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# Rapid Measurement of Molecular Weight by a Novel GPC Column

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## Abstract

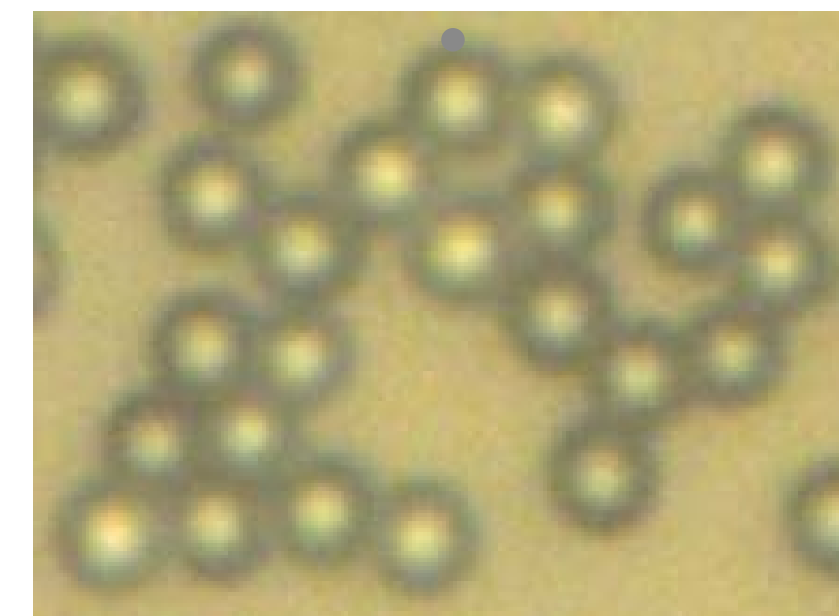
High speed analysis of samples has always been desired for GPC analysis, however the speed of liquid chromatography measurements is directly affected by the efficiency of analysis conditions. The column is the most important point to achieve rapid measurements. The biggest hurdle to overcome is the high back pressure generated by high flow rates which constricts the column design. Polymer gel has been preferably used for size exclusion chromatography (SEC) separation because of its variety of pore sizes, but has not been packed in columns for rapid measurement due to the concern that the high back pressure might crush the polymer gel, not allowing the column to work.

In order to solve the problem a porous cross-linked styrene-divinylbenzene polymer gel with sharp particle size distribution was developed to pack the Shodex HK column. The polymer gel successfully works to suppress back pressure, high flow rate, and GPC column for rapid measurement has been realized with the polymer gel.

The GPC column can be used for molecular weight measurement with the range from 100 to 1 million Da. The column constructed with a 4.6 mm inner diameter and 150 mm length dimension, has elution volume with less than 2.0 mL. It corresponds to less than 2.0 minutes of measurement time in case of 1.0 mL/min flow rate. At this flow rate, back pressure reaches 13.8 MPa, a suitable level for conventional HPLC equipment.

