

# Separation of Styrene Oligomers by Agilent Preparative Gel Permeation Chromatography

# **Application Note**

Materials Testing and Research

#### **Author**

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#### Introduction

Preparative gel permeation chromatography separates and isolates individual components of a sample based on size exclusion. If based on an analytical separation, prep GPC can be used to isolate practical quantities of individual components, which are thus made available for further analysis. The preparative separation of individual oligomers from oligomer distributions and complex mixtures is easily achieved with the Agilent OligoPore preparative GPC column. This application note illustrates the use of Agilent OligoPore preparative columns in the fractionation of polystyrene oligomers.





### **Styrene Oligomer Analysis**

Initially, the optimum loading of polystyrene 580 on the Agilent OligoPore columns was analyzed at an analytical scale. The Agilent polystyrene narrow standard Mp 580 (p/n PL2012-2001) is composed of a distribution of styrene oligomers differing by a repeat unit of relative molecular mass 104.

### **Conditions - Analytical**

Sample Polystyrene 580, 0.5 to 2.0% (w/v)

Columns 2 × Agilent OligoPore, 7.5 × 300 mm

(p/n PL1113-6520)

Eluent THF

Flow rate 1.0 mL/min

Inj vol 100  $\mu L$ 

Detector UV

System Agilent PL-GPC 50

Figure 1 shows a series of analytical chromatograms at concentrations ranging from 0.2% to 2.0% (w/v). The chromatograms indicate that polystyrene 580 could be analyzed at a concentration of 2.0% (w/v) without serious loss of resolution.

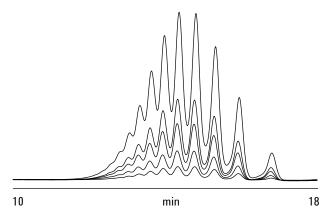


Figure 1. Analytical separation of polystyrene 580 on Agilent OligoPore indicates that a 2.0% w/v concentration is appropriate for preparative analysis.

Agilent OligoPore preparative columns were used to fractionate and collect the individual oligomers. A preparative GPC system was set up with a 2 mL injection loop, two Agilent OligoPore  $25 \times 300$  mm columns and a flow rate of 10.0 mL/min, an approximate ten-fold scale up over the analytical separation. The flow from the columns was split into two lines, approximately 0.5 mL/min went to a UV detector, the remainder of the flow to a waste/fraction collector. The polystyrene sample was injected at a concentration of 1.0% (w/v).

# **Conditions - Preparative**

Sample Polystyrene 580, 1.0% (w/v)

Columns 2 × Agilent OligoPore, 25 × 300 mm

(p/n PL1213-6520)

Eluent THF

Flow rate 10.0 mL/min, about 9.5 mL/min collected;

0.5 mL/min to the detector

Inj vol 2 mL Detector UV

System PL-GPC 50

Figure 2 is a chromatogram of polystyrene 580 obtained on the preparative columns indicating the resolution obtained. The sample was re-run and the individual oligomer fractions collected. Each oligomer fraction was then analyzed on two Agilent OligoPore analytical columns.

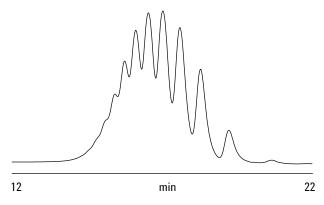


Figure 2. Polystyrene 580 separated on an Agilent OligoPore two-column set.

Figure 3 shows the original analytical chromatogram of polystyrene 580 run at a concentration of 2.0 (w/v) and an overlay of nine analytical chromatograms of the individual oligomer fractions collected from the Agilent OligoPore preparative GPC columns. The individual oligomers can be identified by comparison with the retention times in the chromatogram of polystyrene 580.

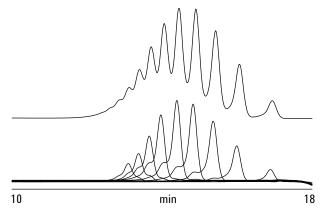


Figure 3. Polystyrene 580 from Figure 1 run at 2.0% (w/v) compared to nine overlaid analytical chromatograms collected from the Agilent OligoPore prep columns.

# **Conclusions**

Preparative gel permeation chromatography can be used to separate and isolate individual oligomers from complex samples once method development has been performed on analytical column equivalents.

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