



TOSOH



SEPARATION REPORT NO. 101

TSKgel SuperHZ SERIES ULTRA-HIGHTHROUGHPUT GPC COLUMNS


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1. Introduction

Ever since its emergence, gel permeation chromatography (GPC) has been a widely accepted tool for polymer analysis and purification. Continuous improvements in GPC methodology, instrument systems, column design and packing material have resulted in superior precision and reproducibility. Recently, the GPC market has evolved to include applications that demand shortened analysis time and decreased solvent consumption. Tosoh Corporation was the first to introduce a group of ultra-high-speed organic solvent GPC columns, "TSKgel SuperH series," to address this need. Continued innovation by Tosoh has resulted in a more recent commercialization of an additional group of organic solvent semi-micro GPC columns, "TSKgel SuperHZ series". This article introduces the basic features as well as application data for the TSKgel SuperHZ series.

2. Features of TSK-GEL SuperHZ Series

The TSKgel SuperHZ series consists of Super HZ1000 – 4000. Each grade consists of a different pore size packing material. Subsequently, a unique separation range for each column results, allowing researchers to choose a column grade that is designed for the sample type being analyzed. Three mixed bed columns are also available. SuperHZM-N, M-M, and M-H are mixed-bed columns of various pore sizes designed to provide a linear calibration curve and simultaneously extend the range of measurable molecular weights (**Table-1**).

TSKgel Super HZ1000 – 4000 columns are capable of measuring monomers, polymer additives, oligomers and polymers up to a molecular weight of several hundred thousands with proper selection of pore size/column grade. Ultra fine particles (3µm) have been developed to provide high resolution over the entire molecular weight range. This is especially important for the separation of low molecular weight compounds.

Additionally, the mixed-bed columns (SuperHZM-N, M-M, and M-H) are capable of measuring oligomers and polymers with molecular weights up to tens of millions with proper selection of the grade. The various particle sizes of the mixed-bed packing materials have been optimized to ensure resolution in the low-molecular weight range while avoiding shear degradation of polymers in the high-molecular weight region. The features of SuperHZ series are shown in **Table-2**. Calibration curves of SuperHZ series are shown in **Figures-1** and **-2**

Table-1 List of TSKgel SuperHZ Series

Grade	Exclusion limit (polystyrene)	Particle size (µm)	Theoretical plates (TP/15cm)	Column size (mm I.D × cm)
TSKgel SuperHZ1000	1 × 10 ³	3	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZ2000	1 × 10 ⁴	3	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZ2500	2 × 10 ⁴	3	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZ3000	6 × 10 ⁴	3	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZ4000	4 × 10 ⁵	3	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZM-N	7 × 10 ⁵	3	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZM-M	4 × 10 ⁶	3&5	16,000	4.6 × 15
				6.0 × 15
TSKgel SuperHZM-H	4 × 10 ⁸ (Estimate)	10	9,000	4.6 × 15
				6.0 × 15

Table-2

Feature	Advantage
1) Ultra fine particles used in packing material	<ul style="list-style-type: none"> • Short measurement time is achieved. Resolution equivalent to conventional columns (30cm) can be obtained in 1/2 measurement time. • Resolution does not deteriorate even under a high flow rate.
2) Semi-micro column (4.6mmID and 6.0mmID)	<ul style="list-style-type: none"> • Reduction in solvent consumption (running costs, effluent processing costs) 1/6 to 1/3 solvent consumption compared to conventional columns.
3) Optimization of particle size in the packing material	<ul style="list-style-type: none"> • Shear degradation in polymers with high molecular weight can be prevented.
4) Adoption of low-absorption packing materials	<ul style="list-style-type: none"> • Applicable to wide range of samples.

3. Basic Properties

3-1. Resolution

As shown in **Table-1**, TSKgel SuperHZ1000 - SuperHZ4000 and TSKgel SuperHBM-N are packed with 3 μ m particles. The ultra-fine particles allow for high efficiency separations of low-molecular weight substances such as oligomers. These columns have theoretical plates values (per unit length) which are twice those of the conventional columns. As a result, equal resolution can be obtained with half the measurement time of the conventional products. An example is shown in **Figure-3**. To achieve the high theoretical plates, special attention should be paid to minimizing sources of dead volume which cause band-broadening. Optimization of the detector, sample injector, tubing in the system, etc. is recommended in combination with optimization of the measurement conditions. When using the 4.6mmID semi-micro column, the effect of band broadening becomes significant. For this reason, a micro flow cell (for UV detection) and a high performance GPC system able to resist the effects of moderate temperature change and deliver reproducible mobile phase volumes at low flow rates is recommended.

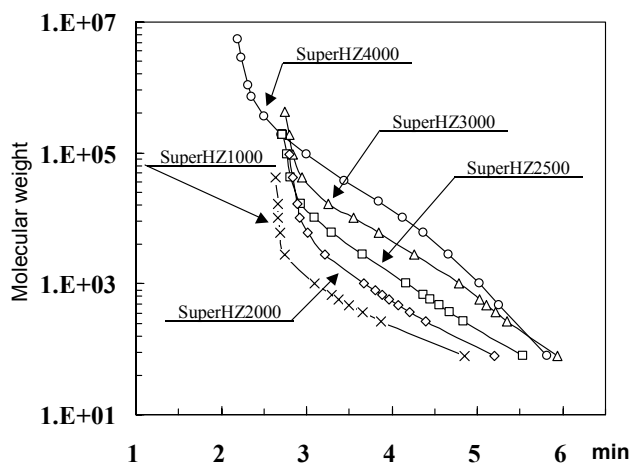


Figure-1 Calibration Curves of TSKgel SuperHZ Series

Column: TSKgel SuperHZ series
(4.6mm I.D. \times 15cm)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 25°C
Sample: standard polystyrene
Injection volume: 2 μ L

As shown in **Figure-2**, the advantage of TSKgel SuperHBM series (HZ mixed-bed series) is that a wide separation range of molecular weights with a linear calibration curve results. A mixed-bed column eliminates the need to use several columns in tandem for separation of sample compounds with a wide molecular weight range. Furthermore, similar to the SuperHZ series for oligomer measurement, TSKgel SuperHBM-N (molecular weight separation range 7×10^5 - 266), SuperHBM-M (molecular weight separation range 4×10^6 - 266), and SuperHBM-H (molecular weight separation range 1×10^8 - 1,000 (estimate)) are capable of measuring molecular weight and molecular weight distribution in 50% less measurement time than conventional products.

Figure-4 shows a comparison of polyisobutylene chromatograms by GMH_{XL}, which is a packing material with particle size of 9 μ m, and SuperHBM-H with particle size of 10 μ m.