## Feature of novel GPC column : HK-404L

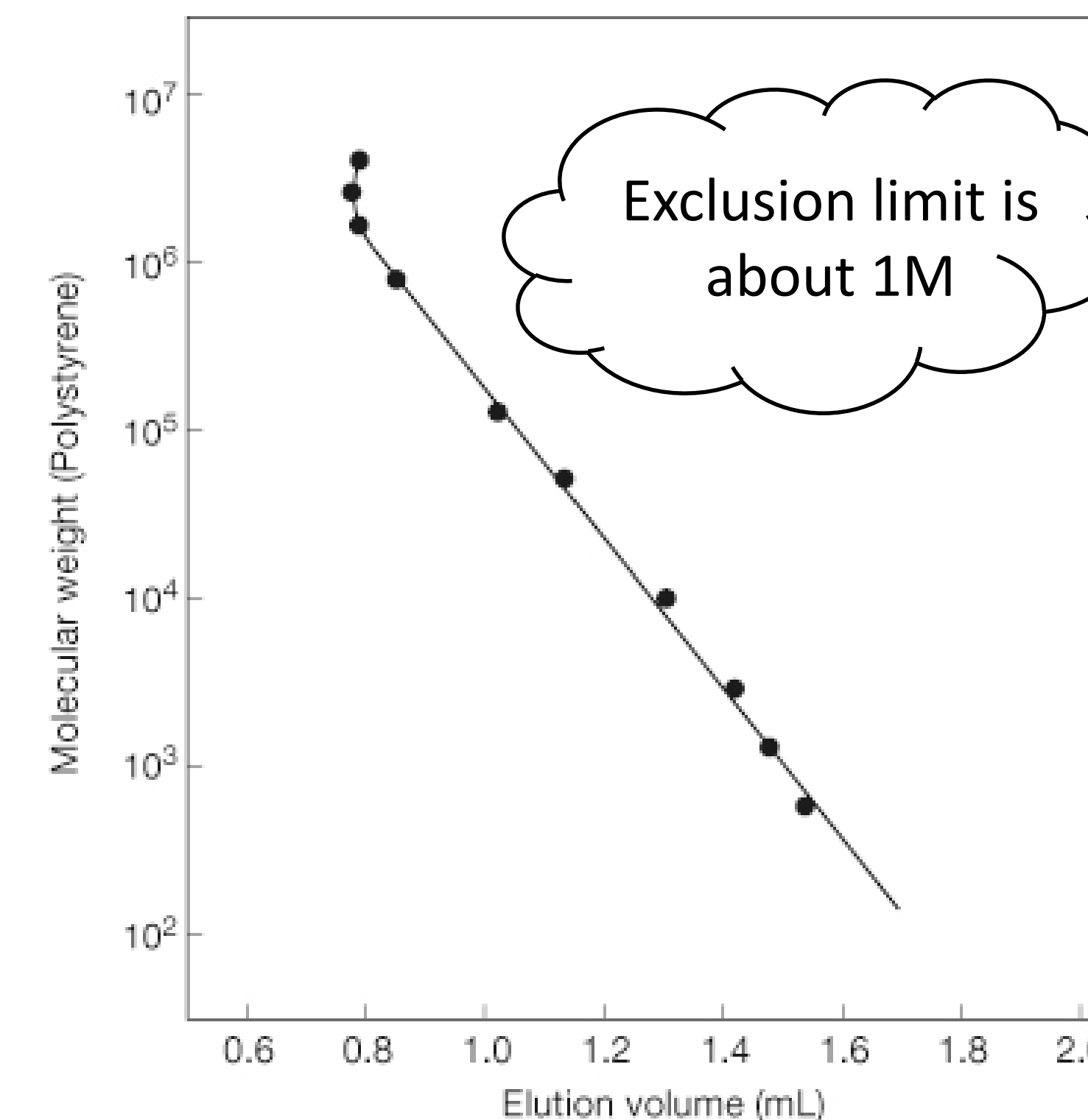
- Styrene-divinylbenzene copolymer with **very sharp size distribution** was the packing material.
- Excellent size distribution allows for a higher flow rate, because increase of column pressure is suppressed.
- Analysis of a wide molecular weight range, 100 to 1,000,000 by calibration curve with high linearity.
- The column is compatible with typical organic solvents for GPC such as THF, chloroform, DMF, toluene.



