



About: TSKgel H Series Size Exclusion Columns

TSKgel H series columns are recommended for the analysis of organic-soluble polymers and are packed with spherical particles composed of polystyrene crosslinked with divinylbenzene (PS-DVB). This series includes TSKgel H_{XL}, H_{HR}, SuperH, Super HZ, and SuperMultiporeHZ columns. Each line of columns within this series differs in degree of inertness and operating temperature range.

The Super prefix designates short (15 cm) columns packed with particles as small as 3 µm. The smaller particle allows for equivalent resolution to conventional TSKgel H_{XL} columns, with 50% reduction in analysis time due to the shorter column length. The TSKgel Super series columns are an excellent choice for high throughput polymer analysis.

- The TSKgel H_{XL} columns are conventional GPC columns of 7.8 mm ID × 30 cm. The column line consists of eight columns with different pore sizes, TSKgel G1000H_{XL} through TSKgel G7000H_{XL}, and three columns with an extended linear range of the calibration curve, TSKgel GMH_{XL}, TSKgel GMH_{XL}-L and TSKgel MultiporeH_{XL}-M. The 5 µm particles in the TSKgel MultiporeH_{XL}-M column contain a broad range of pore sizes. This innovative approach essentially creates a linear calibration curve within each particle. As a result, columns with an extended linear calibration curve can now be prepared without mixing particles of different pore sizes.

The main characteristics of TSKgel H_{XL} columns are: ultra-low sample adsorption, i.e., the columns show true size exclusion behavior for most polymers, limited solvent range, and a maximum operating temperature of 60 °C for TSKgel G1000H_{XL} - G3000H_{XL}, and 80 °C for the remaining columns in the TSKgel H_{XL} column line.

- The TSKgel H_{HR} column line consists of eight conventional GPC columns of 7.8 mm ID × 30 cm with different pore sizes, TSKgel G1000H_{HR} through TSKgel G7000H_{HR}, and seven mixed bed columns, in which particles with different pore sizes are blended to provide an extended linear calibration curve. The mixed bed columns feature increasing linear calibration ranges, from TSKgel GMH_{HR}-L, GMH_{HR}-N, GMH_{HR}-M, to GMH_{HR}-H. The main characteristic of these TSKgel H_{HR} columns is a broad solvent range.

In addition, four TSKgel H_{HR} mixed bed columns are available for ultra-high temperature analysis. The maximum operating temperature of these columns is 220 °C.

- The TSKgel SuperH column line consists of eight columns of 6.0 mm ID × 15 cm with different pore sizes, TSKgel SuperH1000 through TSKgel SuperH7000, and four mixed bed columns with an extended linear range of the calibration curve. The mixed bed columns feature increasing linear calibration ranges, from TSKgel SuperHM-L, SuperHM-N, SuperHM-M, to SuperHM-H. TSKgel SuperH columns are high efficiency/high throughput versions of the conventional TSKgel H_{HR} columns. Both column types are based on the same bead chemistry.

The main characteristics of TSKgel SuperH columns are: a maximum operating temperature of 140 °C and the ability to use a broad range of solvents.

- The TSKgel SuperHZ column line consists of five columns of 4.6 mm ID × 15 cm and 6.0 mm ID × 15 cm with different pore sizes, TSKgel SuperHZ1000 through TSKgel SuperHZ4000, and three columns with an extended linear range of the calibration curve. The mixed bed columns feature increasing linear calibration ranges, from TSKgel SuperHZM-L, SuperHZM-N to SuperHZM-H.

The main characteristics of TSKgel SuperHZ columns are: developed for high throughput, high efficiency GPC applications such as those encountered in combinatorial chemistry experiments, ultra-low sample adsorption, limited solvent range, and a maximum operating temperature of 60 °C for TSKgel SuperHZ1000 - SuperHZ3000 and 80 °C for the remaining columns in the TSKgel SuperHZ line.

