Application Note

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Determination of Epoxy Resin Polymers by GPC with the Agilent 1290 Infinity II LC Equipped with the Agilent 1290 Infinity II RID

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Abstract

This application note demonstrates the advantage of using the Agilent 1290 Infinity II Micro Refractive Index Detector (RID) for GPC in an Agilent 1290 Infinity II LC. The UHPLC system in combination with the Micro RID enables the use of GPC columns with narrow inner diameters of 2.1 mm. This leads to better resolution and lower solvent consumption compared to a standard HPLC GPC system equipped with a standard RID.

Introduction

Modern UHPLC instruments provide the highest performance possible for high backpressure, high flow rates, flow precision, and accuracy. Dead volumes are also minimized across the whole system. This enables labs to work with columns of the smallest inner diameter and sub-2 µm particles at elevated flow rates with fast gradients.

Scientists use this advantage in GPC applications. Micro-GPC can use small and long coupled columns with sub-2 µm particles. The high flow accuracy enables the required low flow rates in the µL/min range. The low delay volumes provide the highest oligomer resolution.

This application note describes the concept of micro-GPC using a 1290 Infinity II LC with the 1290 Infinity II Micro-RID. The determination of the molecular weight of different epoxy resin samples demonstrates that the results acquired at different flow rates are the same (comparability to standard conditions has been already proven).[1] Using the Micro-RID enables labs to use smaller columns with less solvent consumption for better resolution.

Experimental

Agilent 1290 Infinity II GPC

- Agilent 1290 Infinity II High Speed Pump (G7120A)
- Agilent 1290 Infinity II Vialsampler (G7129B)
- Agilent 1260 Infinity II Multicolumn Thermostat (G7116A)
- Agilent 1290 Infinity II Refractive Index Detector (G7162A - Micro-RID or
- Agilent 1260 Infinity II Refractive Index Detector (G7162B)

Software

Agilent OpenLab CDS 2.3 and the GPC add on (G7860AA)

Columns

- Agilent MesoPore, 2.1 × 250 mm (PL1913-5325)
- Agilent PLgel, 3 µm MiniMixE, 4.6 × 250 mm (PL1510-5300)

Calibration: EasiVial, PS-L 2 mL (PL2010-0401)

- Red vial (Mp [g/mol]: 47190, 9960, 2980, 580)
- Green vial (Mp [g/mol]: 30230, 7640, 1840, 370)
- Yellow vial (Mp [g/mol]: 18340, 4900, 935, 162)

Samples

Epoxy resins Epikote: A and B of different molecular weight and oligomer distribution

Solvents

THF, tetrahydrofuran ≥99.9% inhibitor-free, CHROMASOLV Plus for HPLC, Riedel-de Haën

Method

Parameter	Value
Solvent	THF, isocratic, Channel B
Flow Rate, Column, and Stop Time	Column / Flow Rate (mL/min) / Stop Time (min) 1 / 0.06 / 23 2 / 0.3 / 22 2 / 0.6 / 11 2 / 1.0 / 7
Column Temperature	35 °C, Two columns in series connected (with 75 µm capillary, 105 mm length)
Injection Volume	20 µL (on 4.6 mm column), 3 µL (on 2.1 mm column)
RID	
Optical Unit Temperature	35 °C
Data Rate	9.25 Hz
Signal Polarity	Positive

Results and discussion

Higher throughput at increased flow rates with consistent molecular weight results

One major benefit of using the 1290 Infinity II LC for GPC is the highly precise delivery of flow rates starting from the µL/min range. This is combined with a pressure range that allows the use of multiple coupled columns with sub-2 µm fully porous solid phase material. The inherent advantage of an increased flow rate is higher sample throughput. The influence of the higher flow rates on resolution and the determined molecular weights was demonstrated by two samples of epoxy resin differing in molecular weight and oligomer distribution (Figures 1 and 2).

The epoxy resin A sample, with the lowest molecular weight and broadest oligomer content, showed sufficient resolution at all flow rates, and had comparable identified molecular weights (Figure 1). The throughput was improved by a factor of three with almost no compromise in the resolution of the low molecular weight oligomers inherent in this resin.