Particle size is uniform

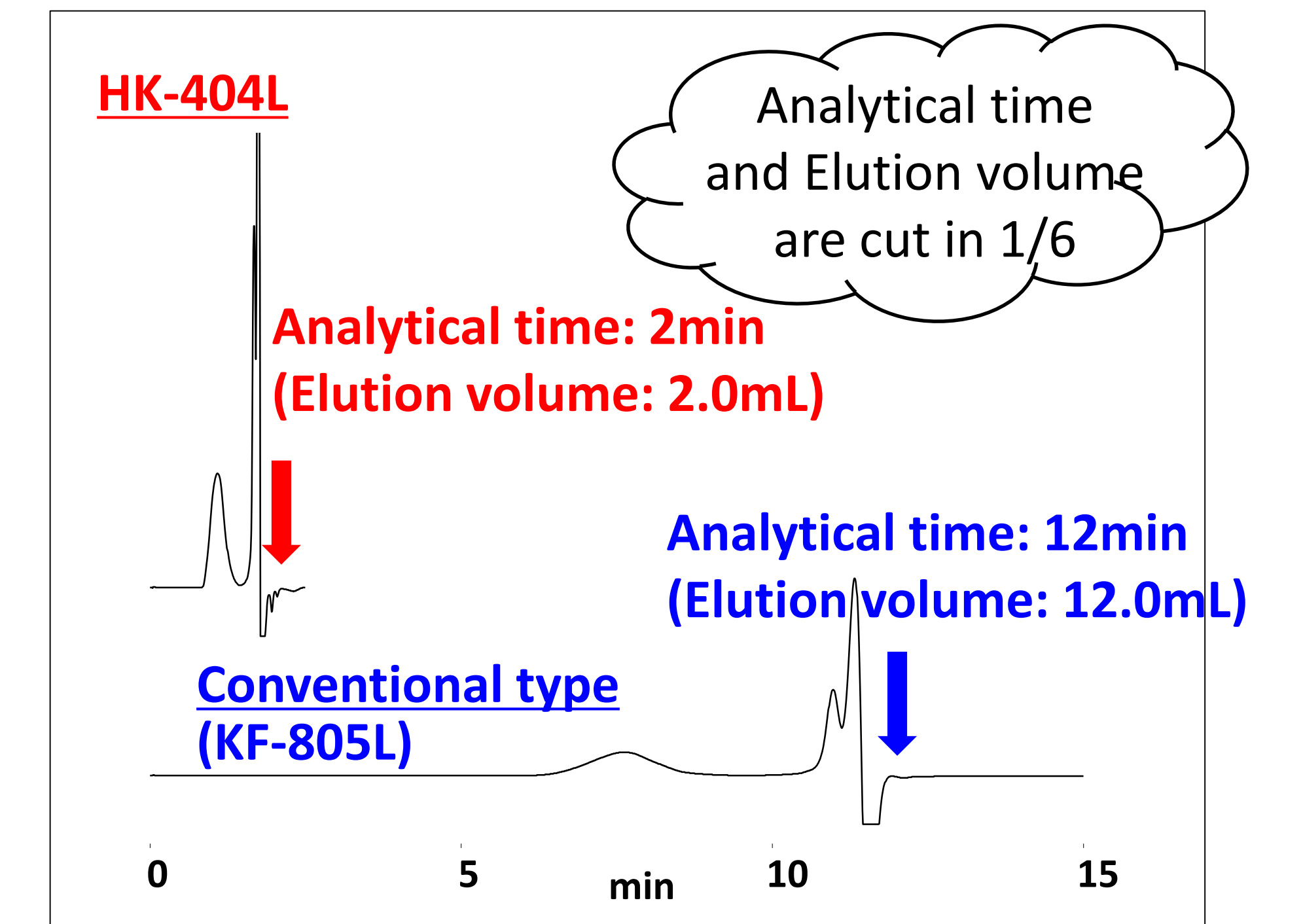
Optical microscopic image of novel styrene-divinylbenzene copolymer particle with well controlled the size at 3.5µm.