- The TSKgel SuperMultiporeHZ column line consists of three columns of 4.6 mm ID × 15 cm with particles sizes of 3, 4 and 6 µm. The particles in TSKgel SuperMultiporeHZ columns are monodisperse in size and exhibit a broad range of pore sizes. Each particle, by design, has an extended linear calibration curve, thereby greatly diminishing chromatograms with inflection points.

A comparison of TSKgel H series columns is detailed in [Table 20](#). The cross-linking of the polystyrene particles in TSKgel H series columns ensures minimal shrinking and swelling of the column bed when the organic solvent is changed according to the solvent recommendations outlined in [Table 21](#). Suggested flow rates for solvent exchange in TSKgel SuperH and H_{HR} columns are outlined in [Table 22](#). [Table 23](#) lists the recommended solvents by application for TSKgel H series columns.

Table 20: Comparison of TSKgel H series columns

TSKgel series	SuperMultiporeHZ	SuperHZ	SuperH	H _{XL}	H _{HR}
Application focus	Ultra-high performance with a low dead volume and a wide pore distribution in each particle for superior linearity	High throughput polymer analysis with ultra-low polymer adsorption, limited solvent compatibility range	High throughput polymer analysis with expanded solvent compatibility range	Conventional polymer analysis with ultra-low polymer adsorption, limited solvent compatibility range	Conventional polymer analysis with expanded solvent compatibility range
Particle size	3 µm, 4 µm, and 6 µm, depending on pore size	3 µm, 5 µm, and 10 µm, depending on pore size	3 µm and 5 µm, depending on pore size	5 µm, 6 µm, 9 µm, and 13 µm, depending on pore size	5 µm, 13 µm, 20 µm, and 30 µm
Particle matrix	Polystyrene divinylbenzene (PS-DVB)	Polystyrene divinylbenzene (PS-DVB)	Polystyrene divinylbenzene (PS-DVB)	Polystyrene divinylbenzene (PS-DVB)	Polystyrene divinylbenzene (PS-DVB)
Number of solvent substitutions	None	One time only	Several ¹	One time only	Several ¹

¹ After switching to a very polar solvent such as acetone, switching back to a nonpolar solvent is not recommended.

Table 21: Solvent compatibility for TSKgel H series columns

TSKgel series	Shipping solvent*	Can be replaced with:
SuperHZ ¹	Tetrahydrofuran ^{3,4}	benzene, chloroform, toluene, xylene, dichloromethane, dichloroethane
	Dimethylformamide*, cyclohexane*	
SuperHZ and H _{XL} ¹	Tetrahydrofuran ^{3,4}	benzene, chloroform, toluene, xylene, dichloromethane, dichloroethane
	Acetone*	carbon tetrachloride ⁵ , <i>o</i> -dichlorobenzene, dimethylformamide, dimethyl sulfoxide, 1,4-dioxane, ethylacetate, FC-113, hexafluoroisopropanol/chloroform, methyl ethyl ketone, quinoline, cyclohexane, N-methylpyrrolidone
	Chloroform*	<i>m</i> -cresol/chloroform, up to 10% hexafluoroisopropanol/chloroform
	Dimethylformamide*	dimethyl sulfoxide, dioxane, tetrahydrofuran, toluene
SuperH and H _{HR} ²	Tetrahydrofuran ³	acetone, ethanol, quinoline, benzene, <i>o</i> -dichlorobenzene, ethyl acetate, dodecane, FC-113, carbon tetrachloride ⁵ , dichloromethane, dichloroethane, trichloroethane, <i>n</i> -hexane, cyclohexane, xylene, chloroform, 1,4-dioxane, hexafluoroisopropanol, toluene, 1-chloronaphthalene, N,N-dimethylacetamide, methyl ethyl ketone, trichlorobenzene, <i>m</i> -cresol/chloroform, dimethylformamide, <i>o</i> -chlorophenol/chloroform, dimethyl sulfoxide, pyridine, N-methylpyrrolidone, hexafluoroisopropanol/chloroform
SuperMultiporeHZ	Tetrahydrofuran ³	<u>Cannot</u> be replaced. TSKgel SuperMultiporeHZ columns can be used only in tetrahydrofuran

¹ In case of TSKgel SuperHZ and H_{XL}, keep flow rate as mentioned below during solvent change. Solvent can be changed one way/one time only.

