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Application News

High performance Liquid Chromatography

GPC Analysis Using Integrated LC System ~Calculation of Molecular Weight Distribution for Synthetic Polymers~

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User Benefits

- GPC system with small footprint can be setup by adding refractive index detector (RID-20A) to Integrated LC System "i-Series LC-2070".
- LabSolutions™ GPC option software provides intuitive operation from creation of calibration curve to calculation of molecular weight distribution.

■ Introduction

GPC (gel permeation chromatography) analysis is widely used as a straightforward analytical method for confirming the molecular weight distribution of synthetic polymers. Refractive index detector, which responds to the weight of the target compounds is often used for the detection for GPC analysis.

Integrated LC System "i-Series LC-2070" can be applied to GPC analysis by just configuring refractive index detector "RID-20A", resulting in small footprint GPC system setup. The column oven can accommodate up to three 300 mm columns, which are commonly used in GPC analysis despite the compact size of LC-2070. Fig. 1 shows the system configuration. In this article, the basic procedures from GPC analysis to data reprocessing, as well as the calculation of molecular weight distribution calculation using our workstation LabSolutions GPC option software are introduced.

(1) i-Series LC-2070
(2) RID-20A
(3) Reservoir tray

Fig. 1 System setup

■ Selection of eluent, standard, and column

In GPC analysis, the eluent is selected to be the same as the sample solvent. In non-aqueous GPC analysis, solvents such as tetrahydrofuran(THF), chloroform, and *N*,*N*-dimethylformamide(DMF) are often used, as they are capable of sufficiently dissolving the target compounds.

Next, select the GPC columns. If the molecular weight distribution of the sample is known, select appropriate columns based on the exclusion limits of the molecular weight of the GPC columns. It is essential a sufficient margin for the molecular weight range since the molecular weight distribution of the target polymer is often wider than expected. In some cases, multiple columns are serially connected to be used. If the molecular weight of the target polymers is unknown, there is an option of using mixed gel column having a wide target molecular weight range.

Next, select molecular weight standards (hereinafter referred to as standards) for creating calibration curves. In GPC analysis, approximately ten standards with specified peak top molecular weights are used to create a calibration curve, which indicates the relationship between elution time and molecular weight. The molecular weight of the target polymer is calculated based on the calibration curve. Select standards whose molecular weight range covers the molecular weight distribution of the target polymer to avoid the extrapolation from the real calibration points, resulting in reliable results. Additionally, the molecular weight determined by GPC analysis is essentially the equivalent molecular weight of the polymer employed as the standards.

■ Analytical conditions and sample preparation

In this study, GPC analysis acrylonitrile-styrene copolymer (AS) resin was performed employing THF and polystyrene (PS) as the sample solvent/eluent and the standards respectively. Detailed analytical conditions are shown in Table 1.

Eleven PS standards with molecular weights ranging from 580 to 1,230,000 were divided into three groups by taking every third standard in the order of molecular weight into same vials to avoid peak overlapping. The concentration of each standard was approximately 0.5 mg/mL.

The AS resin sample was dissolved in the eluent at a concentration of 1.0 mg/mL by manual stirring of the vials. Ultrasonication or stirring with a stirrer was not employed during sample preparation to prevent the cleavage of long polymer chain.

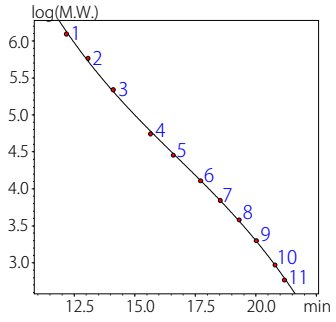
Table 1 Analysis Conditions

System	: i Series LC-2070*1	
Column	: Shim-pack™ GPC 805*2	(300 mm × 8.0 mm I.D.)
	Shim-pack GPC 803*3	(300 mm × 8.0 mm I.D.)
Flow rate	: 1.0 mL/min	
Mobile phase	: THF	
Column temp.	: 40 ˚C	
Injection vol.	: 10 µL	
Vial	: TORAST for LC 1.5 mL, Glass*4	
Detection	: RID-20A	
	Polarity	: +
	Cell temp.	: 40 ℃
	Response	: 1.5 sec

*1 Automatic rinsing kit is not used.
*2 P/N：228-20808-91
*3 P/N：228-20806-91
*4 P/N：GLCTV-901(Shimadzu GLC Ltd.)

■ Calibration curve

The calibration curve created from the analyses of the mixed standard solutions is shown in Fig. 2. LabSolutions GPC option software allows the creation of an optimal calibration curve by selecting proper approximation formula while confirming the chromatogram of the standard solution and the created trial calibration curve in the same window (Fig. 3).