### Calibration curve with Polystyrene standard



Column : HK-404L (4.6mm I.D. x 150mm)  
 Eluent : THF  
 Flow rate : 1.0mL/min  
 Column temp. : 40°C  
 Detector : RI (small cell volume)  
**Pressure : 13.8MPa**

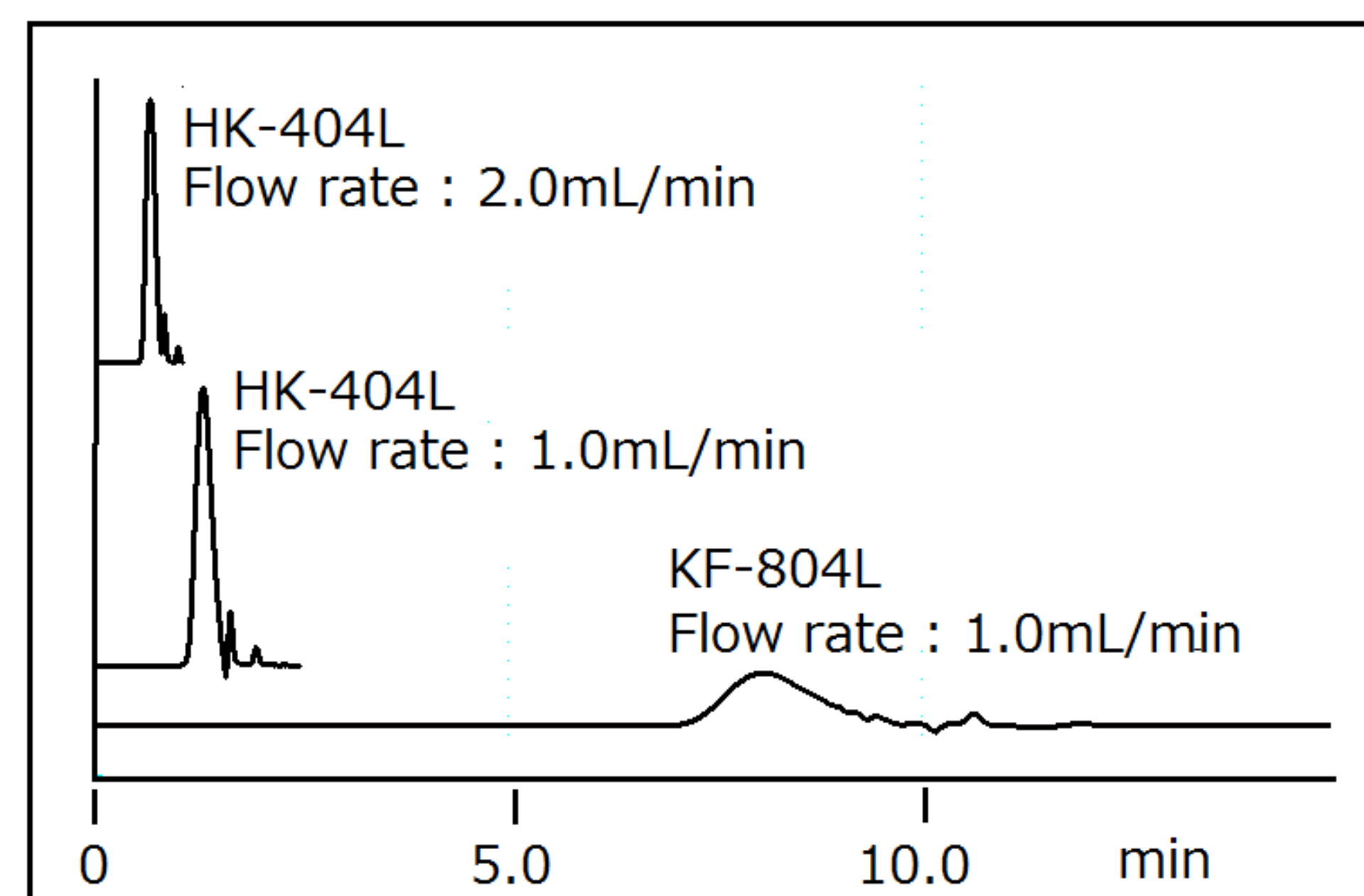
### Comparison with conventional GPC column