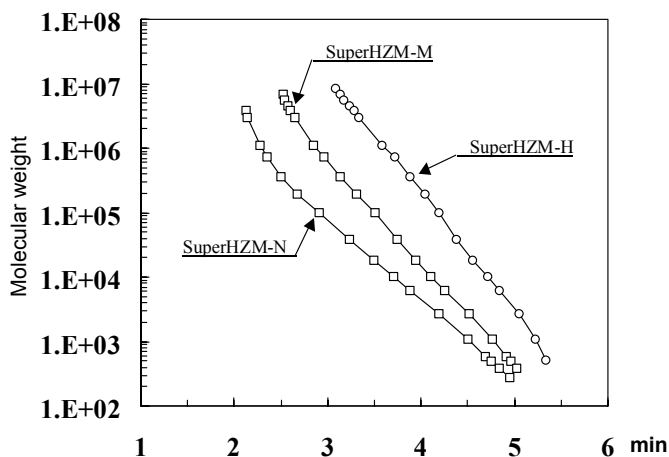


Figure-2 Calibration Curves of TSKgel SuperHBM Series

Column: TSKgel SuperHBM series
(4.6mm I.D. \times 15cm)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 25°C
Sample: standard polystyrene
Injection volume: 2 μ L

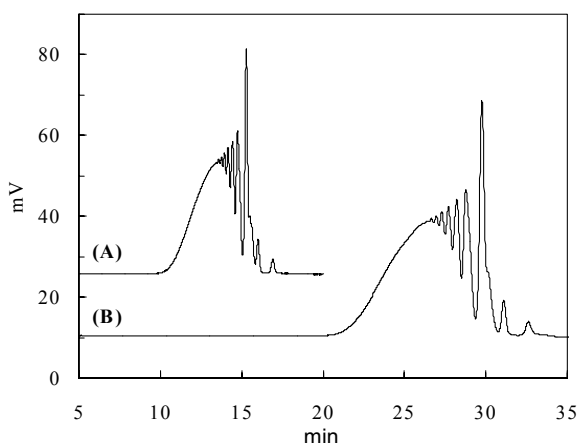


Figure-3 Comparison between SuperHZ and H_{XL}

Column: (A) TSKgel SuperHZ
(4000 + 3000 + 2500)
(4.6mm I.D. × 15cm × 3)
(B) TSKgel H_{XL}
(4000 + 3000 + 2500)
(7.8mm I.D. × 30cm × 3)

Eluent: THF
Flow rate: (A) 0.35mL/min
(B) 1.0mL/min
Temperature: 40°C
Detection: RI
Sample: phenolic resin
Injection volume: (A) 5μL, (B) 30μL

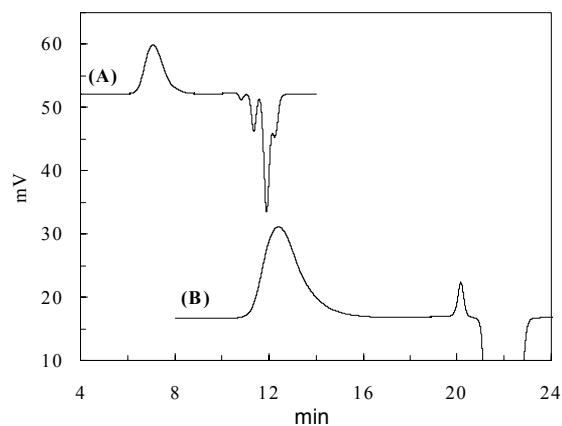


Figure-4 Comparison between SuperH_{2M}-H and GMH_{XL}

Column: (A) TSKgel SuperH_{2M}-H
(4.6mm I.D. × 15cm × 2)
(B) TSKgel GMH_{XL}
(7.8mm I.D. × 30cm × 2)

Eluent: THF
Flow rate: (A) 0.35mL/min
(B) 1.0mL/min
Temperature: 40°C
Detection: RI
Sample: phenolic resin
Injection volume: (A) 10μL, (B) 100μL

3-2. Dependence of Height Equivalent to a Theoretical Plate (HETP) on Flow Rate

Figure-5 shows the results of comparing the flow rate dependence of HETP in TSKgel G2500H_{XL} (particle size 5μm) and TSKgel SuperHZ2500 (particle size 3μm) using a low-molecular-weight sample compound. While HETP gradually increases for the larger particle size TSKgel G2500H_{XL} column, there is little change in HETP at high flow rates with TSKgel Super HZ2500. HETP flow rate dependency of SuperHZ2500, SuperH_{2M}-N, SuperH_{2M}-M, and SuperH_{2M}-H (inner diameter 4.6mm and 6.0mm) columns are shown in Figures-6, -7, -8, and -9, respectively.

In actual measurement, HETP flow rate dependency will also depend on the sample's molecular size (molecular weight), eluent type (viscosity) and measurement temperature. As the flow rate increases, the loss in efficiency becomes more significant as the molecular weight increases. Therefore, a low flow rate is recommended when measuring a high molecular weight polymer.

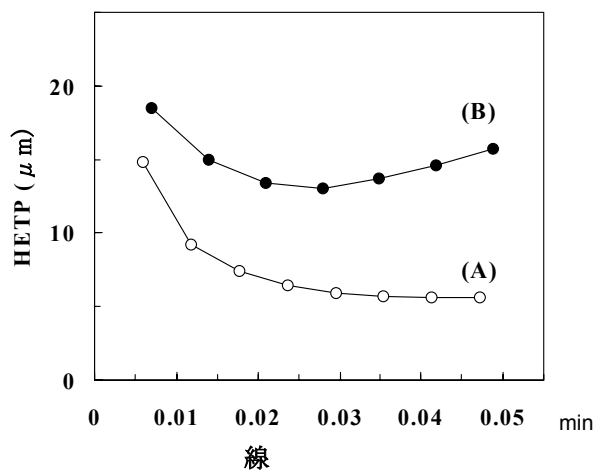


Figure-5 Comparison of the Effect of Linear Velocity on HETP in SuperHZ2500 and G2500H_{XL}

Column: (A) TSKgel SuperHZ2500
(6.0mm I.D. × 15cm)
(B) TSKgel G2500H_{XL}
(7.8mm I.D. × 30cm)

Eluent: THF
Temperature: 25°C
Sample: (A) dicyclohexyl phthalate (DCHP),
(B) benzene
Injection volume: (A) 2μL
(B) 20μL

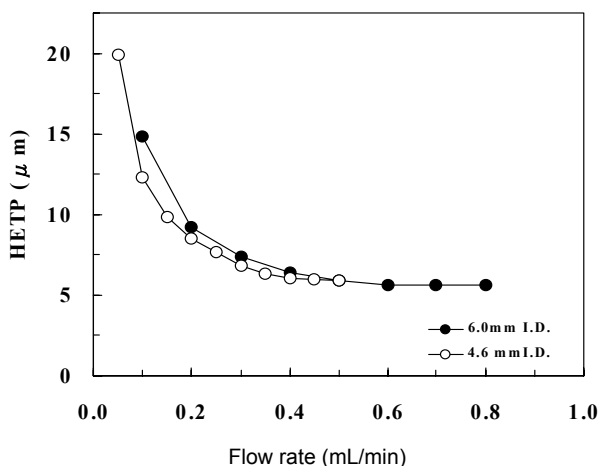


Figure-6 Relationship between HETP and Flow Rate in SuperHZ2500

Column: TSKgel SuperHZ2500
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Temperature: 25°C
 Sample: DCHP
 Injection volume: 2μL (6.0mm I.D. × 15cm)
 1μL (4.6mm I.D. × 15cm)

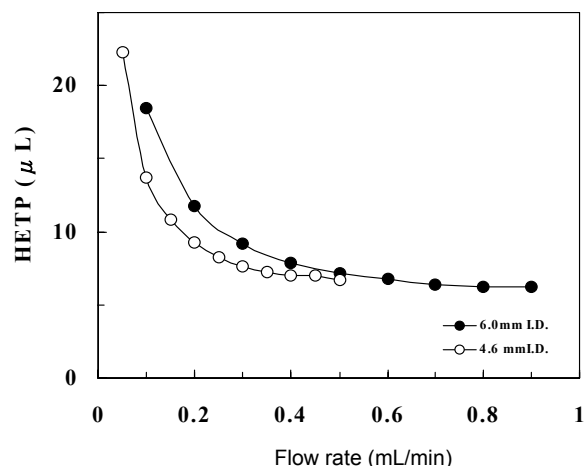


Figure-7 Relationship between HETP and Flow Rate in SuperH2M-N

Column: TSKgel SuperH2M-N
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Temperature: 25°C
 Sample: DCHP
 Injection volume: 2μL (6.0mm I.D. × 15cm)
 1μL (4.6mm I.D. × 15cm)