TSKgel H_{XL}: below <0.5 mL/min

TSKgel SuperHZ (4.6 mm ID): below <0.15 mL/min

TSKgel SuperHZ (6.0 mm ID): below <0.3 mL/min

² In case of TSKgel SuperH and H_{HR}, see Table 22 below for appropriate flow rates for solvent exchange. After switching to a very polar solvent, switching to a nonpolar solvent is not recommended.

³ All TSKgel H_{XL}, H_{HR}, SuperHZ, SuperH, SuperMultipore, and GMH analytical columns are shipped containing tetrahydrofuran (THF), except the TSKgel high temperature columns, which contain *o*-dichlorobenzene (ODCB).

⁴ THF in TSKgel SuperHZ1000 and G1000H_{XL} columns cannot be replaced with dichloromethane or dichloroethane.

⁵ Prolonged exposure to carbon tetrachloride can corrode the stainless steel parts of a column and an HPLC system.

* TSKgel H series columns may be specially ordered with this shipping solvent.

Please note: 100% methanol cannot be used with TSKgel H series columns; use this solvent with TSKgel SW or Alpha columns.



Table 22: Recommended flow rates (mL/min) for TSKgel SuperH and H_{HR} columns

Solvent	TSKgel SuperH, 6.0 mm ID × 15 cm	TSKgel H _{HR} , 7.8 mm ID × 30 cm
<i>n</i> -Hexane	0.5	0.9
methyl ethyl ketone	0.4	0.7
dichloromethane, ethyl acetate	0.35	0.6
toluene, chloroform	0.3	0.5
dimethylformamide	0.2	0.4
carbon tetrachloride, pyridine	0.15	0.3
dimethyl sulfoxide, dioxane, ethanol, N-methylpyrrolidone, <i>o</i> -dichlorobenzene	0.1	0.2
quinoline, hexafluoroisopropanol, 1-chloronaphthalene	0.05	0.1

Table 23: Recommended solvents by application for TSKgel H series columns

Recommended solvent	Application
THF	polystyrene, epoxy resin, phenoxy resin, polycarbonate, polyisoprene, polyvinyl acetate, polyvinyl chloride, monoglycerides, fatty acids, polybutadiene, poly(methyl methacrylate), poly(styrene-butadiene), poly(styrene-acrylonitrile)
N,N-Dimethylformamide (DMF) + 5 mmol/L LiBr	polyvinyl chloride, polyvinyl fluoride, urea resins, polyurethane, polystyrene, polyester, polyimido ether, polyimido ester, polyphenol (aqueous solution), polyacrylonitrile
<i>o</i> -Dichlorobenzene (ODCB)	polyethylene, polypropylene
Chloroform	polycarboxylic ether, acrylic resin, epoxy resin, polystyrene
<i>m</i> -Cresol/Chloroform	nylon, polyester, polyamide, poly (ethylene terephthalate)
Toluene	polybutadiene, polysiloxane

About: TSKgel H_{XL} Size Exclusion Columns

TSKgel H_{XL} columns are conventional GPC columns of 7.8 mm ID × 30 cm containing 5, 6, 9, or 13 μm particles composed of PS-DVB. The TSKgel H_{XL} column lines consists of eight columns with different pore sizes, TSKgel G1000H_{XL} through TSKgel G7000H_{XL}, and three columns with an extended linear range of the calibration curve, TSKgel GMH_{XL}, TSKgel GMH_{XL}-L and TSKgel MultiporeH_{XL}-M.