Column : HK-404L (4.6mm I.D. x 150mm)  
 KF-805L (8.0mm I.D. x 300mm)  
 Eluent : THF  
 Flow rate : 1.0mL/min  
 Column temp. : 40°C  
 Detector : RI (small cell volume)  
 Sample : 0.2% Poly(isobutyl methacrylate) 5µL

## Rapid analysis of Polycarbonate

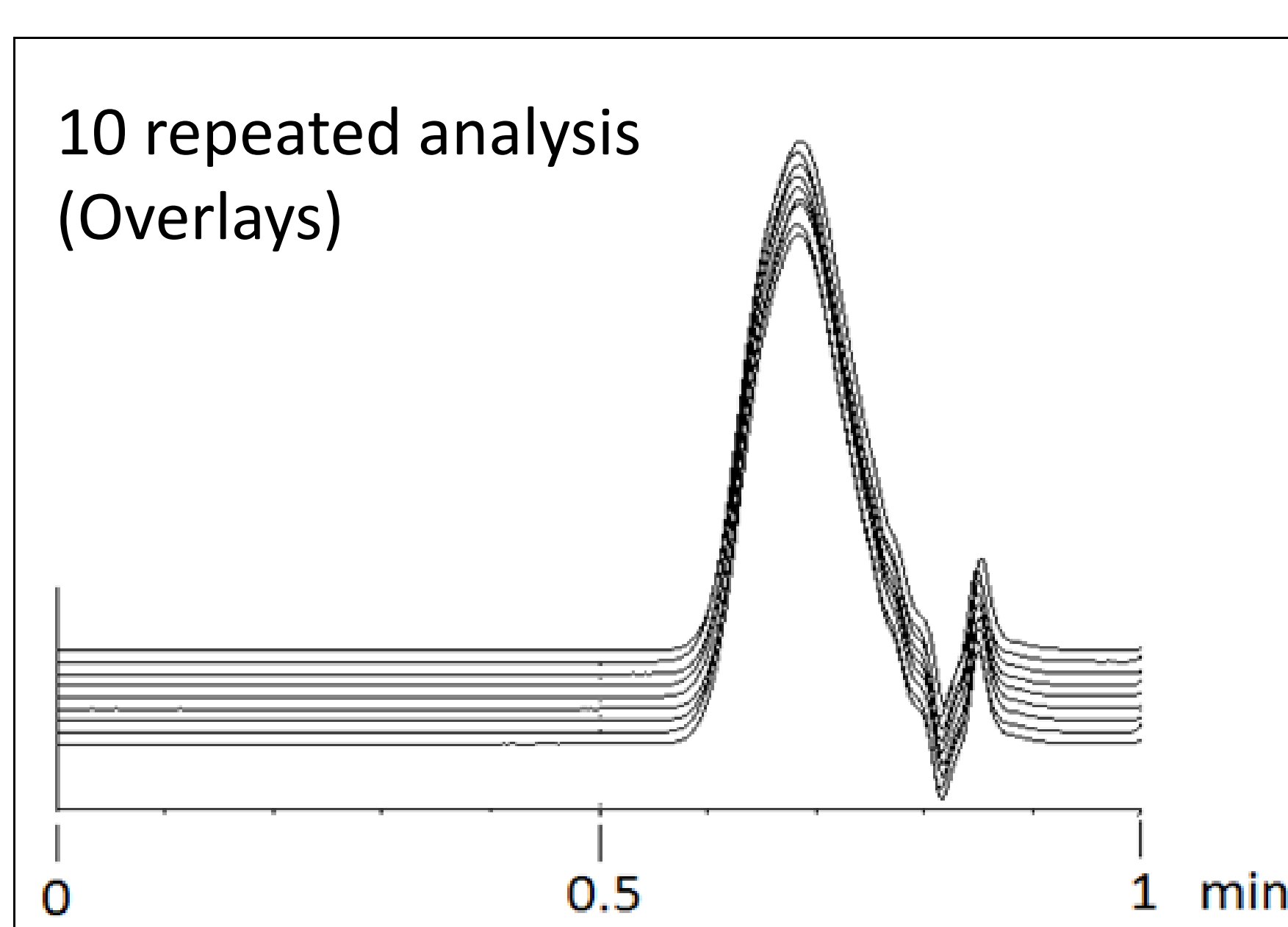
### Comparison with conventional column



Column : HK-404L (4.6mm I.D. x 150mm)  
 KF-804L (8.0mm I.D. x 300mm)  
 Eluent : THF  
 Column temp. : 40°C  
 Detector : UV 254nm (small cell volume)  