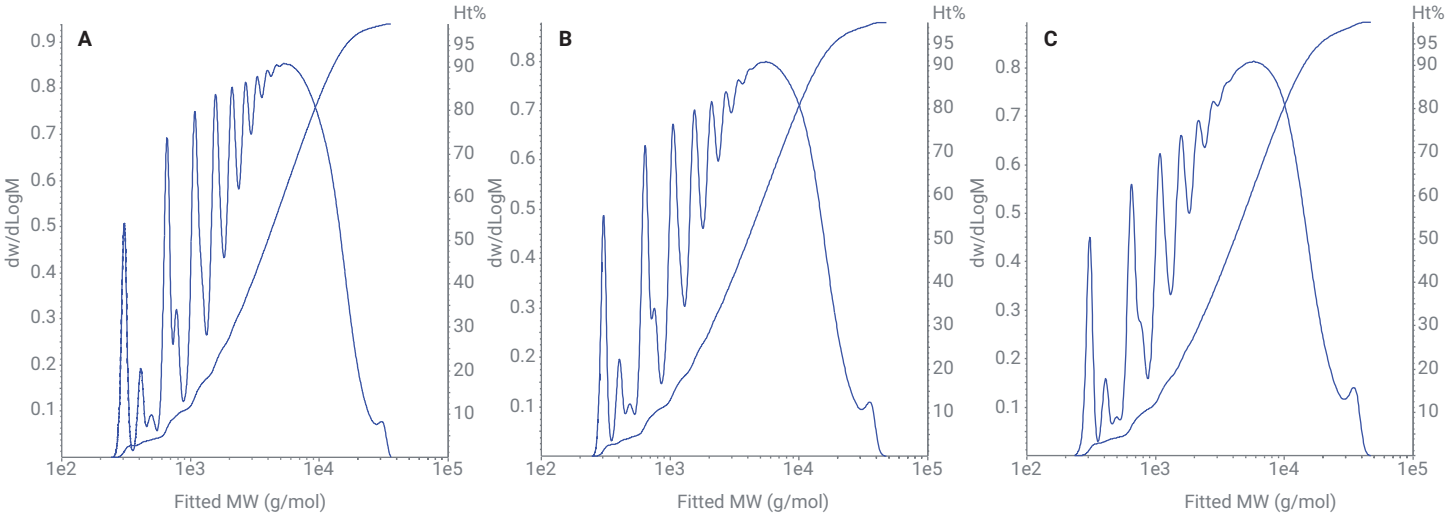

Figure 1. Epoxy resin A separated on two coupled PLGel columns (4.6 × 250 mm). The detection was done by the Agilent 1290 Infinity II Micro-RID. A) Flow rate: 0.3 mL/min, retention time: 11.69 minutes, Mn: 2,034 g/mol. B) Flow rate: 0.6 mL/min, retention time: 5.87 minutes, Mn: 1,999 g/mol. C) Flow rate: 1.0 mL/min, retention time: 3.53 minutes, Mn: 2,043 g/mol.