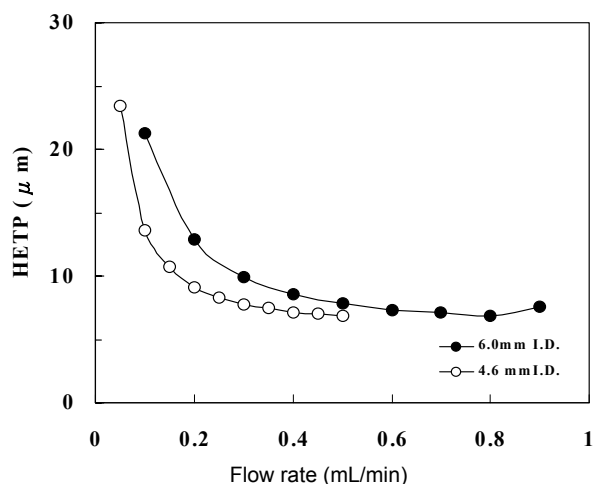


Figure-8 Relationship between HETP and Flow Rate in SuperH2M-M

Column: TSKgel SuperH2M-M
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Temperature: 25°C
 Sample: DCHP
 Injection volume: 2μL (6.0mm I.D. × 15cm)
 1μL (4.6mm I.D. × 15cm)

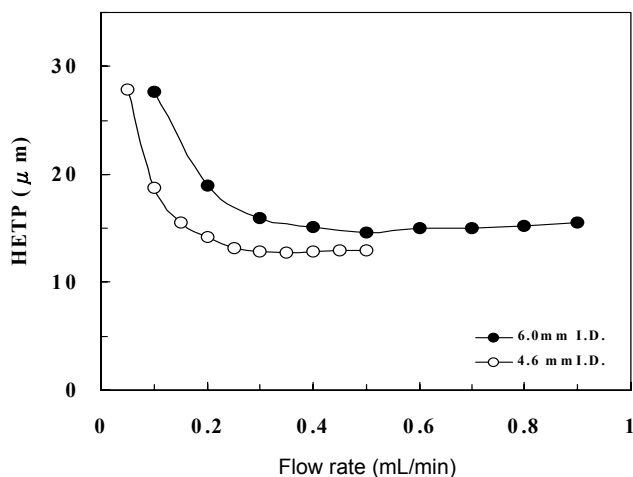


Figure-9 Relationship between HETP and Flow Rate in SuperH2M-H

Column: TSKgel SuperH2M-H
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Temperature: 25°C
 Sample: DCHP
 Injection volume: 2μL (6.0mm I.D. × 15cm)
 1μL (4.6mm I.D. × 15cm)

3-3. Effect of Sample Injection Volume

As mentioned earlier, TSKgel SuperHZ series are packed with ultra fine particles. In combination with the narrower bore of the TSKgel SuperHZ, sample injection volume becomes a critical factor in maintaining column performance. As shown in **Figure-10**, the HETP value for TSKgel Super HZ2500 grows larger and the column performance deteriorates as the sample injection volume increases. HETP deterioration due to an increase in the sample injection volume is more pronounced with high-performance columns containing small particle sizes. As shown in **Figures-10, -11, -12, and -13**, the maximum sample injection volume of each column is 2 μ L (inner diameter 4.6mm I.D.) and 4 μ L (inner diameter 6.0mm I.D.) for Super HZ2500 and SuperHZN for oligomer measurement. For synthetic polymer measurements, 2 μ L (inner diameter 4.6mm I.D.) and 5 μ L (inner diameter 6.0mm I.D.) injection volumes are

recommended for Super HZM-M. Lastly, synthetic polymer measurements with TSKgel Super HZM-H should use injection volumes of 5 μ L (inner diameter 4.6mm I.D.) and 10 μ L (inner diameter 6.0mm I.D.) to avoid loss in performance.

In measurement of polymers including oligomers in which high resolution is most important, it is necessary that the measurement is taken with high sample concentration so that the sample injection volume is less than the maximum volume. On the other hand, for synthetic polymers that do not contain oligomers and have relatively large molecular weights, measurements should be taken with low concentration and large injection volume because the dependency of measured molecular weight on the sample injection volume is small. In this case, sample injection volume can be optimized with consideration of overloading, detection sensitivity, etc.

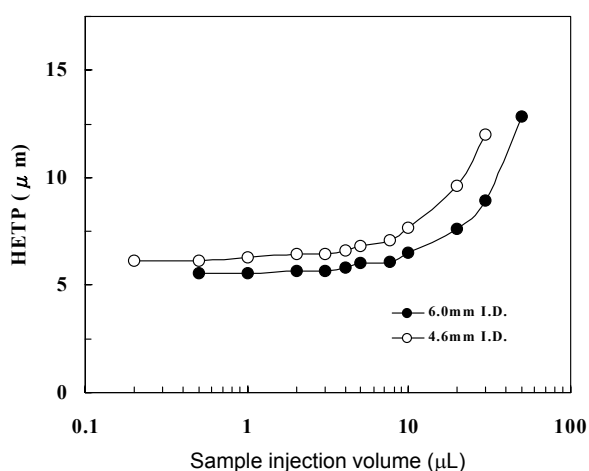


Figure-10 Relationship between HETP and Sample Injection Volume in SuperHZ2500

Column: TSKgel SuperHZ2500
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Flow rate: 0.6mL/min (6.0mm I.D. × 15cm)
 0.35mL/min (4.6mm I.D. × 15cm)
 Temperature: 25°C
 Sample: DCHP

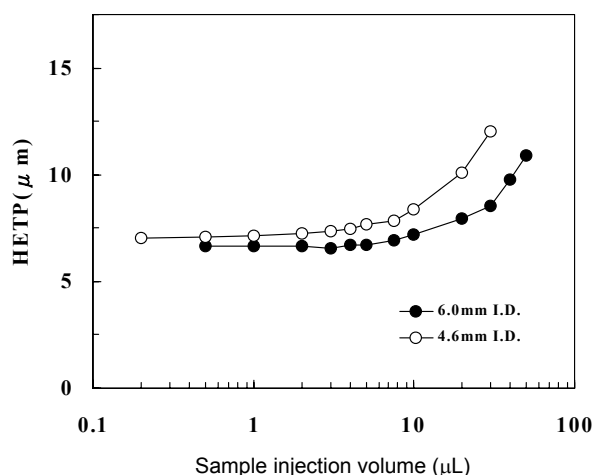


Figure-11 Relationship between HETP and Sample Injection Volume in SuperHZN

Column: TSKgel SuperHZN-N
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Flow rate: 0.6mL/min (6.0mm I.D. × 15cm)
 0.35mL/min (4.6mm I.D. × 15cm)
 Temperature: 25°C
 Sample: DCHP

