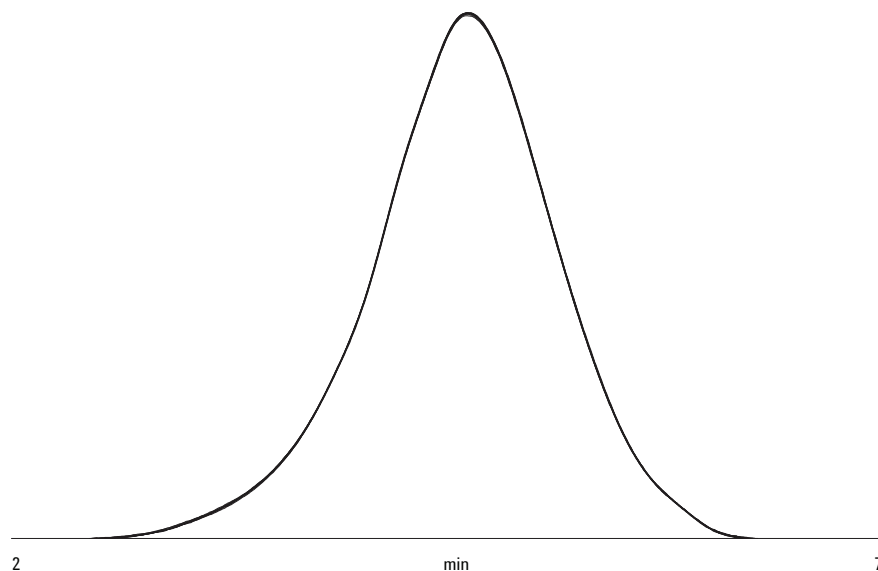
| 2 | min | 7 |

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

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.

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