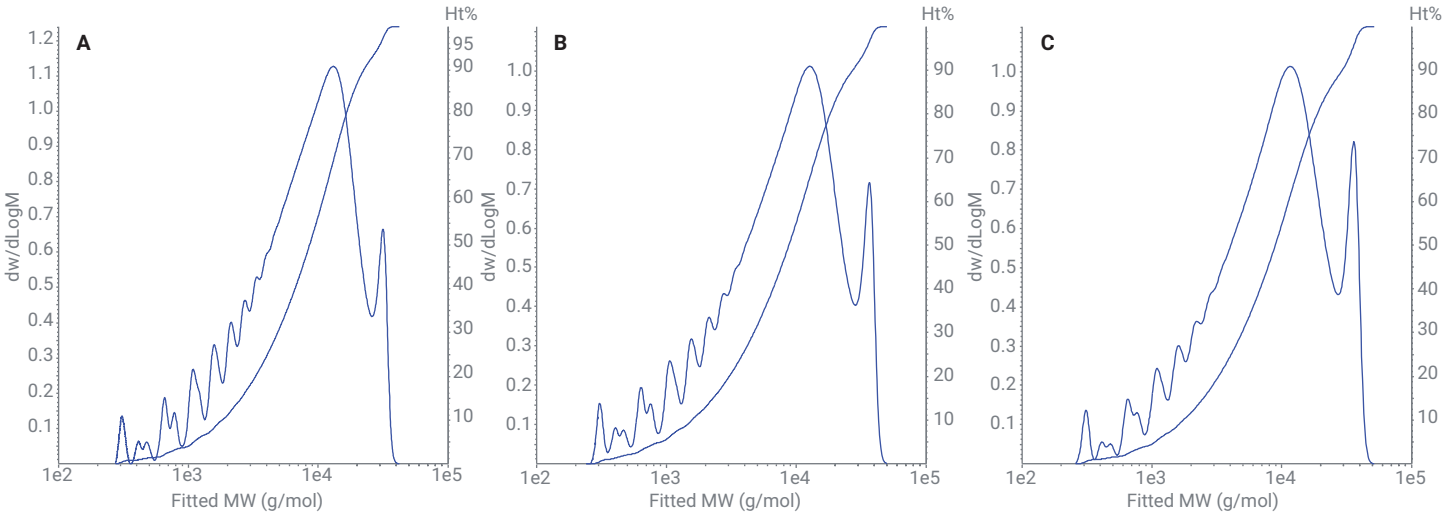

Figure 2. Epoxy resin B separated on two coupled PLGel columns (4.6 × 250 mm). The detection was done by the Agilent 1290 Infinity II Micro-RID. A) Flow rate: 0.3 mL/min, retention time: 10.85 minutes, Mn: 4,016 g/mol. B) Flow rate: 0.6 mL/min, retention time: 5.52 minutes, Mn: 3,625 g/mol. C) Flow rate: 1.0 mL/min, retention time: 3.36 minutes, Mn: 3,950 g/mol

In general, the epoxy resin B sample elutes at lower retention times for all applied flow rates due to the higher molecular weight and narrower oligomer distribution (Figure 2). Overall, the possibility for higher throughput could be retained, and the resolution for the oligomer fraction is good in all tests. In addition, a higher molecular weight fraction is resolved out of the major peak at the high molecular weight of distribution.

Table 1 displays a summary of the results from the experiments with the epoxy resin A and B samples. The retention times show the effect of the increase in flow rate and the possible increase in throughput. A comparison of the elution times at identical flow rates unravels the difference in molecular weights of the different samples. The calculated molecular weights for one sample are comparable over the different applied flow rates.

Maximum resolution using micro-GPC

The capability of the 1290 Infinity II High Speed Pump to deliver precise flow rates in the lower µL/min range opens the door to micro-GPC applications. Together with MicroBore columns, it offers the chance to save a large amount of solvent, which is typically harmful and expensive in GPC. In addition, the 1290 Infinity II Micro-RID used in the system offers excellent resolution (Figures 3 and 4). The oligomers inherent in the epoxy resin A sample were separated at a low flow rate of 60 µL/min in a comparative experiment using the Micro-RID and a standard RID (Figure 3). The lower molecular weight oligomer content of the sample showed better separation on the Micro-RID equipped with the low volume cell compared to the standard RID. The higher molecular weight epoxy resin B sample showed the same effect on the low molecular weight side of the distribution (Figure 4).

Table 1. Summary table of the GPC experiment done with the epoxy resin A and B samples. The calibration for the calculation of the molecular weights was done with the mentioned polystyrene calibration mixtures (see Experimental section).

mL/min	Sample	RT (min)	Mp (g/mol)	Mn (g/mol)	Mol wt (g/mol)	PD
0.3	A	11.694	6,326	2,034	5,629	2.77
0.6		5.871	6,733	1,999	6,107	3.06
1		3.536	7,087	2,043	6,221	3.05
0.3	B	10.853	13,385	4,016	10,499	2.61
0.6		5.520	13,118	3,625	11,536	3.18
1		3.362	12,045	3,950	11,871	3.01

Mn: number average molecular weight, Mol wt: weight average molecular weight, Mp: molecular weight at peak maximum, PD: polydispersity index