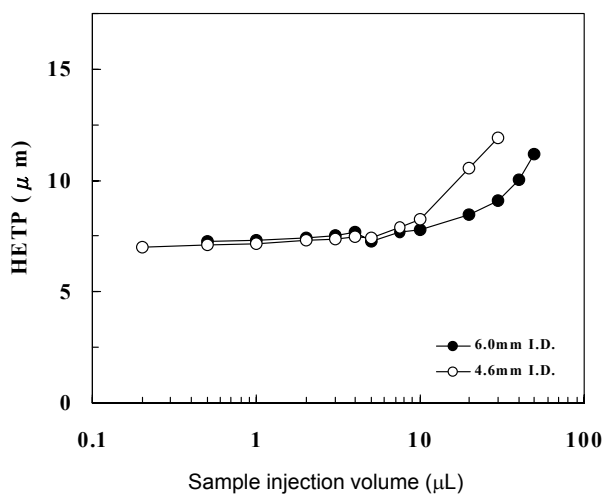


Figure-12 Relationship between HETP and Sample Injection Volume in SuperHZM-M

Column: TSKgel SuperHZM-M
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Flow rate: 0.6mL/min (6.0mm I.D. × 15cm)
 0.35mL/min (4.6mm I.D. × 15cm)
 Temperature: 25°C
 Sample: DCHP

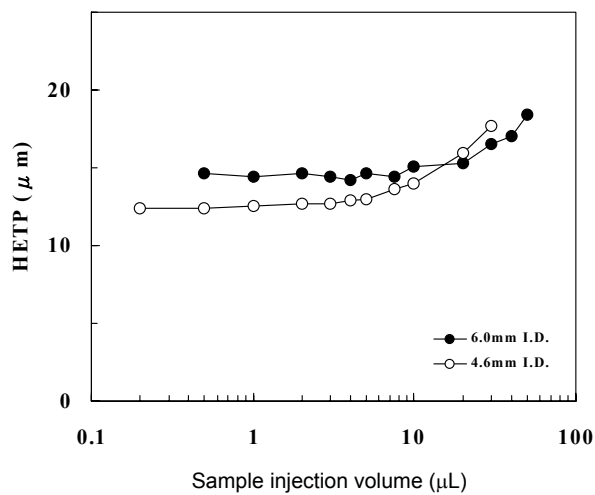


Figure-13 Relationship between HETP and Sample Injection Volume in SuperHZM-H

Column: TSKgel SuperHZM-H
 (6.0mm I.D. × 15cm)
 (4.6mm I.D. × 15cm)
 Eluent: THF
 Flow rate: 0.6mL/min (6.0mm I.D. × 15cm)
 0.35mL/min (4.6mm I.D. × 15cm)
 Temperature: 25°C
 Sample: DCHP

3-4. Effect of Sample Concentration

As sample concentration increases, the apparent sample molecular weight becomes smaller and elution times increase. This phenomenon (as shown in **Figure-14**) is similar with regard to particle size as the effect of increasing sample injection volume. It becomes more significant as particle size is decreased. As a result, extra care is needed to prevent sample overload. As shown in **Figure-15**, if sample overload occurs, the calibration curve shifts resulting in erroneous, elevated molecular weight determinations. To ensure accurate molecular weight determinations, it is best that both the sample measurements and the standard measurements occur at low concentrations to prevent an overloading phenomenon.

The relationship between the standard polystyrene sample concentration and elution time in TSKgel SuperHZ mix grade (4.6mm I.D.) is shown in **Figures-16, -17 and -18**. When actual polymers are measured, optimal sample concentration varies depending on the molecular weight distribution and the column to be used in the molecular weight measurement. However, when epoxy resin is measured using TSKgel Super HZM-N (4.6mm I.D.) as shown in **Figure-19**, little change is seen

in various average molecular weights and resolutions as long as the sample concentration stays below 20g/L (load 100 μ g). This relatively high sample concentration is due to epoxy's small molecular weight. **Figure-20** shows the chromatograms of epoxy resin under different concentrations. With concentration of 100g/L (load 500 μ g), slowing in elution position due to overloading is seen.

Figures-21, and -22 show the relationship between the sample concentration and molecular weight. Measurement should be performed with a maximum load of 20 μ g when polystyrene SRM706 (Mw257000) is used as the measured sample with TSKgel Super HZM-M (4.6mm I.D.). Deterioration of the molecular weight value due to overloading is evident at sample concentration of 2g/L (load 20 μ g) or higher. On the other hand, when polyisobutylene is measured with TSKgel Super HZM-H (4.6mm I.D.), deterioration in the molecular weight value occurs at sample concentration's of 1g/L (load 10 μ g) because the molecular weight of the sample is large. Thus, it is necessary that measurements be taken under a sufficiently low concentration when a sample with large molecular weight is measured.

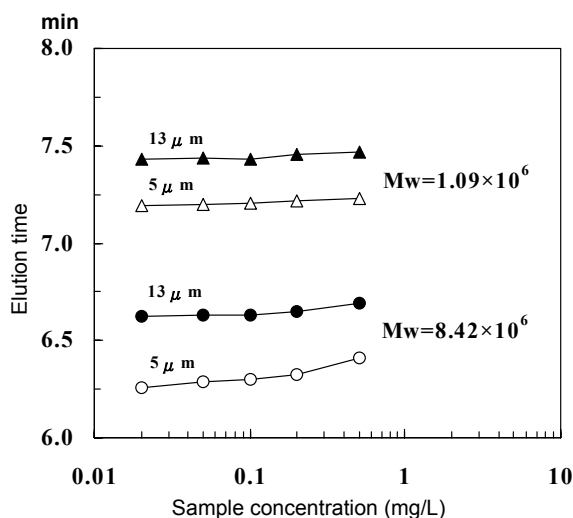


Figure-14 Effect of Sample Concentration on Elution Time

Column: TSKgel SuperHZM-H \times 2
(4.6mm I.D. \times 15cm \times 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C
Detection: RI
Sample: standard polystyrene
Injection volume: 10 μ L

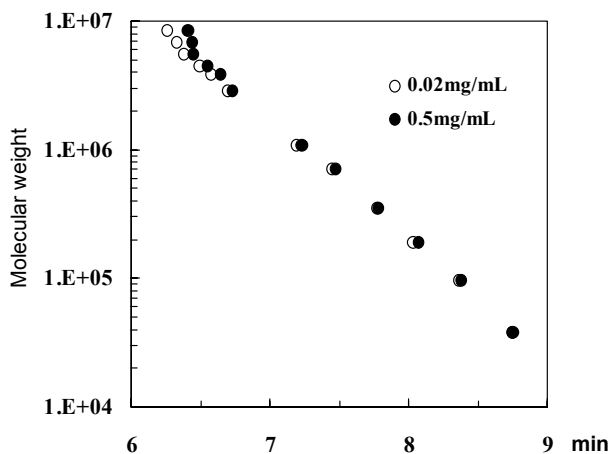


Figure-15 Effect of Sample Concentration on Elution Time (Calibration Curve)

Column: TSKgel SuperHZM-H \times 2
(4.6mm I.D. \times 15cm \times 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C
Detection: RI
Sample: standard polystyrene
Injection volume: 10 μ L

