

GPC/SEC Analysis of Oligomers and Low Molar Mass Polymers in THF

High resolution with Agilent SDV 3 μm columns

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Abstract

This application brief illustrates a comparison of oligomeric resolution using one Agilent SDV 1,000 Å column with particles sizes of 3, 5, and 10 μm for GPC/SEC measurements. In addition, polymethylmethacrylate (PMMA) oligomers are analyzed with a set of three SDV 3 μm columns, demonstrating their separation power and achievable resolution.

Introduction

The use of small particles in GPC/SEC columns leads to an increase of resolution, due to reduction of the interstitial volume resulting in less band broadening. Smaller particle sizes lead to higher backpressure, which needs to be considered for the analysis of ultrahigh molar mass species, due to the potential risk of shear degradation.¹

However, for GPC/SEC measurements of oligomers and low molar mass samples, when high resolution with separation of defined oligomers is required, columns with small particle sizes of 3 μm or even less need to be chosen.

Results and discussion

SDV 1,000 \AA columns with particle sizes of 3, 5, and 10 μm were used as stationary phase for GPC/SEC of a polystyrene (PS) oligomer with a molecular weight of 564 Da. An overlay of the obtained elugrams is shown in Figure 1.

As expected, the resolution of the PS oligomer increases with decreasing particle size of the SDV column. Comparing the elugrams with a 3 and 5 μm column, the increase of resolution is more distinct, and a higher number of oligomers is separated.

Experimental

Table 1. Instrument and sample conditions.

	Conditions
Pump	Isocratic pump Flow rate: 1.0 or 0.5 mL/min Mobile phase: tetrahydrofuran
Injection System	Autosampler Injection volume: 20 μL
Columns	SDV 3 μm 1,000 \AA , 8 \times 300 mm (p/n SDA0830031e3) SDV 5 μm 1,000 \AA , 8 \times 300 mm (p/n SDA0830051e3) SDV 10 μm 1,000 \AA , 8 \times 300 mm (p/n SDA0830101e3) SDV ultralow MW combination: SDV 3 μm precolumn, 8 \times 50 mm (p/n SDA080503) SDV 3 μm 100 \AA , 8 \times 300 mm (p/n SDA0830031e2) SDV 3 μm 100 \AA , 8 \times 300 mm (p/n SDA0830031e2) SDV 3 μm 100 \AA , 8 \times 300 mm (p/n SDA0830031e2)
Temperature	23 $^{\circ}\text{C}$
Sample Concentration	1.5 mg/mL
Detectors	Refractive index (RI) detector
Software	Agilent WinGPC

The oligomeric resolution can be further improved by extending the separation distance (using a higher number of SDV 3 μm columns) and reducing the flow rate to 0.5 mL/min. As an example, the separation of four low molar mass PMMA samples using SDV ultralow MW combination, composed of three SDV

3 μm 100 \AA columns together with a guard column, is shown in Figure 2. The monodisperse oligomer P2 with two monomer units and a molecular weight of 202 Da is included as a reference. For the PMMA samples, oligomeric resolution up to P16 with 16 monomer units was achieved.

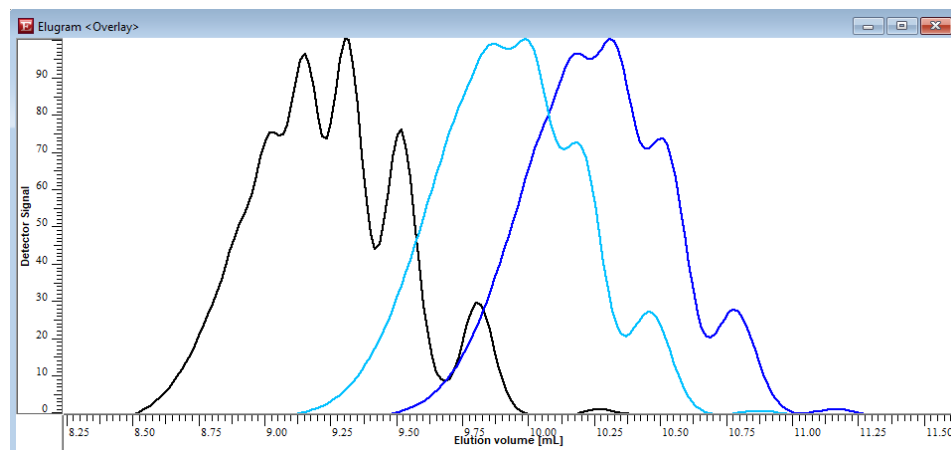


Figure 1. Overlay of PS Mw 564 Da (RI-traces, normalized detector response): SDV 3 μm 1,000 \AA (black line), SDV 5 μm 1,000 \AA (dark blue line), SDV 10 μm 1,000 \AA (light blue line).

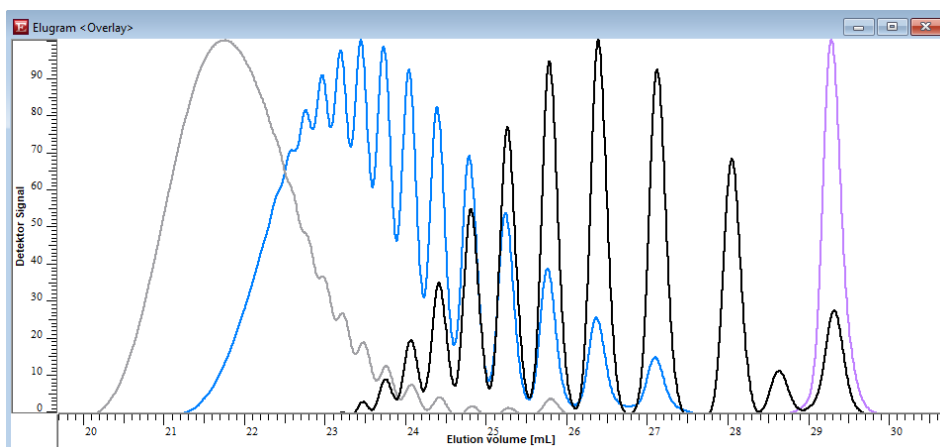


Figure 2. Overlay of four PMMA standards (RI-traces, normalized detector response): Mw 202 Da (purple line), Mw 573 Da (black line), Mw 1,220 Da (blue line), Mw 2,180 Da (grey line).

Conclusion

The use of Agilent SDV 3 μm columns significantly increases resolution, especially in GPC/SEC separation of oligomers and low molar mass polymers using THF as mobile phase. High resolution can be achieved using a set of three SDV 3 μm and reduced flow rate.

Reference

1. Striegel, A. M. *et al.* Modern Size-Exclusion Liquid Chromatography; Second Edition, Wiley & Sons Inc., **2009**.