

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Application of Preparative Liquid Chromatography (No.3)

Recycle Separation of Oligomers on GPC Columns

■ Recycle Separation System Using the LC-6AD

The principle of recycle separation was reported in the Application News No.186. In order to successfully conduct a recycle separation, it is necessary to reduce extra-column band broadening. For this purpose, a parallel, double-plunger type of pump of small total volume is suitable. From this point of view, the LC-6AD can be made suitable by having its piping modified a little. The LC-8A (flow rate <math><150\text{ml}/\text{min}</math>) is also suitable for recycle separations, however, its inner volume is relatively large, so for a semipreparative separation (flow rate $\leq 20\text{ml}/\text{min}</math>), the use of the LC-6AD is more advantageous.$

Introduced here are application of this system to the efficiently conducted GPC of oligomers and low molecular compounds.

■ Recycle Separation of Styrene Oligomers

Figure 2 shows recycle separations of a commercially available molecular weight marker of polystyrene (MW = 761, Mw/Mn <math><1.15</math>), and the analytical conditions are shown in Table 1.

Recycle separations were conducted in the intervals shown by the \leftrightarrow mark. It can be seen that despite the small dispersivity of this marker, it is separated into more than 14 peaks.

In the case a refractometer is used to monitor the analysis, a solvent ghost peak appears (arrow in upper trace). To prevent solvent peak overlap with the target

Table 1 Analytical Conditions for Styrene Oligomers

Instrument	: LC-6AD Recycle Separation System
Sample	: Molecular Weight Marker of Polystyrene (MW = 761) 1% (w/v) Chloroform Solution $\times 1\text{ml}$
Column	: Shim-pack GPC-20025C + 2002C + 2001C (Each is 20mm I.D. $\times 300\text{mm}$ L.)
Mobile Phase	: Chloroform
Flow Rate	: 3ml/min.
Column Temp.	: Room Temperature (28°C)
Detector	: UV 254nm 0.04AUFS (0.1mmL. Cell)
Differential	: Refractive Index Detector 128×10^{-6} RIUFS (Temp. off)

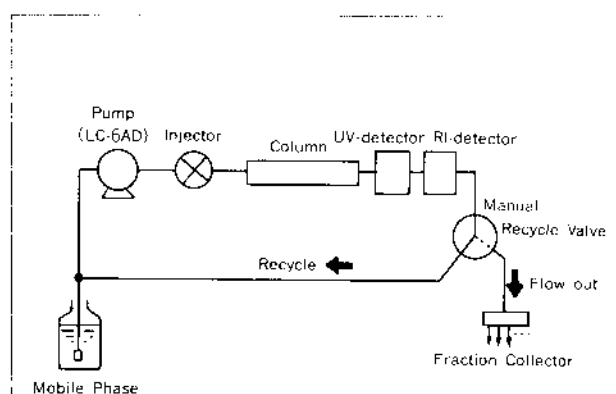


Fig. 1 Flow Diagram for Recycle Separations

components on the second and later elutions, it may be necessary to select a somewhat larger packing pore size than that which would normally be used in the non-recycle mode.

■ Recycle Separation of Triton X-100

Separation of Triton X-100 is shown in Fig. 3, and the analytical conditions in Table 2. It is seen that after six recycles, it was separated into more than 15 peaks.

Table 2 Analytical Conditions for Triton X-100

Instrument	: LC-6AD Recycle Separation System
Sample	: Triton X-100 1% (w/v) THF Solution $\times 1\text{ml}$
Column	: Shim-pack GPC-20025 + 2002 + 2001 (Each is 20mm I.D. $\times 300\text{mm}$ L.)
Mobile Phase	: THF
Flow Rate	: 3ml/min.
Column Temp.	: Room Temperature (25°C)
Detector	: UV 275nm 0.08AUFS (0.5mmL. Cell)