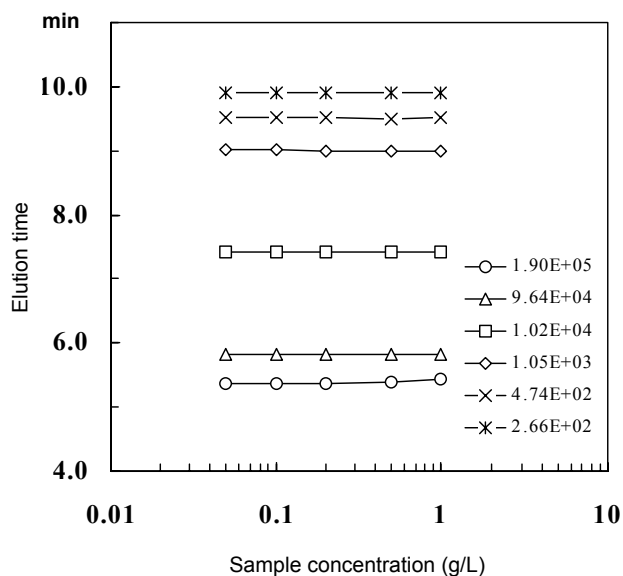
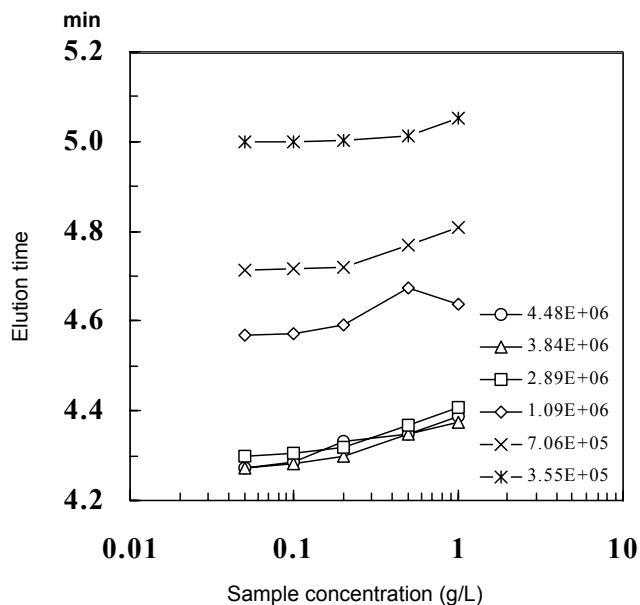


Figure-16 Effect of Sample Concentration on Elution Time

Column: TSKgel SuperHZM-N × 2
 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C
 Detection: RI
 Sample: standard polystyrene
 Injection volume: 5µL

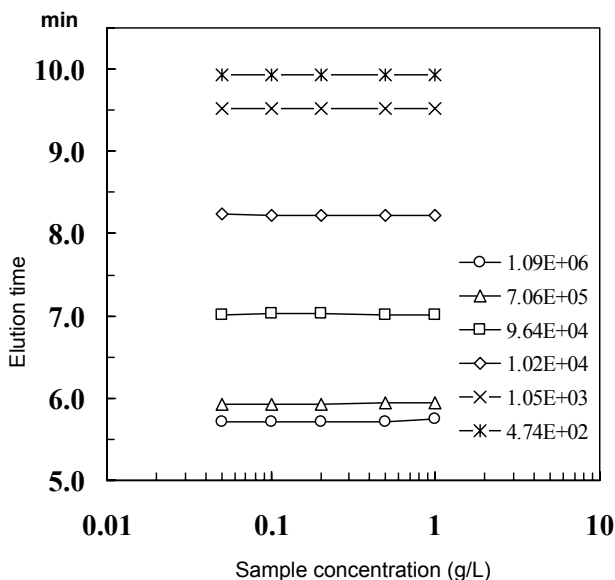
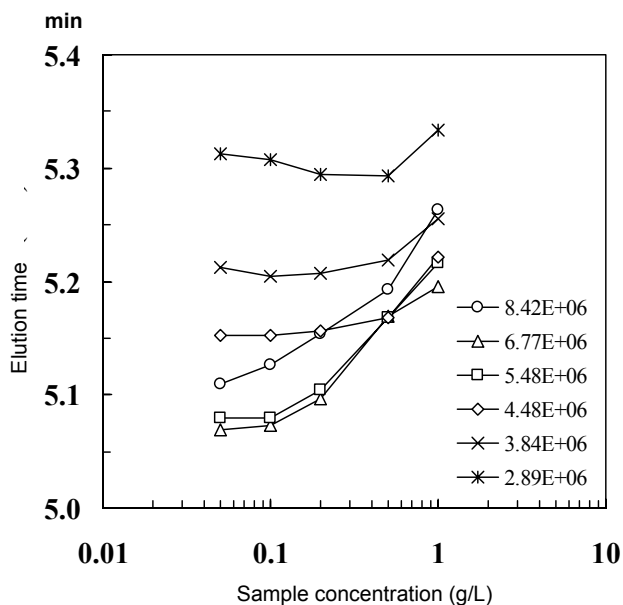


Figure-17 Effect of Sample Concentration on Elution Time

Column: TSKgel SuperHZM-M × 2
 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C
 Detection: RI
 Sample: standard polystyrene
 Injection volume: 10µL

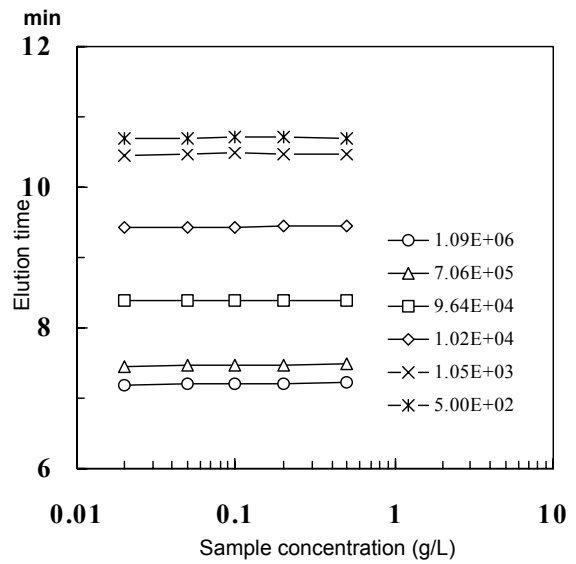
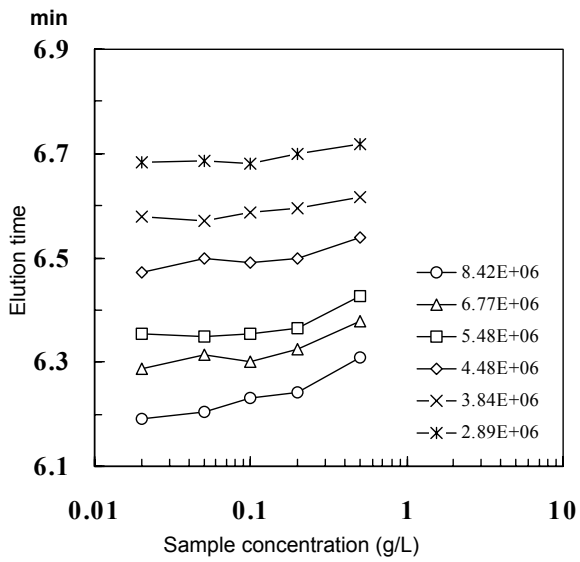


Figure-18 Effect of Sample Concentration on Elution Time

Column: TSKgel SuperH2M-H × 2
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C
Detection: RI
Sample: standard polystyrene
Injection volume: 5µL