 Sample : 0.2% Polycarbonate, 5µL

Molecular weight measurement resulted with comparable peaks at higher flow rate

### Reproducibility of analysis



Column : HK-404L (4.6mm I.D. x 150mm)  
 Eluent : THF  
 Flow rate : 2.0mL/min  
 Column temp. : 40°C  
 Detector : UV 254nm (small cell volume)  
 Sample : 0.2% Polycarbonate, 5µL

Good reproducibility

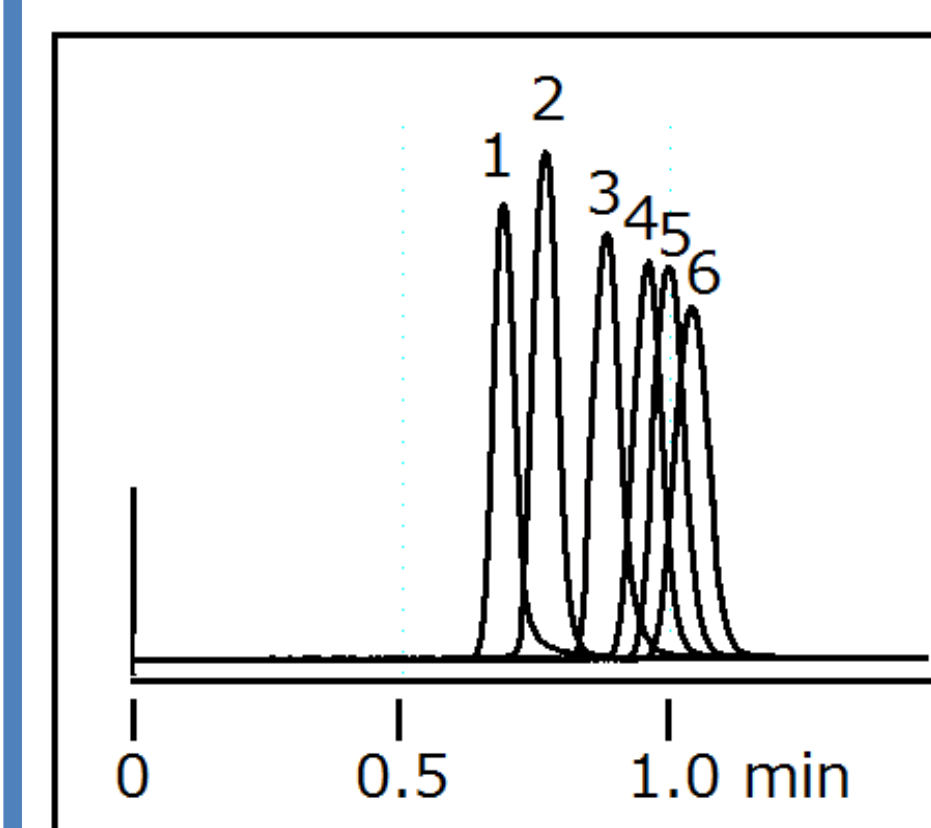
	Mn	Mw	Mw/Mn
HK-404L 2.0mL/min	4668	9854	2.11
HK-404L 1.0mL/min	4737	9370	1.98
KF-804L 1.0mL/min	4729	9927	2.10