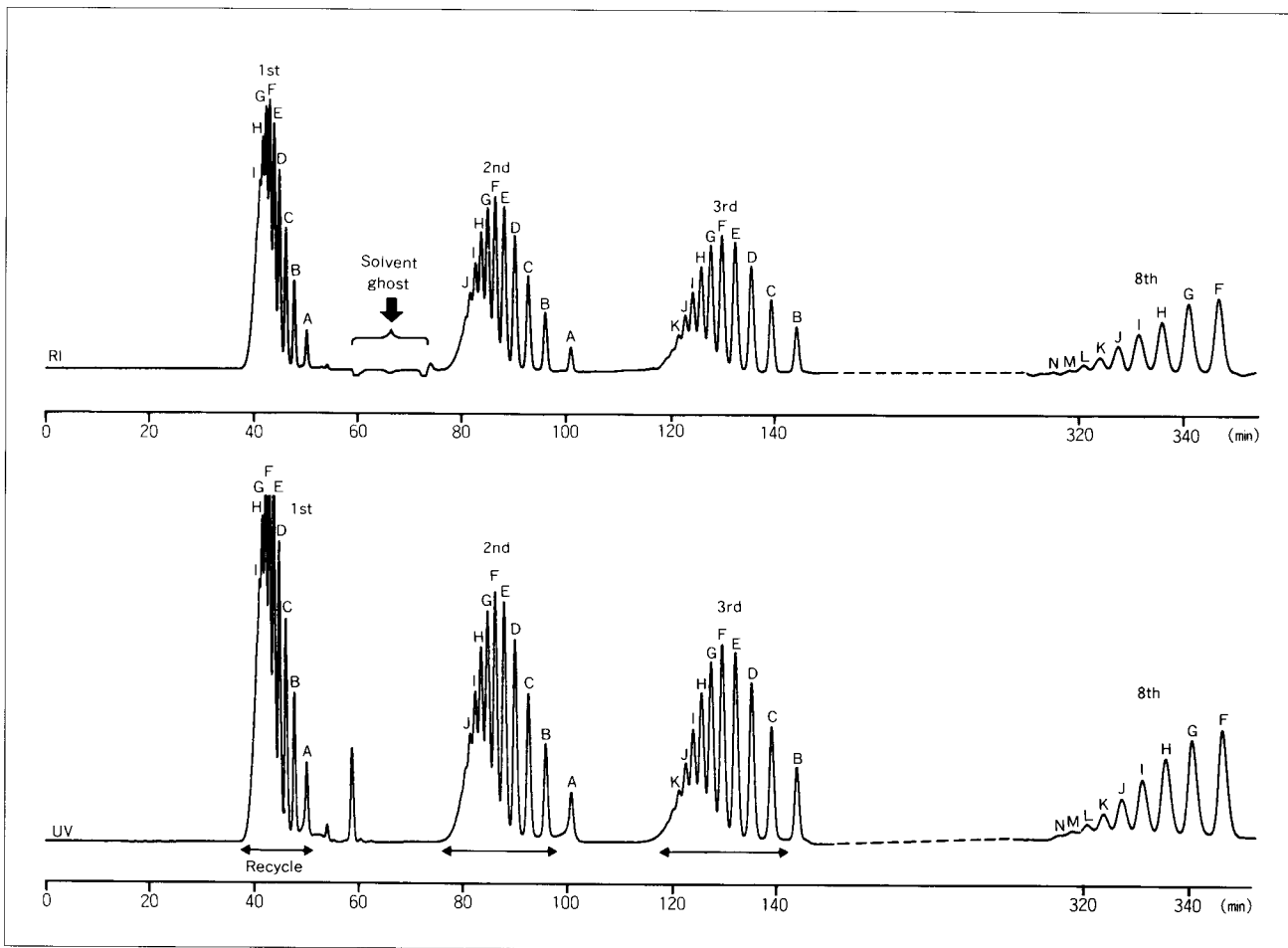


Fig. 2 Recycle Chromatograms for Styrene Oligomers

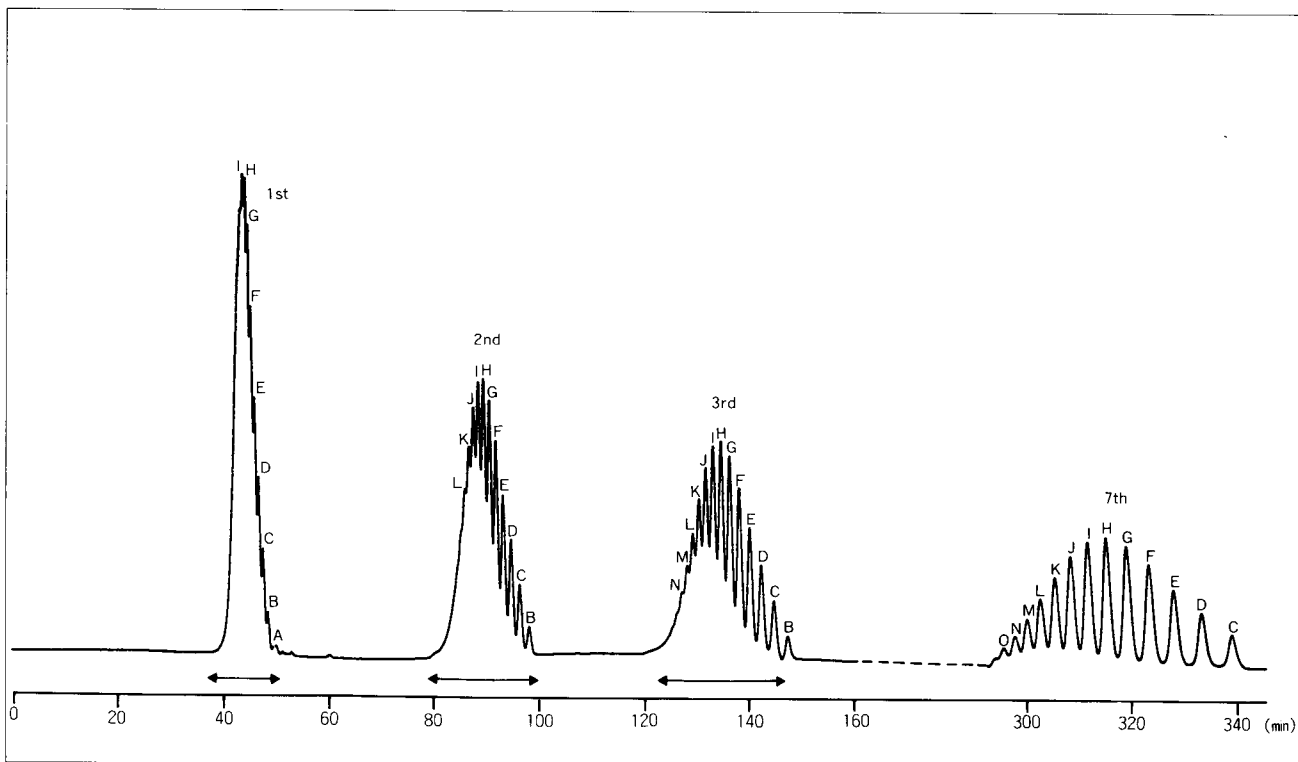


Fig. 3 Recycle Chromatogram for Triton X-100



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