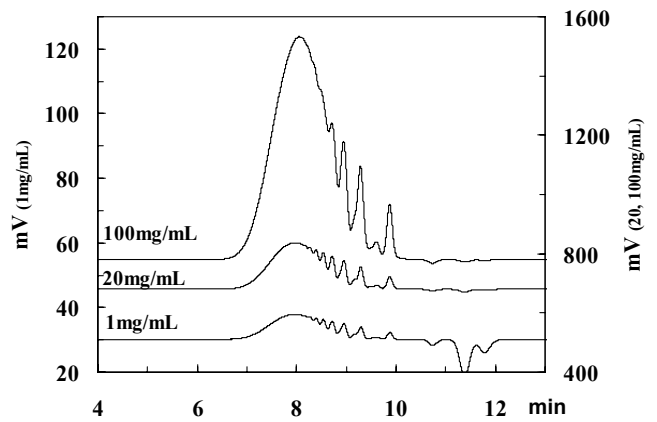
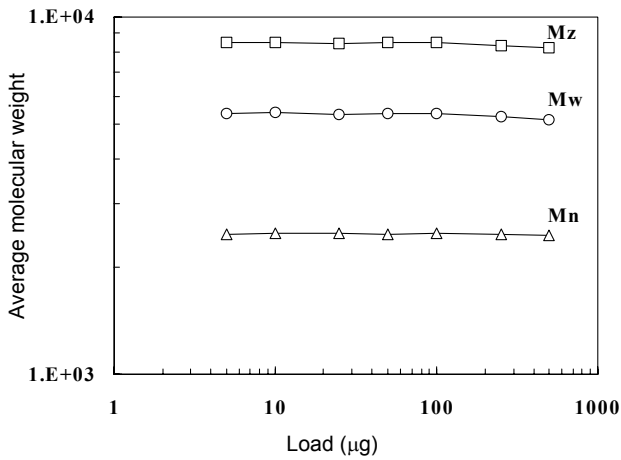


Figure-19 Effect of Sample Injection Volume on Average Molecular Weight

Column: TSKgel SuperH2M-N
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C
Detection: RI
Sample: epoxy resin
Injection volume: 5µL

Figure-20 Effect of Sample Load on Chromatogram

Column: TSKgel SuperH2M-N
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C
Detection: RI
Sample: epoxy resin
Injection volume: 5µL

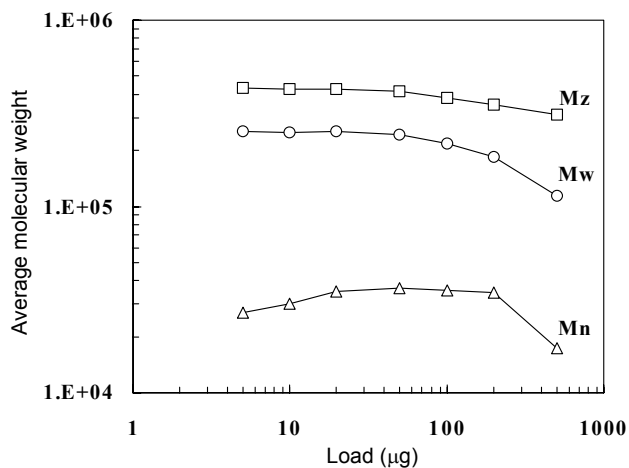


Figure-21 Effect of Sample Injection Volume on Average Molecular Weight

Column: TSKgel SuperH2M-M
 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C
 Detection: RI
 Sample: polystyrene SRM706
 Injection volume: 10µL

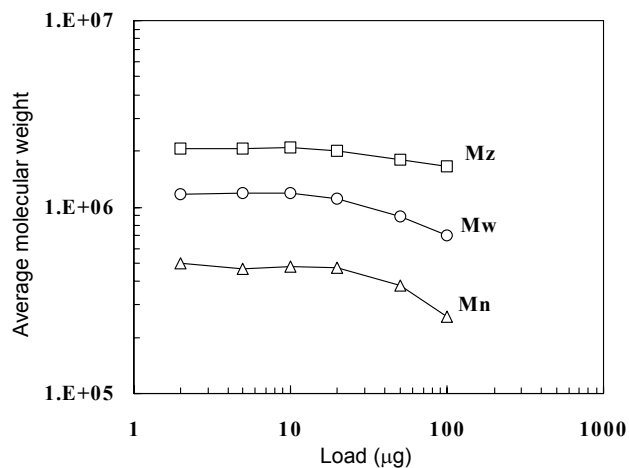


Figure-22 Effect of Sample Injection Volume on Average Molecular Weight

Column: TSKgel SuperH2M-H
 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C
 Detection: RI
 Sample: polyisobutylene
 Injection volume: 10µL

3-5. Optimization of Packing Material Particle Size

When measurement is taken using a column packed with fine-particle packing materials or under a high flow rate, it becomes more likely that chain breaking in polymers will occur. Therefore, in TSKgel SuperHZ series, an optimal particle size that corresponds to the range of molecular weight fractionation is adopted in each of the columns designed for polymer analysis (TSKgel SuperH2M-M and SuperH2M-H). **Figure-23** shows the calibration curve obtained by measuring polystyrene standards using packing materials of different particle sizes and different measurement flow rates. When a packing material with a particle size of 3 μ m is used for samples with molecular weight of 1 million or higher, delay in elution due to breaking in branched chains becomes prevalent at 0.35mL/min. Furthermore, delay in elution can also be seen for samples with molecular weight of several hundred thousands or larger. This phenomenon appears more drastically as the particle size

becomes smaller and the measurement flow rate becomes higher. On the other hand, when a packing material with particle size of 13 μ m is used, delay in sample elution is not seen throughout the molecular weight range under any flow rate. Based on this fact, SuperH2M-H, which targets measurement of average molecular weight of several millions to several hundreds of thousands, has been packed with a 10 μ m packing material and SuperH2M-M, which targets measurement of average molecular weight of several hundred thousands to several tens of thousands, has been packed with 3 μ m or 5 μ m packing material. In addition, a packing material with a particle size of 3 μ m has been used for SuperH2M-N, in which the focus is to target samples with average molecular weights of several tens of thousands such as oligomers. By varying the particle sizes in the TSKgel Super H2M series, researchers are able to choose an optimal column for their sample type.

