

Reduction of GPC Analysis Time and Eluent Consumption by Column Downsizing

Gel permeation chromatography (GPC) analysis using a refractive index detector is conducted in measurements of the molecular weight distribution of synthetic polymers. In order to secure separation performance in GPC analyses, the elution volume up to the permeation limit is increased generally using a column with an internal diameter of 8 mm. In case where separation cannot be achieved with a single column, two or more columns in the same product series are sometimes connected in series. However, to meet the demand for reduced analysis time and eluent consumption in recent years, columns that realize the equivalent separation with a smaller elution volume have appeared and high speed analysis has become possible.

The Shimadzu Nexera™ GPC system is capable of covering the full range from conventional analysis to UHPLC analysis with a single setup and also supporting applications that require enough column length because the column oven can accommodate up to three columns with lengths of 300 mm generally used in GPC analysis.

This article introduces comparisons of analysis times and eluent consumptions on columns of various sizes in polystyrene (PS) analysis using Nexera GPC system.

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Reduction of Analysis Time and Eluent Consumption in GPC Analysis of Polystyrene

The sample used here was polydisperse PS (number average molecular weight: approximately 30,000). Fig. 1 shows a comparison of the chromatograms when PS (0.2 w/v%) was analyzed with three types of columns in different sizes, and Table 1 shows the analytical conditions. The analytical columns employed were a Shim-pack™ GPC-804 with an internal diameter of 8 mm for conventional GPC analysis, a Shodex™ GPC LF-604 (internal diameter: 6 mm, multi-pore type packing material), and a Shodex GPC HK-404L (internal diameter: 4.6 mm) for reduced analysis time and eluent consumption. The detector was an RID-20A refractive index detector with a dual temperature control function for the optical system and flow cell. Only when using the GPC HK-404L analytical column, a Shodex RI-504 semi-micro refractive index detector was used to minimize column external diffusion.

Table 2 summarizes the analysis time and eluent consumption when using the various columns. In comparison with the conventional GPC-804, the use of the GPC LF-604 reduced the analysis time by 50% and eluent consumption by 70%. Furthermore, both of the analysis time and the eluent consumption were able to be reduced by about 80% by using the GPC HK-404L.