Mn: Number average, Mw: Weight average

	Mn	Mw	Mw/Mn
1 <sup>st</sup> meas.	4668	9854	2.11
10 <sup>th</sup> meas.	4692	9933	2.12
Average	4675	9872	2.11
CV	1.8%	1.6%	0.3%

## Application data

### Polystyrene standard samples

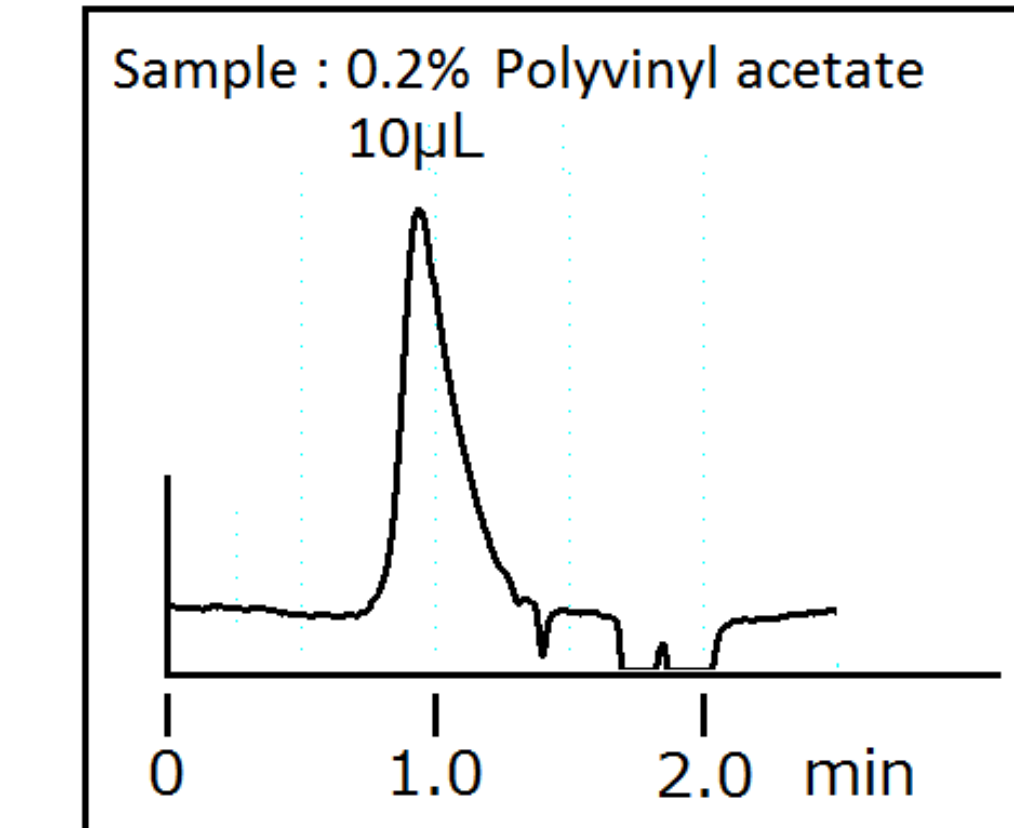


Sample : 5µL  
 1. Polystyrene (Mw: 127,000) 0.1%  
 2. Polystyrene (Mw: 51,000) 0.1%  
 3. Polystyrene (Mw: 10,000) 0.1%  
 4. Polystyrene (Mw: 2,900) 0.1%  
 5. Polystyrene (Mw: 1,300) 0.1%  
 6. Polystyrene (Mw: 580) 0.1%

Column : HK-404L (4.6mm I.D. x 150mm)  
 Eluent : THF  
 Flow rate : 1.5mL/min  
 Column temp. : 40°C  
 Detector : UV 254nm (small cell volume)