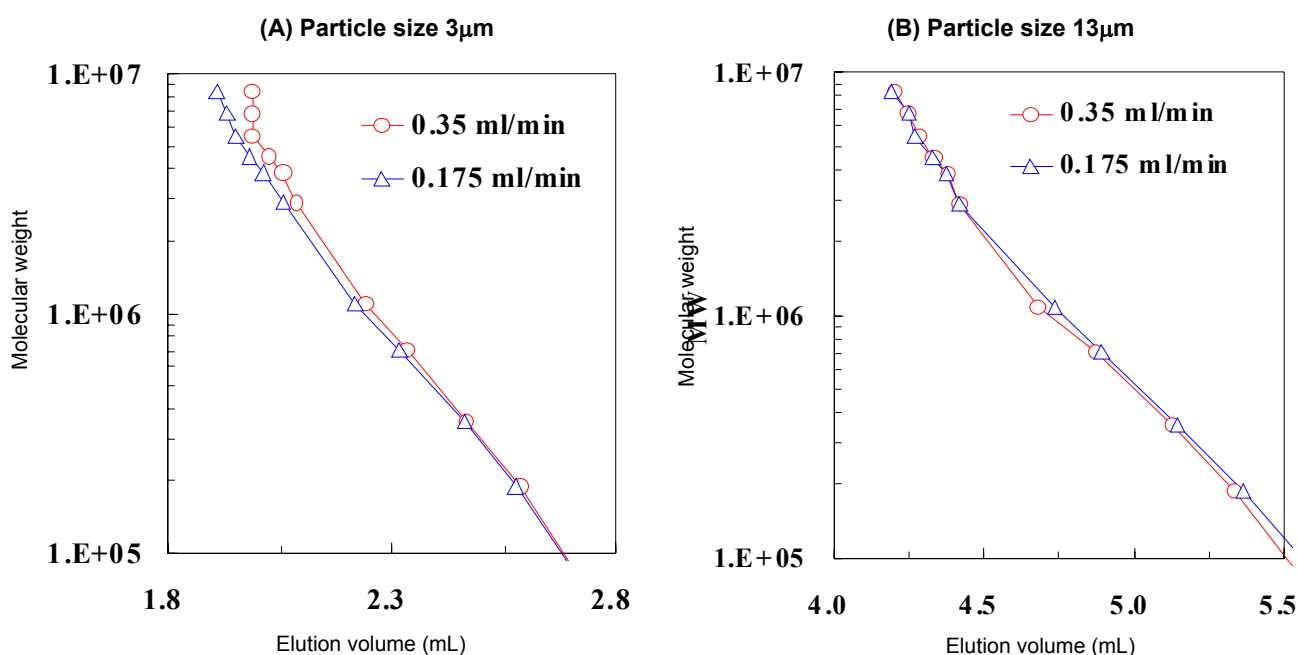


Figure-23 Effect of Measurement Flow Rate and Particle Size on Calibration Curve

Column: styrene-divinylbenzene copolymerization gel
 (A): (4.6mm I.D. \times 15cm)
 (B): (4.6mm I.D. \times 30cm)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C
 Detection: RI
 Sample: standard polystyrene
 Injection volume: (A) 5 μ L, (B)10 μ L

4. Application

Figures-24 to -35 show examples of analysis using various samples.

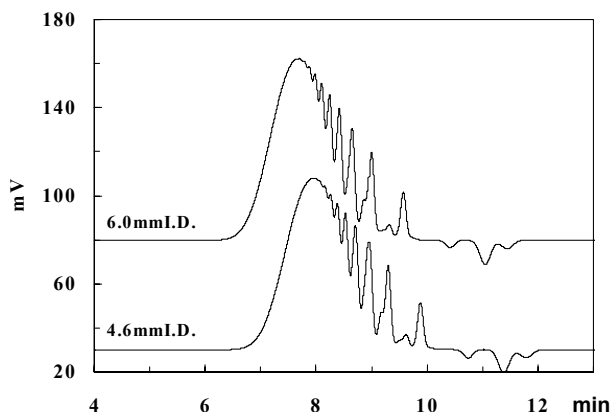


Figure-24 Chromatogram of Epoxy Resin

Column: TSKgel SuperHZM-N × 2
 Eluent: THF
 Flow rate: 0.35mL/min (4.6mm I.D.)
 0.6mL/min (6.0mm I.D.)
 Temperature: 40°C Detection: RI
 Sample: epoxy resin (10g/L)
 Injection volume: 5µL (4.6mm I.D.)
 9µL (6.0mm I.D.)

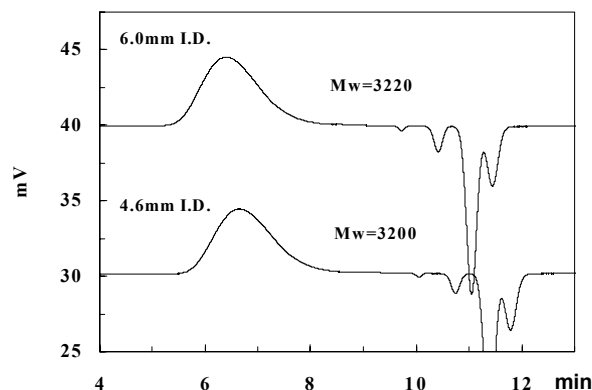


Figure-25 Chromatogram of Polymethyl Methacrylate

Column: TSKgel SuperHZM-N × 2
 Eluent: THF
 Flow rate: 0.35mL/min (4.6mm I.D.)
 0.6mL/min (6mm I.D.)
 Temperature: 40°C Detection: RI
 Sample: polymethyl methacrylate (1g/L)
 Injection volume: 5µL (4.6mm I.D.)
 9µL (6.0mm I.D.)

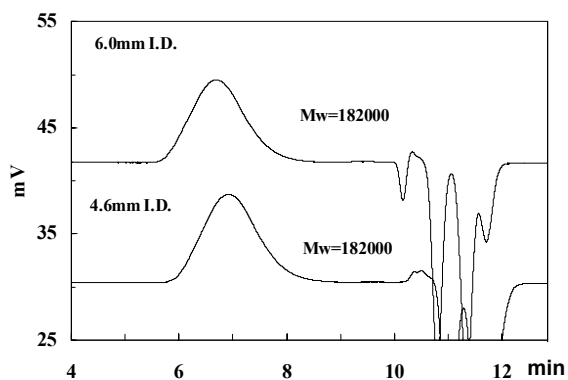


Figure-26 Chromatogram of Butylmethacrylate-Isobutylmethacrylate Copolymer

Column: TSKgel SuperHZM-M × 2
 Eluent: THF
 Flow rate: 0.35mL/min (4.6mm I.D.)
 0.6mL/min (6mm I.D.)
 Temperature: 40°C Detection: RI
 Sample: butylmethacrylate-isobutylmethacrylate copolymer (1g/L)
 Injection volume: 10µL (4.6mm I.D.)
 17µL (6.0mm I.D.)

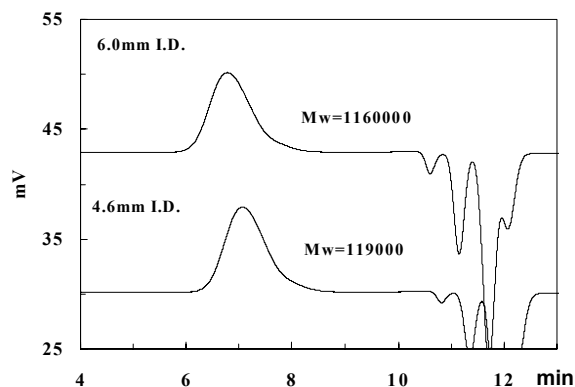


Figure-27 Chromatogram of Polyisobutylene

Column: TSKgel SuperHZM-M × 2
 Eluent: THF
 Flow rate: 0.35mL/min (4.6mm I.D.)
 0.6mL/min (6.0mm I.D.)
 Temperature: 40°C Detection: RI
 Sample: polyisobutylene (0.5g/L)
 Injection volume: 10µL (4.6mm I.D.)
 17µL (6.0mm I.D.)

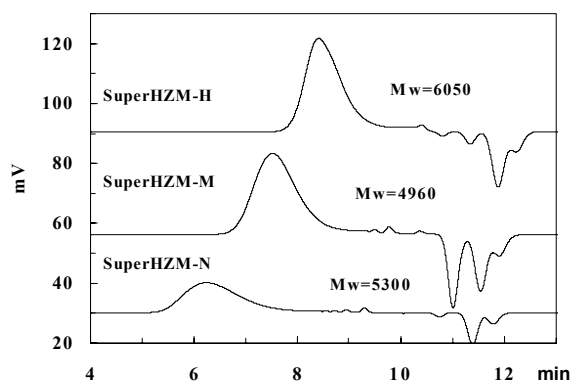


Figure-28 Chromatogram of Polysulfone
 Column: TSKgel SuperHZM × 2 (4.6mm I.D.)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C Detection: RI
 Sample: polysulfone (1g/L)
 Injection volume: 5µL (SuperHZM-N),
 10µL (SuperHZM-M, -H)