The TSKgel H_{XL} column line consists of the following columns:

- TSKgel G1000H_{XL}
- TSKgel G2000H_{XL}
- TSKgel G2500H_{XL}
- TSKgel G3000H_{XL}
- TSKgel G4000H_{XL}
- TSKgel G5000H_{XL}
- TSKgel G6000H_{XL}
- TSKgel G7000H_{XL}
- TSKgel GMH_{XL} mixed bed
- TSKgel GMH_{XL}-L mixed bed
- TSKgel MultiporeH_{XL}-M

Three of the linear columns are mixed bed columns, in which particles with different pore sizes are blended to provide an extended linear calibration curve. The remaining column is a multi-pore column, in which each particle contains a range of pore sizes that provide a linear calibration curve. The innovative multi-pore approach, pioneered by Tosoh, is a synthetic chemistry answer to the question of how to obtain a column with an extended linear calibration curve, while mixed bed columns represent a mechanical way of obtaining a linear calibration curve. In general, Multipore columns have a smoother, more linear, calibration curve.

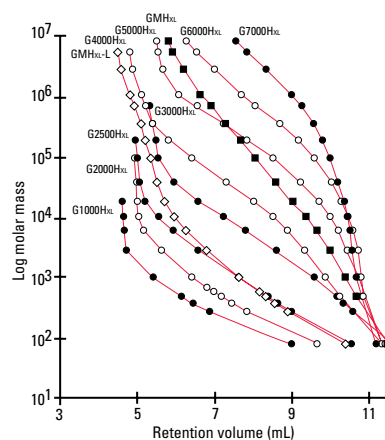
Attributes and Applications:

Product attributes of all of the TSKgel H_{XL} columns are shown in Table 24. These columns are for the use of conventional polymer analysis and show ultra-low polymer absorption, i.e., the columns show true size exclusion behavior for most polymers. TSKgel H_{XL} columns are shipped in THF, with the exception of the TSKgel GMH_{XL}-HT column, which is shipped in *o*-dichlorobenzene. These columns can be exchanged for a limited number of organic solvents. See the table within the TSKgel H series column overview for a listing of these solvents. Figures 82-83 show the calibration curves for the TSKgel H_{XL} columns.

Table 24: Product attributes

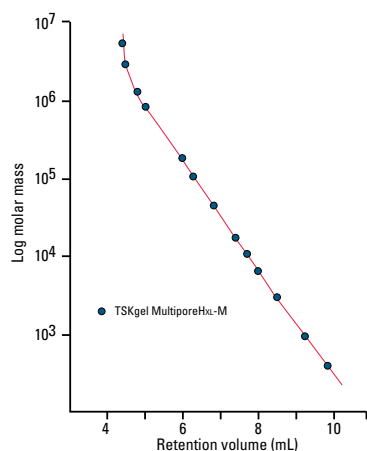
TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
G1000H _{XL}	5 μm	1.5 nm	1,000 Da	60 °C
G2000H _{XL}	5 μm	2 nm	1.0 × 10 ⁴ Da	60 °C
G2500H _{XL}	5 μm	3 nm	2.0 × 10 ⁴ Da	60 °C
G3000H _{XL}	5 μm	7.5 nm	6.0 × 10 ⁴ Da	60 °C
G4000H _{XL}	5 μm	20 nm	4.0 × 10 ⁵ Da	80 °C
G5000H _{XL}	9 μm	65 nm	4.0 × 10 ⁶ Da	80 °C
G6000H _{XL}	9 μm	>65 nm	4.0 × 10 ⁷ Da	80 °C
G7000H _{XL}	9 μm	>65 nm	4.0 × 10 ⁸ Da	80 °C
GMH _{XL}	9 μm	mixed pore sizes	4.0 × 10 ⁸ Da	80 °C
GMH _{XL} -L	5 μm	mixed pore sizes	4.0 × 10 ⁶ Da	80 °C
MultiporeH _{XL} -M	5 μm	broad distribution of pore size in each particle	2.0 × 10 ⁶ Da	60 °C

Figure 82: Calibration curves of TSKgel H_{XL} columns



Column: TSKgel H_{XL} columns, 7.8 mm ID × 30 cm
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Sample: polystyrene standards