Each standard eluted with good peak shape

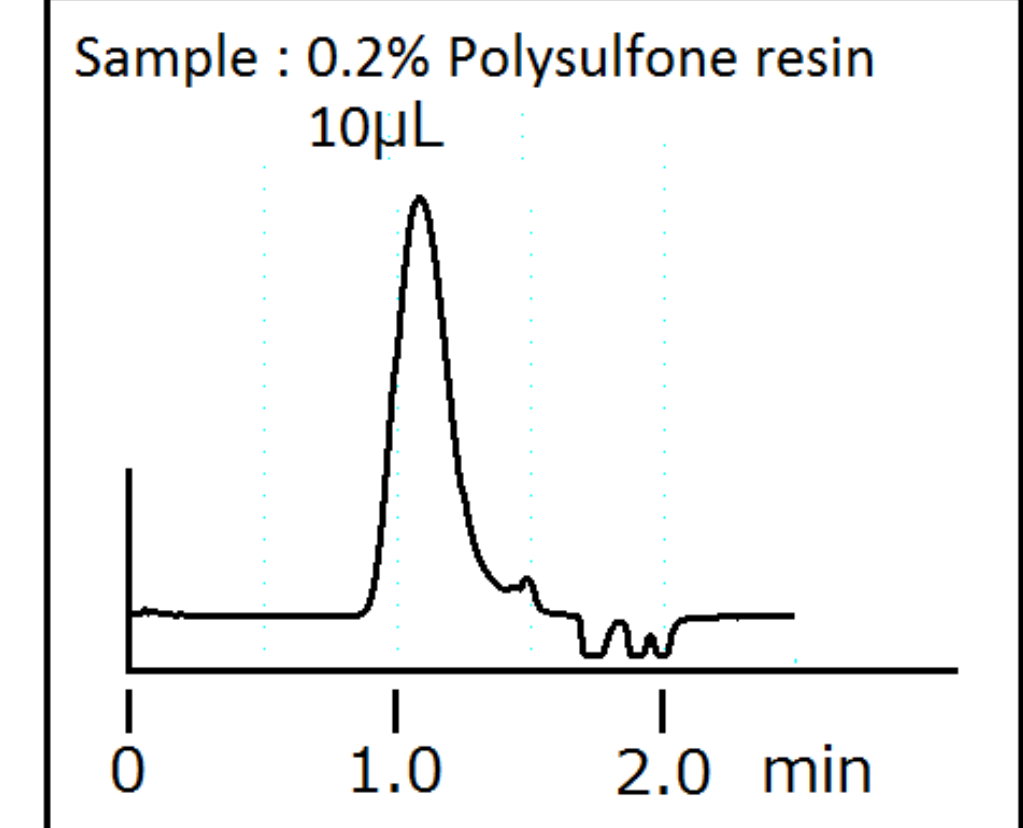
### Polyvinylacetate



Sample : 0.2% Polyvinyl acetate 10µL

Column : HK-404L (4.6mm I.D. x 150mm)  
 Eluent : THF  
 Flow rate : 1.0mL/min  
 Column temp. : 40°C  
 Detector : RI (small cell volume)

### Polysulfone



Sample : 0.2% Polysulfone resin 10µL

Common polymers are analyzed on HK-404L

## Conclusion

- Novel GPC column HK-404L packed with porous styrene-divinylbenzene copolymer having very sharp distribution of particle size can achieve suppressed pressure.
- HK-404L can elute polycarbonate within 2min with 1.0mL/min flow rate, while it takes about 12min to elute with conventional columns. In addition, calculation results of molecular weight are comparable to conventional column.
- Repeatability was confirmed as the CV value of molecular weight measurement for continuous 10 injections was less than 2.0%, even at higher flow rate (2.0mL/min).
- Polymers other GPC such as polyvinylacetate, polysulfone, and phenoxy resin can be analyzed. Additionally, typical organic solvents for GPC are also available to HK-404L.