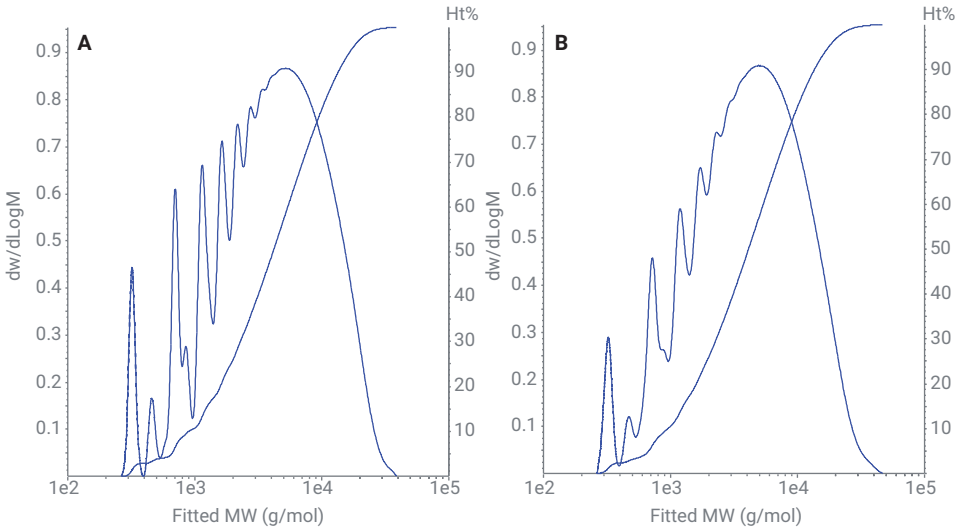

Figure 3. Epoxy resin A separated with a MesoPore column (2.1 × 250 mm) at a flow rate of 60 µL/min. A) Micro-RID, 2 µL cell, data rate 9.25 Hz. B) Standard RID, 8 µL cell, data rate 9.25 Hz

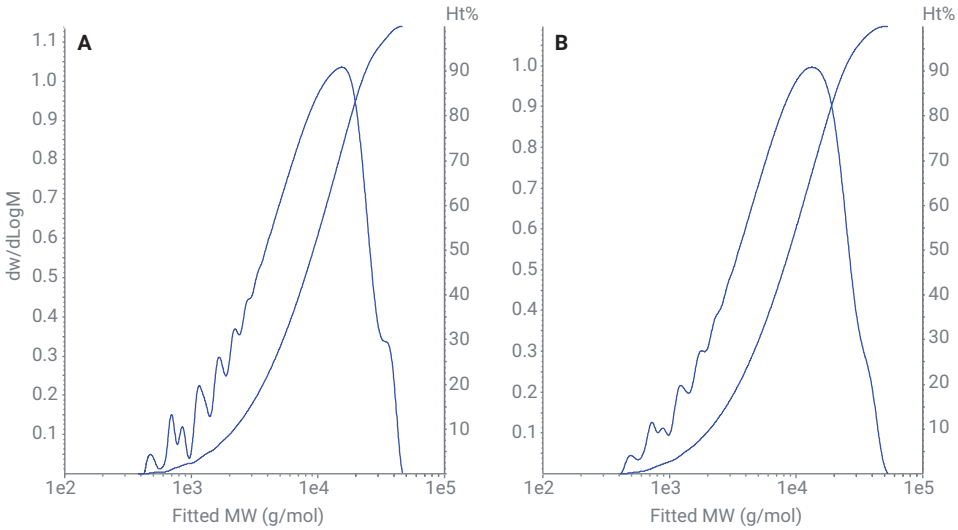

Figure 4. Epoxy resin B separated with a MesoPore column (2.1 × 250 mm) at a flow rate of 60 µL/min. A) Micro-RID, 2 µL cell, data rate 9.25 Hz. B) Standard RID, 8 µL cell, data rate 9.25 Hz

There was also a shoulder on the higher end of the molecular weight distribution, which was not very visible on the standard RID. Table 2 shows a summary of the experiments.

Conclusion

This application note demonstrates the superior capability of the Agilent 1290 Infinity II LC equipped with an Agilent 1290 Infinity II Micro-RID for GPC experiments. This study shows the possibility of working under micro-GPC conditions and saving up to 80% of solvent. In comparison to the standard RID, the micro-RID offers better resolution, which could lead to molecular weight calculations that better reflect the reality of the sample. The throughput could be increased by a factor of three while maintaining the resolution of the oligomer distribution in the epoxy resin samples and consistent molecular weight results.

Reference

1. Improved GPC with the Agilent 1290 Infinity II Micro RID, *Agilent Technologies Application Note*, publication number 5994-1089EN, **2019**.

Table 2. Summary table of the GPC experiment done with the epoxy resin A and B samples to compare the results obtained by means of the Agilent 1290 Infinity II Micro-RID and the standard RID. The calibration for the calculation of the molecular weights has been done with the mentioned polystyrene calibration mixtures (see Experimental section).

	Sample	RT (min)	Mp (g/mol)	Mn (g/mol)	Mol wt (g/mol)	PD
Micro	A	13.678	5,920.00	2,175.00	5,865.00	2.70
Std		15.392	5,703.00	2,239.00	5,949.00	2.66
Micro	B	12.446	16,206.00	4,962.00	11,468.00	2.31
Std		14.156	14,465.00	4,773.00	11,261.00	2.36

Mn: number average molecular weight, Mol wt: weight average molecular weight, Mp: molecular weight at peak maximum, PD: polydispersity index

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