Application Brief Materials Testing & Research



GPC/SEC with SDV Columns and 2-Methyltetrahydrofuran

Using a mobile phase from a renewable source

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Abstract

2-Methyltetrahydrofuran (2-methylTHF) is used as an eluent in GPC/SEC measurements with SDV columns. Reliable chromatography is displayed with polystyrene and polymethyl methacrylate standards as examples.

Introduction

Typical eluents for GPC/SEC are nonpolar organic solvents such as tetrahydrofuran (THF) and toluene, which are based on petrochemicals. Recently, various solvents from renewable sources have become available. 2-MethyITHF is one example, which can be derived from biomass and is a potential sustainable replacement for THF.¹

For an alternative solvent such as 2-methyITHF to be used as the mobile phase for GPC/SEC analysis, the following requirements need to be fulfilled: compatibility with the stationary phase is required, as well as good solubility of analytes. In addition, interaction-free chromatography and detection of the analytes with typical detectors such as refractive index (RI) detectors or UV-Vis detectors is essential.

Experimental

 Table 1. Instrument and sample conditions.

	Conditions
Pump	Isocratic pump Flow rate: 1 mL/min Mobile phase: 2-methyltetrahydrofuran
Injection System	Autosampler Injection volume: 20 μL
Columns	SDV high MW combination: SDV 5 μm precolumn, 8 × 50 mm (p/n SDA080505) SDV 5 μm 1,000 Å, 8 × 300 mm (p/n SDA0830051e3) SDV 5 μm 100,000 Å, 8 × 300 mm (p/n SDA0830051e5) SDV 5 μm 1,000,000 Å, 8 × 300 mm (p/n SDA0830051e6)
Temperature	23 °C
Sample Concentration	1 mg/mL (0.5 mg/mL >1,000,000 Da)
Calibration	Agilent ReadyCal-Kit polystyrene (p/n PSS-PSKITR1) Agilent ReadyCal-Kit polymethyl methacrylate (p/n PSS-MMKITR1)
Detectors	Refractive index (RI) detector
Software	Agilent WinGPC

Results and discussion

SDV columns are a typical stationary phase for nonpolar solvents, and these columns are fully compatible with 2-methyITHF. Solvent exchange between THF and 2-methyITHF is straightforward, without any loss of column efficiency when measuring plate count.

Polystyrene (PS) and polymethyl methacrylate (PMMA) reference materials show good solubility in 2-methylTHF, and GPC/SEC measurements are tested. Using a RI detector leads to reasonable signal-to-noise ratios for both polymer types. Figure 1 depicts an overlay of three different PS mixtures with 12 standards in total covering a molecular weight range of Mp 474 to 2,520,000 Da.

The overlay of 12 different PMMA standards as three mixtures, covering a comparable molecular weight range from Mp 800 to 2,200,000 Da, is shown in Figure 2.

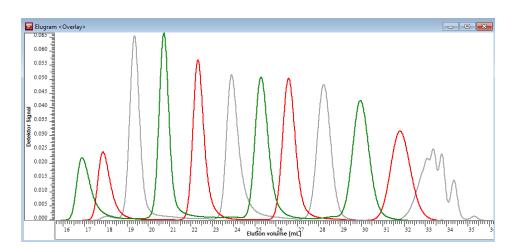


Figure 1. Overlay of three different PS mixtures (RI traces).

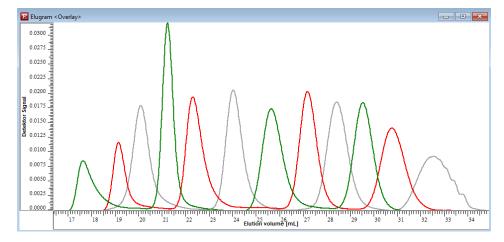


Figure 2. Overlay of three different PMMA mixtures (RI traces).

Conclusion

2-MethylTHF, a sustainable replacement for THF, is feasible as a mobile phase in GPC/SEC for common nonpolar applications such as polystyrene or poly(meth)acrylate measurements. The use of SDV columns as stationary phase enables robust and reliable GPC/SEC measurements with RI detection.

References

- 1. Pace, V. et al.
 - 2-Methyltetrahydrofuran (2-MeTHF): A Biomass-Derived Solvent with Broad Application in Organic Chemistry. *Chem. Sus. Chem.* **2012**, *5*(8), 1369–1379.

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