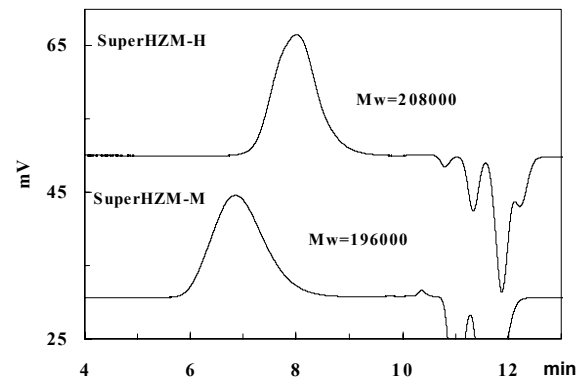


Figure-29 Chromatogram of 1, 2-Polybutadiene
 Column: TSKgel SuperHZM × 2 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C Detection: RI
 Sample: polybutadiene (1.0g/L)
 Injection volume: 10µL

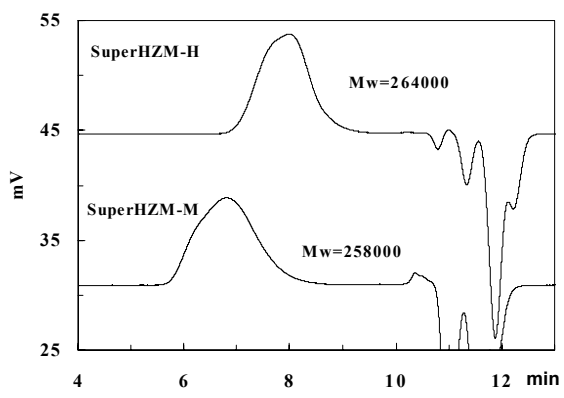


Figure-30 Chromatogram of Polyethylmethacrylate
 Column: TSKgel SuperHZM × 2 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C Detection: RI
 Sample: polyethylmethacrylate (1.0g/L)
 Injection volume: 10µL

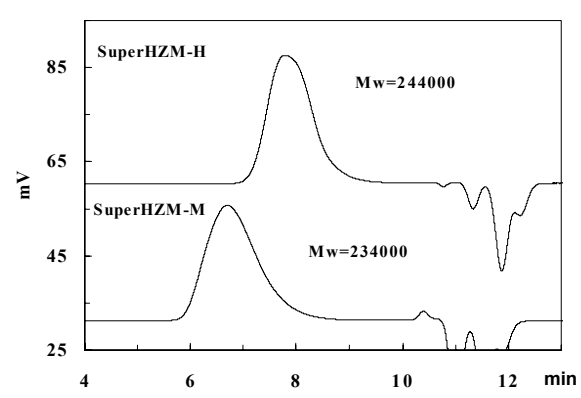


Figure-31 Chromatogram of Polystyrene
 Column: TSKgel SuperHZM × 2 (4.6mm I.D. × 15cm × 2)
 Eluent: THF
 Flow rate: 0.35mL/min
 Temperature: 40°C Detection: RI
 Sample: polystyrene (1.0g/L)
 Injection volume: 10µL

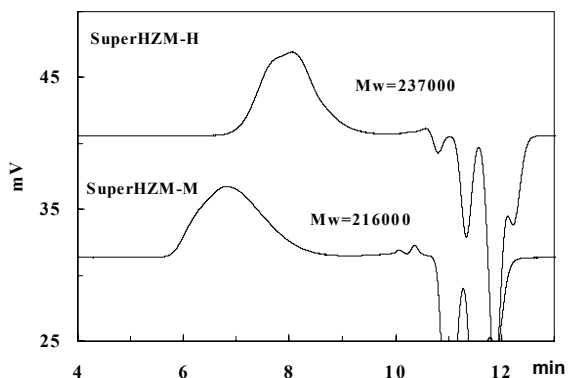


Figure-32 Chromatogram of Polyvinyl Acetate

Column: TSKgel SuperH2M × 2
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C Detection: RI
Sample: polyvinyl acetate (1.0g/L)
Injection volume: 10µL

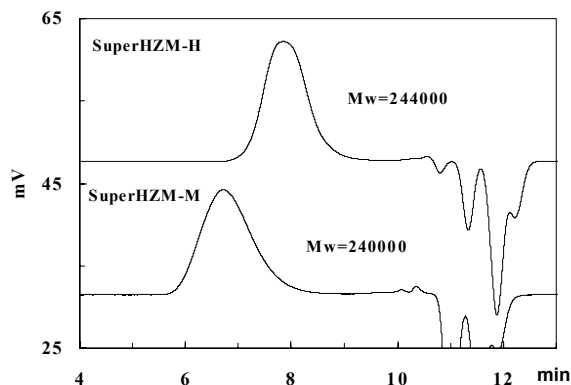


Figure-33 Chromatogram of Polyvinyl Chloride

Column: TSKgel SuperH2M × 2
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C Detection: RI
Sample: polyvinyl chloride (1.0g/L)
Injection volume: 10µL

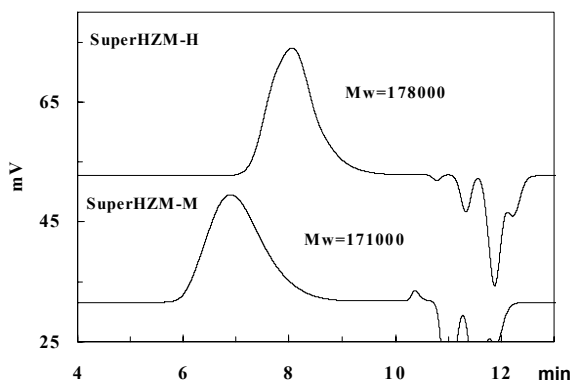


Figure-34 Chromatogram of Styrene-Acrylonitrile Copolymer

Column: TSKgel SuperH2M × 2
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C Detection: RI
Sample: styrene-acrylonitrile copolymer (1.0g/L)
Injection volume: 10µL

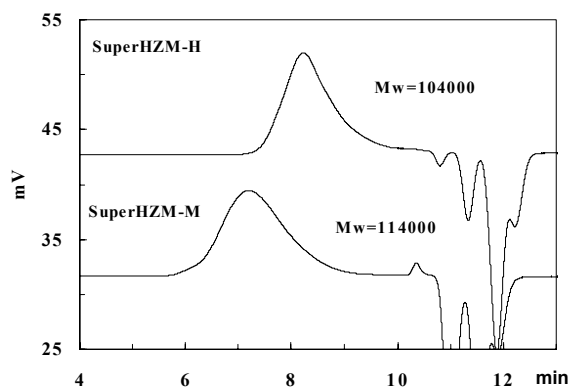


Figure-35 Chromatogram of Vinyl Alcohol-Vinyl Butyral Copolymer

Column: TSKgel SuperH2M × 2
(4.6mm I.D. × 15cm × 2)
Eluent: THF
Flow rate: 0.35mL/min
Temperature: 40°C Detection: RI
Sample: vinyl alcohol-vinyl butyral copolymer (1.0g/L)
Injection volume: 10µL

5. Conclusion

By optimizing the particle size of the packing material for each molecular weight measurement range, TSKgel SuperH2 series is capable of reducing the analysis time in half when compared to conventional columns. TSKgel SuperH2 columns also provide high resolution in low-molecular weight range (especially useful for oligomer analysis) and eliminate chain breaking of polymers in the high-molecular weight range. As described earlier, the superior efficiency of the ultra fine particles of TSKgel SuperH2, can be quickly

deteriorated by not reducing sources that contribute to band broadening. It is important that the system be optimized system under the optimal measurement conditions. While columns with inner diameters 4.6mm and 6.0mm are available, we recommend that measurements be taken with a high-speed GPC system which is not influenced by temperature changes of a minimum of 25 °C and excels in liquid feeding repeatability, when 4.6mmID columns are used in conjunction with a low flow rate.