Figure 83: Calibration curve of TSKgel MultiporeH_{XL}-M column

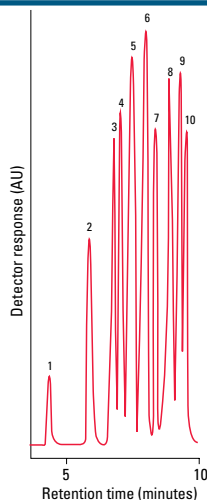


Columns: **TSKgel MultiporeH_{XL}-M, 5 μ m, 7.8 mm ID \times 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Temperature: 40 $^{\circ}$ C
 Sample: polystyrene standards

Phthalate Esters

Figure 84 demonstrates the high efficiency separation on a TSKgel G1000H_{XL} column for low molar mass phthalate esters. Resolution was close to baseline even though the molar masses of the esters differed by less than 50 Da.

Figure 84: High resolution of phthalate esters

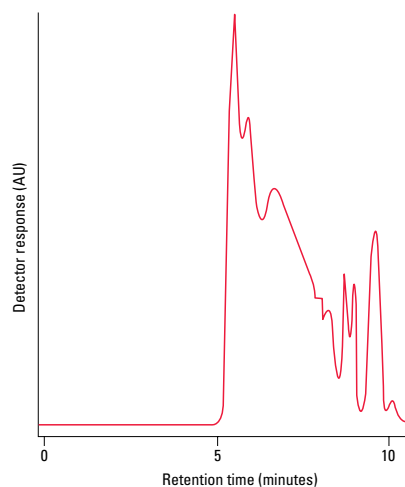


Column: **TSKgel G1000H_{XL}, 5 μ m, 7.8 mm ID \times 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Samples: 1. polystyrene (1.0×10^4 Da) 2. dioctylphthalate (391 Da)
 3. dibutylphthalate (278 Da) 4. dipropylphthalate (250 Da)
 5. diethylphthalate (222 Da) 6. dimethylphthalate (194 Da)
 7. n-propylbenzene (120 Da) 8. ethylbenzene (116 Da)
 9. toluene (92 Da) 10. benzene (78 Da)

Phenol Resin

The TSKgel GMH_{XL}-L column has been designed to provide a complete profile for high molar mass samples that contain low molar mass additives. The calibration curve for this mixed bed column is shallow in the low molar mass range of oligomers. Sample adsorption is not observed. For example, the complete profile of a phenol resin, with high resolution of the low molar mass components, is shown in Figure 85. Other applications for the TSKgel GMH_{XL}-L column include analyses of paint materials, bond and adhesive components and synthetic polymer additives.

Figure 85: Separation of phenol resin

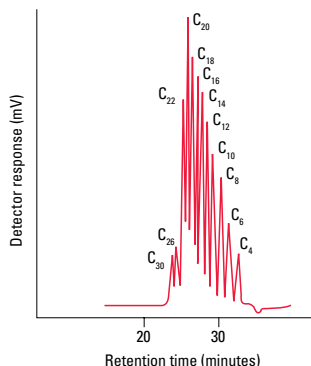


Column: **TSKgel GMH_{XL}-L, 5 μ m, 7.8 mm ID \times 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Sample: phenol resin

Fatty Acids

In **Figure 86**, two TSKgel G2000H_{XL} columns in series separate a mixture of fatty acids ranging from C₄ to C₃₀.

Figure 86: Separation of fatty acids

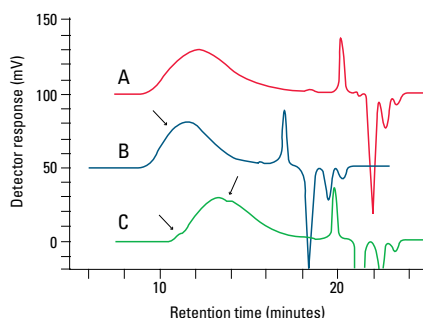


Column: **TSKgel G2000H_{XL}, 5 μ m, 7.8 mm ID \times 30 cm \times 3**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: RI
 Sample: fatty acids

Acrylic Polymer

Figure 87 shows the separation of an acrylic polymer on the TSKgel MultiporeH_{XL}-M column compared with two commercially available mixed bed columns. The arrows illustrate the inflections seen in the chromatograms from mixed bed columns and the improvement achieved when using the TSKgel MultiporeH_{XL}-M column.