1. PS (MW 1,230,000)
2. PS (MW 579,000)
3. PS (MW 217,900)
4. PS (MW 55,100)
5. PS (MW 28,500)
6. PS (MW 12,800)
7. PS (MW 6,930)
8. PS (MW 3,790)
9. PS (MW 1,990)
10. PS (MW 935)
11. PS (MW 580)

Fig. 2 Calibration curve created by PS standards

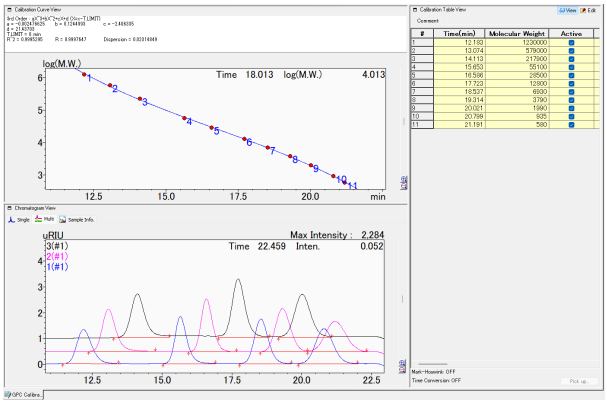


Fig. 3 Calibration curve creation screen (Upper left : calibration curve view, lower left : data file view, right : calibration table view)

■ GPC chromatogram and calculation for molecular weight distribution

Fig. 4 shows the chromatogram of AS resin (1.0 mg/mL) obtained using RID-20A for detection. The molecular weights corresponding to the start and end points of the peak are approximately 940,000 and 7,000 respectively, which are located within the range of the standard samples used for the calibration curve creation, suggesting that appropriate GPC calculation results can be obtained. The GPC data analysis screen of the software allows simultaneous confirmation of the chromatogram, calibration curve, and differential/integral molecular weight distribution curves (Fig. 5).

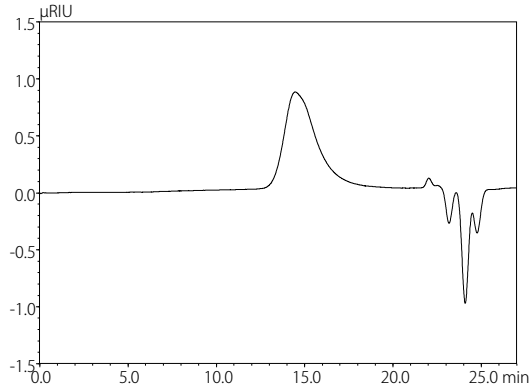


Fig. 4 Chromatogram of AS resin (RID-20A)

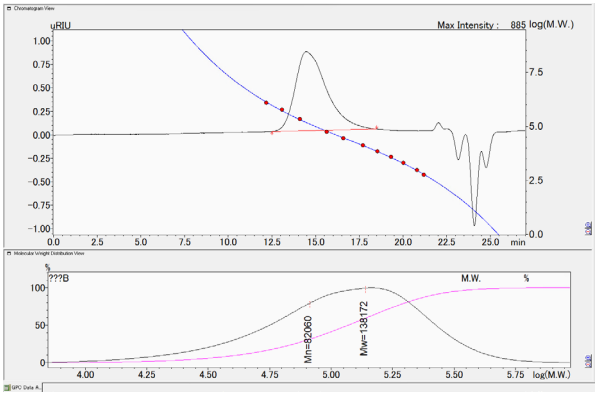


Fig. 5 GPC data analysis screen
(Upper : chromatogram view, lower : molecular weight distribution view)

■ Repeatability

Fig. 6 shows a comparison of chromatograms obtained by consecutive six times analyses of AS resin. Table 2 shows the mean values of the number-average molecular weight (Mn), weight-average molecular weight (Mw), and polydispersity (Mw/Mn), as well as their repeatabilities. In consecutive analyses, the repeatabilities of each average molecular weight was 0.8% or less, which was excellent value owning to the stabilities of solvent delivery, detector response, and column oven temperature.

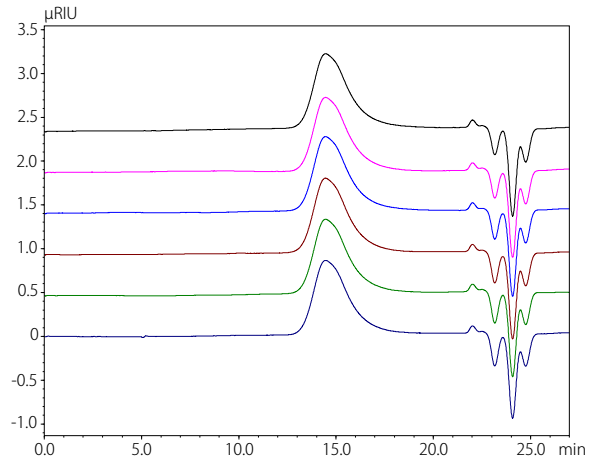


Fig. 6 Comparison of chromatograms obtained through consecutive analyses

Table 2 GPC calculation results of AS resin sample
(n=6, polystyrene equivalent molecular weight)

	Mn	Mw	Mw/Mn
AS resin	8.29×10^4	1.39×10^4	1.67
%RSD	0.75	0.34	0.54

■ Conclusion

Integrated LC System “i-Series LC-2070” is compatible with GPC analysis by adding refractive index detector “RID-20A,” and reliable data can be obtained from this compact system setup. The workstation option software provides an easy-to-understand data analysis screen that allows the smooth creation of calibration curves and the calculation of molecular weight distribution.

01-00930-EN First Edition: Sep. 2025

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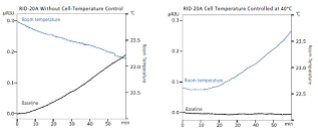
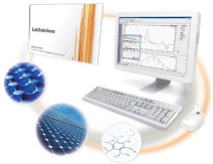


› RID-20A
High-Sensitivity Refractive Index Detector

› Shim-pack GPC Series
HPLC Column



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GPC Software

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