Separate Epoxy Resin Oligomers with Agilent Preparative Gel Permeation Chromatography

Application Note

Materials Testing and Research

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Introduction

Preparative gel permeation chromatography (GPC) can be used to separate and isolate individual components of a sample based on size exclusion. By scaling up analytical separations, preparative GPC isolates practical quantities of individual components that can be used in further analysis. The Agilent OligoPore preparative GPC column is ideally suited to the separation and isolation of individual oligomers from oligomer distributions and complex mixtures. In this example, the columns are employed for the fractionation of epoxy oligomers.

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Epoxy Resin Olgomer Analysis

Figure 1 shows the general structure of an epoxy oligomer such as Epikote 828. This commercial epoxy resin is composed of two main epoxy oligomers where n = 0 and n = 1, and small amounts of the mono- and di-epoxy water adducts.

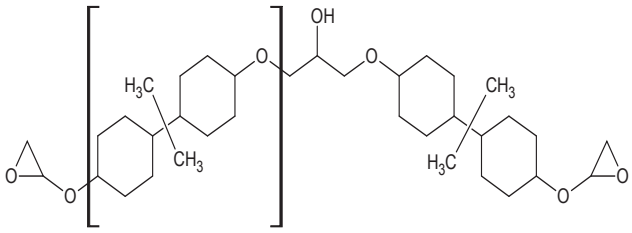

Figure 1. General structure of Epikote 828 epoxy resin oligomers.

Initially, the optimum loading of Epikote 828 on the OligoPore columns was analyzed on an analytical scale. Figure 2 shows analytical chromatograms at concentrations of 0.5% to 2.0% (w/v). They indicate that Epikote 828 could be analyzed at a concentration of 2.0% (w/v) without serious loss of reduction.

Conditions - Analytical

Samples	Epikote 828, 0.5-2.0% (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 µL
Detector	UV
System	Agilent PL-GPC 50

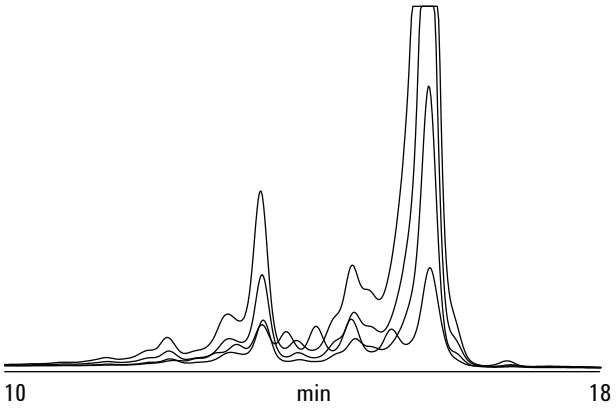

Figure 2. Analytical separation of Epikote 828 on Agilent OligoPore columns indicates that a 2.0% w/v concentration is appropriate for preparative analysis.

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

Figure 3 shows a chromatogram of Epikote 828 obtained on the preparative columns indicating the resolution. The sample was re-run and the two oligomers n = 0 and n = 1 were collected. The fractions were then analyzed on two Agilent OligoPore analytical columns.

Conditions - Preparative

Samples	Epikote 828, 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

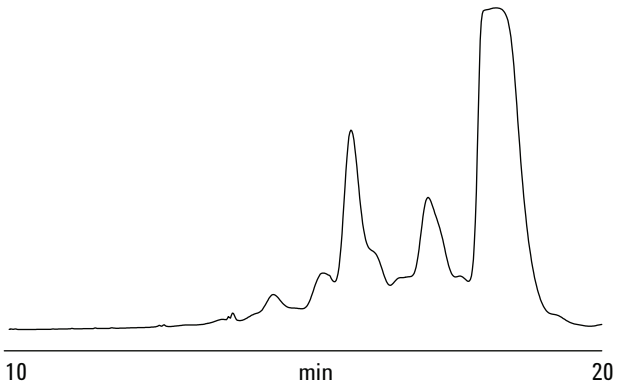

Figure 3. Epikote 828 separated on an Agilent OligoPore two-column set.

Figure 4 shows the original analytical chromatogram of Epikote 828 run at a concentration of 2.0% (w/v) and an overlay of analytical chromatograms of the n = 0 and n = 1 oligomers collected from the Agilent OligoPore preparative GPC columns.

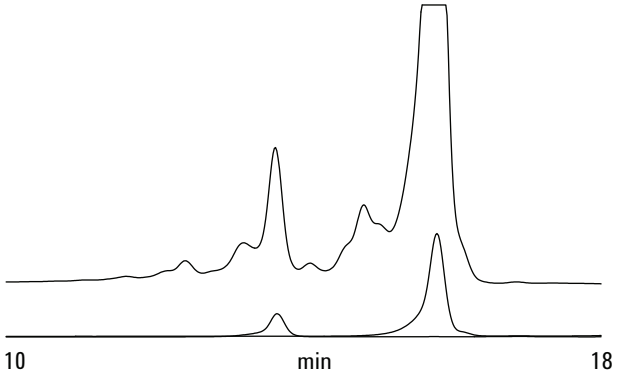

Figure 4. Epikote 828 analytical chromatogram from Figure 1 run at 2.0% (w/v) compared to overlaid analytical chromatograms of the n = 0 and n = 1 oligomers collected from the Agilent preparative GPC system.

Conclusions

Low pore size preparative GPC columns from Agilent can be used to isolate individual oligomers from complex samples after method development with an equivalent analytical-scale column.

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