Application Brief

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Characterization of Polystyrene Sulfonic Acid

GPC/SEC analysis in aqueous solution without using organic modifiers as additives

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Abstract

This application brief describes the GPC/SEC characterization of poly(styrene sulfonic acid) (PSA) by using Agilent MCX columns under aqueous alkaline conditions.

Introduction

Poly(styrene sulfonic acid) (PSA) is prepared from polystyrene by sulfonation or by polymerization of styrene sulfonic acid (alkaline salt). It is a strong polyanion with good solubility in aqueous media. However, the presence of a phenyl group in each monomer causes a hydrophobic character, which hinders interaction-free separation during GPC/SEC analysis.[1]

MCX columns are based on porous sulfonated styrene-divinylbenzene particles, which allow robust and reliable chromatography of strong anionic macromolecules such as PSA in aqueous mobile phases without adding organic modifiers such as acetonitrile or methanol. The stationary phase has an anionic surface under basic conditions, and MCX columns are ideal for GPC/SEC analysis of polyanions in aqueous eluents, having a pH in the range of 7 to 13.

Experimental

Table 1. Instrument and sample conditions.

	Conditions
Pump	Isocratic pump Flow rate: 1 mL/min Mobile phase: aqueous 0.067 M disodium hydrogen phosphate
Injection System	Autosampler Injection volume: 20 µL
Columns	MCX high MW combination: MCX 10 µm precolumn, 8 × 50 mm (p/n MCA080510) MCX 10 µm 1,000 Å, 8 × 300 mm (p/n MCA0830101e3) MCX 10 µm 100,000 Å, 8 × 300 mm (p/n MCA0830101e5) MCX 10 µm 10,000,000 Å, 8 × 300 mm (p/n MCA0830101e7)
Temperature	23 °C
Sample Concentration	2 to 3 mg/mL
Calibration	Agilent kit polystyrene sulfonate sodium salt (p/n PSS-PSSKIT)
Detectors	Variable wavelength UV-Vis detector (VWD) at λ = 254 nm Refractive index (RI) detector
Software	Agilent WinGPC

Results and discussion

Use of a basic mobile phase like aqueous 0.067 M disodium hydrogen phosphate solution with a pH of about nine transfers the polymeric sulfonic acid sample into polystyrene sulfonate sodium salt (PSS). The stationary phase has a comparable surface under this condition, which enables robust and reliable GPC/SEC.

Figure 1 shows the elugram of a broadly distributed PSS measured with a set of three MCX 10 µm columns and a 10 µm guard column.

Conventional calibration with polystyrene sulfonate sodium salt standards allows the analysis of absolute molar masses, as shown in Figure 2.

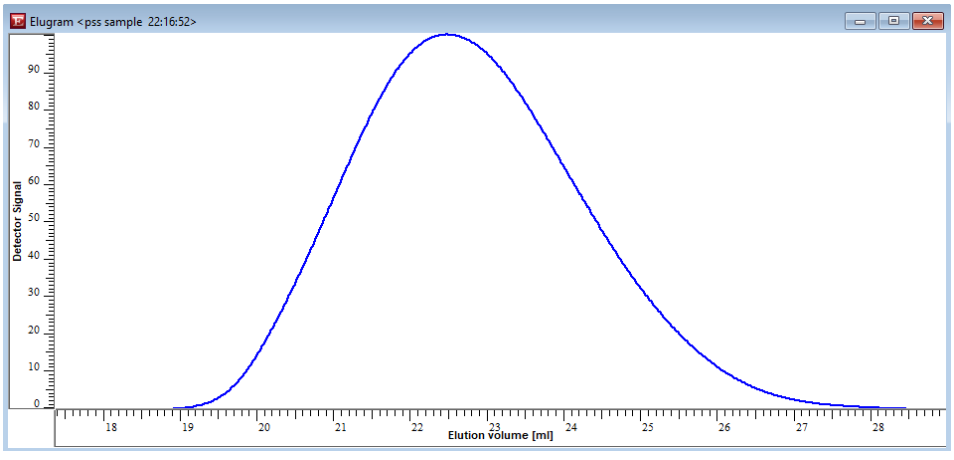

Figure 1. Elugram (UV at 254 nm trace) of a broadly distributed PSS sample.

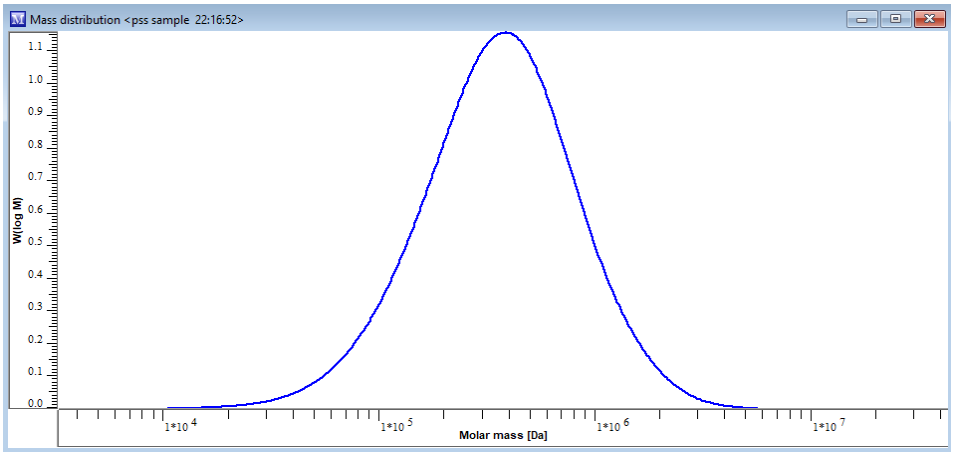

Figure 2. Molecular weight distribution (MWD) (UV at 254 nm trace) of a broadly distributed PSS sample.

Conclusion

Robust and reliable GPC/SEC analysis of PSA and PSS in aqueous basic mobile phases (e.g., aqueous 0.067 M disodium hydrogen phosphate), without addition of organic modifiers, can be achieved by using Agilent MCX columns as stationary phase.

Reference

1. Coughlin, J. E. *et al.* Sulfonation of Polystyrene: Toward the "Ideal" Polyelectrolyte *J. Polym. Sci., Part A: Polym. Chem.* **2013**, *51*, *11*, 2416–2424.

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