Figure 87: Separation of acrylic resin

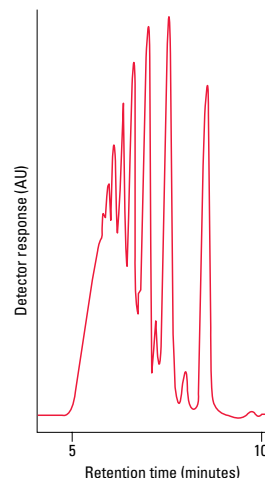


Columns: **A. TSKgel MultiporeH_{XL}-M, 5 μ m, 7.8 mm ID \times 30 cm \times 2 in series**
B. Competitor P, 7.5 mm ID \times 30 cm \times 2 in series, mixed bed type
C. Competitor S, 8.0 mm ID \times 30 cm \times 2 in series, mixed bed type
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Temperature: 40 °C
 Detection: RI
 Sample: acrylic polymer (0.1%, 50 μ L)

Epoxy Resin

The analysis of a commercial epoxy resin, Epikote 1001, using a TSKgel G2500H_{XL} column is shown in **Figure 88**.

Figure 88: Separation of epoxy resin

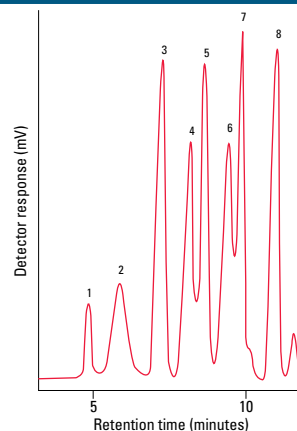


Column: **TSKgel G2500H_{XL}, 5 μ m, 7.8 mm ID \times 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Sample: Epikote 1001 epoxy resin

Polystyrene

For polymer mixtures that contain low and high molar mass compounds, a TSKgel G4000H_{XL} column provides high resolution of samples ranging in size from benzene to 1.126×10^6 Da as shown in **Figure 89**.

Figure 89: Separation of polystyrene standards



Column: **TSKgel G4000H_{XL}, 5 μ m, 7.8 mm ID \times 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Samples: polystyrene standards
 1. 1.126×10^6 Da 2. 1.86×10^5 Da 3. 4.28×10^4 Da
 4. 1.67×10^4 Da 5. 1.02×10^4 Da 6. 2,800 Da
 7. 890 Da 8. benzene, 78 Da



About: TSKgel H_{HR} Size Exclusion Columns

TSKgel H_{HR} columns are conventional GPC columns with dimensions of 7.8 mm ID × 30 cm containing spherical particles composed of PS-DVB. The TSKgel H_{HR} column line consists of eight columns with different pore sizes, TSKgel G1000H_{HR} through TSKgel G7000H_{HR}, and ten columns with an extended linear range of the calibration curve.

The TSKgel H_{HR} column line consists of the following columns:

- TSKgel G1000H_{HR}
- TSKgel G2000H_{HR}
- TSKgel G2500H_{HR}
- TSKgel G3000H_{HR}
- TSKgel G4000H_{HR}
- TSKgel G5000H_{HR}
- TSKgel G6000H_{HR}
- TSKgel G7000H_{HR}
- TSKgel GMH_{HR}-H mixed bed
- TSKgel GMH_{HR}-L mixed bed
- TSKgel GMH_{HR}-M mixed bed
- TSKgel GMH_{HR}-N mixed bed
- TSKgel G2000H_{HR} (20) HT
- TSKgel GMH_{HR}-H (S) HT mixed bed
- TSKgel GMH_{HR}-H HT mixed bed
- TSKgel G2000H_{HR} (20) HT2
- TSKgel GMH_{HR}-H (S) HT2 mixed bed
- TSKgel GMH_{HR}-H HT2 mixed bed

The linear, or mixed bed columns, contain particles with different pore sizes that are blended to provide an extended linear calibration curve. The mixed bed columns feature increasing linear calibration ranges, from TSKgel GMH_{HR}-L, GMH_{HR}-N, GMH_{HR}-M, to GMH_{HR}-H. All of the TSKgel high temperature mixed bed columns are shipped in ODCB (*o*-dichlorobenzene).