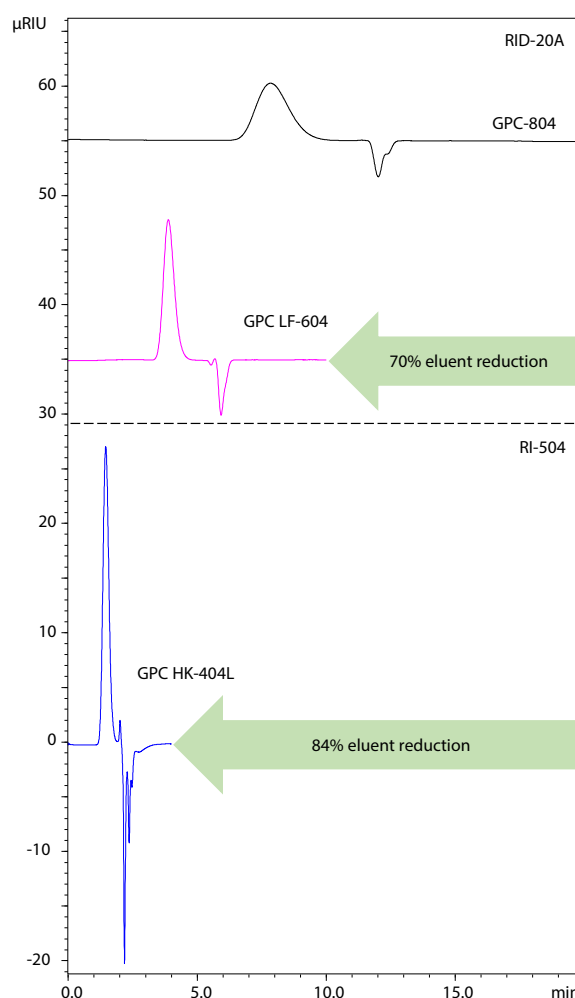


Fig. 1 Chromatograms of PS

Table 1 Analytical Conditions

System	: Nexera GPC system	<8 mm I.D. column>	<4.6 mm I.D. column>
Column	: Shim-pack GPC-804*1 (300 mm × 8.0 mm I.D.)	Shodex GPC LF-604 (150 mm × 6.0 mm I.D., 6 μm)	Shodex GPC HK-404L (150 mm × 4.6 mm I.D., 3.5 μm)
Flow rate	: 1.0 mL/min	0.6 mL/min	0.8 mL/min
Mobile phase	: THF (without stabilizer)	THF (without stabilizer)	THF (without stabilizer)
Column temp.	: 40 °C	40 °C	40 °C
Injection volume	: 20 μL	10 μL	5 μL
Vial	: SHIMADZU LabTotal for LC 1.5 mL, Glass*2	SHIMADZU LabTotal for LC 1.5 mL, Glass	SHIMADZU LabTotal for LC 1.5 mL, Glass
Detection(RI)	: RID-20A Cell temp. 40 °C	RID-20A Cell temp. 40 °C	Shodex RI-504 Cell temp. 40 °C

*1 P/N: 228-20807-91, *2 P/N: 227-34001-01

Table 2 Summary of Analysis Time and Eluent Consumption

Column	Analysis time	Reduction rate based on GPC-804	Eluent consumption	Reduction rate based on GPC-804
Shim-pack GPC-804	20 min		20 mL	
Shodex GPC LF-604	10 min	50%	6 mL	70%
Shodex GPC HK-404L	4 min	80%	3.2 mL	84%

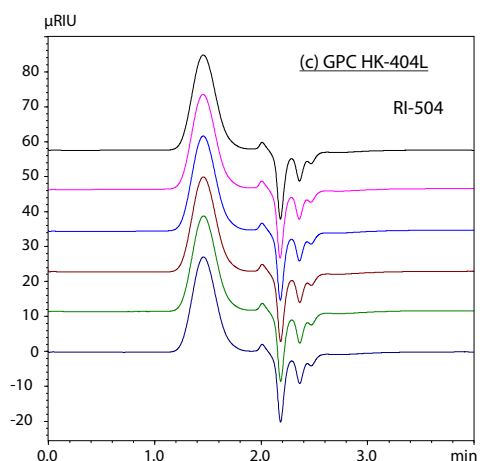
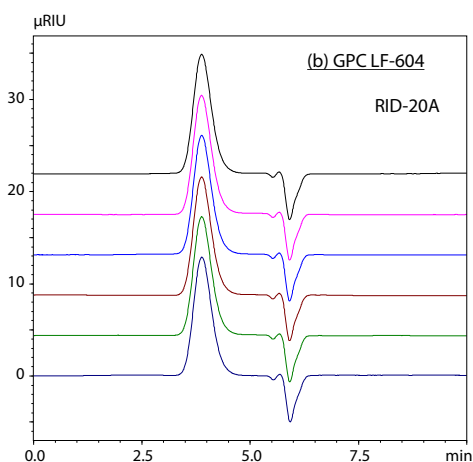
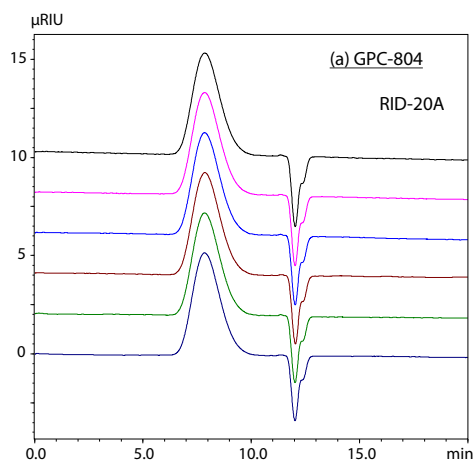


Fig. 2 Comparison of Chromatograms of PS

■ Repeatability

Fig. 2 shows a comparison of the chromatograms obtained when PS was analyzed repeatedly 6 times with each of the columns. Table 3 shows the repeatabilities (n = 6) of the elution time and the weight average molecular weight. Good repeatabilities were obtained with each of the three columns.

Table 3 Repeatability (%RSD, n = 6)

Column	Elution time	Mw
Shim-pack GPC-804	0.06	0.32
Shodex GPC LF-604	0.06	0.77
Shodex GPC HK-404L	0.12	1.15

■ Conclusion

PS was analyzed using a Nexera GPC system and GPC columns of various sizes to compare the analysis time and the eluent consumption. In comparison with the conventional GPC-804 column, the analysis time and eluent consumption were able to be reduced by 50% or more with the GPC LF-604, and 80% or more with the GPC HK-404L.

Satisfactory results for the repeatabilities of the elution time and weight average molecular weight of the PS were obtained with each of the columns.

(Notes)

- SUS parts were used for the piping and fittings downstream from the column outlet.
- An automatic rinsing kit was not used.

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