

Characterization of Polyethylene Terephthalate with GPC/SEC

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Abstract

This application brief describes the characterization of polyethylene terephthalate using Agilent PFG columns in fluorinated solvents (e.g., hexafluoroisopropanol).

Introduction

Polyethylene terephthalate (PET) is prepared by polycondensation of dimethyl terephthalate with ethylene glycol. PET is used to produce chemically stable products with high mechanical rigidity. Bottles, foils, and other food packaging forms are often made from PET.¹

Recently, the recycling of PET has been gaining interest, and requires a robust and reliable analytical method for characterization and quality control.

Experimental

See Table 1.

Results and discussion

Polyethylene terephthalate samples were analyzed in HFIP with 0.05 M potassium trifluoroacetate using a combination of two PFG 7 μm linear M columns with one guard column. PFG columns are based on modified silica particles. Compared to polymer-based particles, these columns are more robust in fluorinated solvents like HFIP and show an excellent pressure stability.

An overlay of three PET samples is shown in Figure 1.

Calibration with polymethyl methacrylate (PMMA) reference material can be used to analyze the molecular weight distribution (MWD) and average molecular weight values of PET species. The received values are relative molar masses based on PMMA molar mass equivalents. In Figure 2, an overlay of the MWDs is shown, and Table 2 summarizes the average molecular weight values.

Table 2. Summary of the average molecular weight values obtained based on PMMA calibration.

Sample	Mn [Da]	Mw [Da]	\bar{D} [Mw/Mn]
PET 1	12,900	23,200	1.80
PET 2	28,400	49,100	1.73
PET 3	61,700	127,000	2.05

Table 1. Instrument and sample conditions.

	Conditions
Pump	Isocratic pump Flow rate: 1 mL/min Mobile phase: hexafluoroisopropanol, 0.05 M potassium trifluoroacetate
Injection System	Autosampler Injection volume: 20 μL
Columns	PFG 7 μm precolumn, 8 \times 50 mm (p/n PFA080507) PFG 7 μm linear M, 8 \times 300 mm (p/n PFA083007LIM) PFG 7 μm linear M, 8 \times 300 mm (p/n PFA083007LIM)
Temperature	23 $^{\circ}\text{C}$
Sample Concentration	2 to 3 mg/mL
Calibration	Agilent ReadyCal-Kit Polymethylmethacrylate (p/n PSS-MMKTR1)
Detectors	Refractive index (RI) detector
Software	Agilent WinGPC

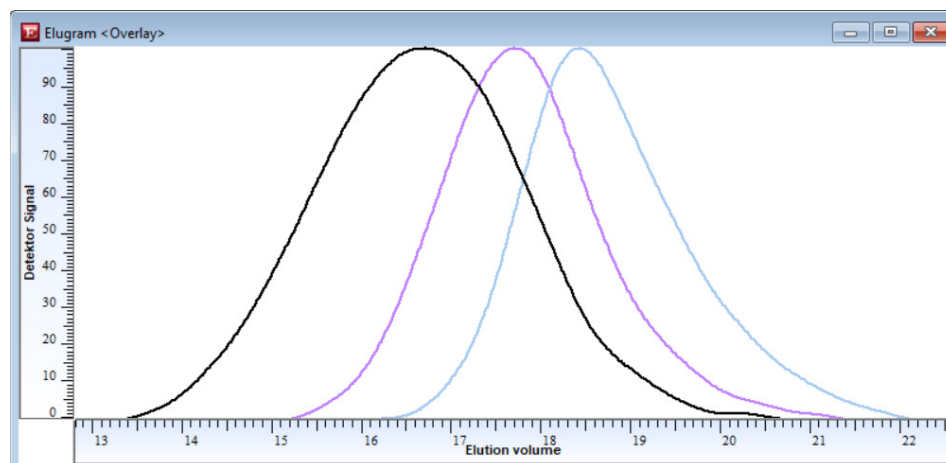


Figure 1. Overlay of three different PET samples (RI traces, normalized detector response).

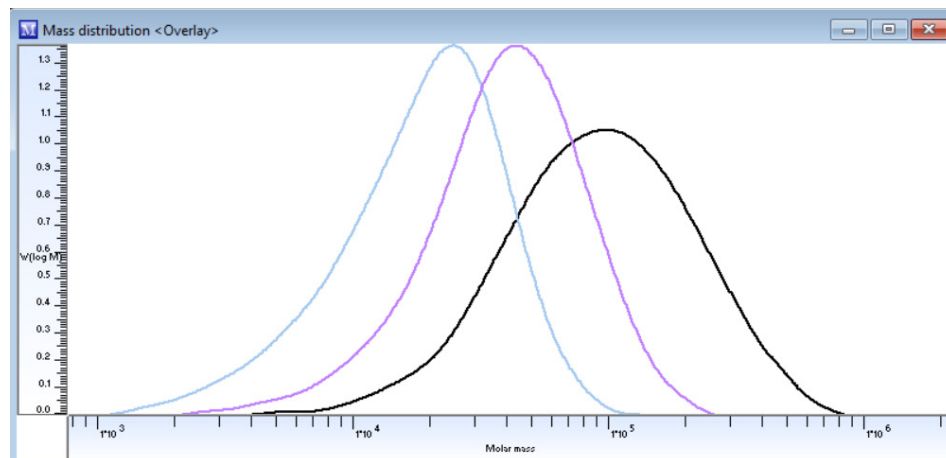


Figure 2. Overlay of the molecular weight distribution (based on calibration with PMMA reference materials, Agilent ReadyCal-Kit PMMA, p/n PSS-MMKTR1).

Conclusion

The robust characterization of PET via GPC/SEC is feasible with Agilent PFG columns as stationary phase and fluorinated mobile phases like HFIP with potassium trifluoroacetate. PMMA reference materials can be used for calibration to obtain apparent molar masses.

References

1. Polymer Data Handbook; Oxford University Press, Inc., **1999**.

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