The TSKgel H_{HR} HT2 mixed bed columns are available for ultra-high temperature analysis. Packed with PS-DVB beads, the maximum operating temperature of these columns is 220 °C.

The issue of shearing that occurs with the analysis of ultra-high molar mass polymers is overcome by the TSKgel GMH_{HR}-M (S), TSKgel GMH_{HR}-H (S), GMH_{HR}-H (S) HT and GMH_{HR}-H (S) HT2 columns. The (S) is a reference to this shearing effect.

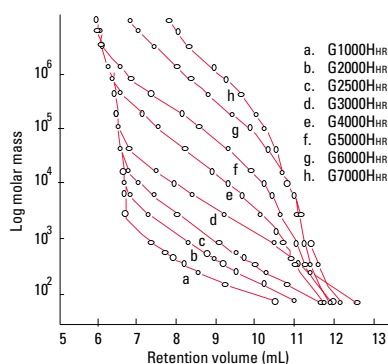
Attributes and Applications:

The product attributes for all of the TSKgel H_{HR} columns is shown in Table 25. TSKgel H_{HR} columns have a broad solvent range and are shipped in THF, except for the high temperature mixed bed columns, which are shipped in ODCB (*o*-dichlorobenzene). THF can be exchanged for a wide variety of organic solvents. See the table within the TSKgel H series column overview for a listing of these solvents. Figures 90-94 show the calibration curves for the TSKgel H_{HR} columns.

Table 25: Product attributes

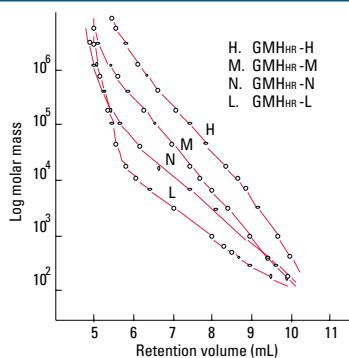
TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
G1000H _{HR}	5 µm	1.5 nm	1,000 Da	140 °C
G2000H _{HR}	5 µm	2 nm	1.0 × 10 ⁴ Da	140 °C
G2500H _{HR}	5 µm	3 nm	2.0 × 10 ⁴ Da	140 °C
G3000H _{HR}	5 µm	7.5 nm	6.0 × 10 ⁴ Da	140 °C
G4000H _{HR}	5 µm	20 nm	4.0 × 10 ⁵ Da	140 °C
G5000H _{HR}	5 µm	65 nm	4.0 × 10 ⁶ Da	140 °C
G6000H _{HR}	5 µm	>65 nm	4.0 × 10 ⁷ Da	140 °C
G7000H _{HR}	5 µm	>65 nm	4.0 × 10 ⁸ Da	140 °C
GMH _{HR} -H	5 µm, 13 µm, 20 µm, 30 µm	mixed pore sizes	4.0 × 10 ⁸ Da	80 °C
GMH _{HR} -H (S) HT	13 µm	mixed pore sizes	4.0 × 10 ⁸ Da	140 °C
TSKgel G2000H _{HR} (20) HT	20 µm	2 nm	1 × 10 ⁴ Da	140 °C
GMH _{HR} -H (20) HT	20 µm	mixed pore sizes	4 × 10 ⁸ Da	140 °C
GMH _{HR} -H (30) HT	30 µm	mixed pore sizes	4 × 10 ⁸ Da	140 °C
GMH _{HR} -H HT	5 µm	mixed pore sizes	4 × 10 ⁸ Da	140 °C
GMH _{HR} -L	5 µm	mixed pore sizes	4.0 × 10 ⁶ Da	80 °C
GMH _{HR} -M	5 µm, 13 µm	mixed pore sizes	4.0 × 10 ⁶ Da	80 °C
GMH _{HR} -N	5 µm	mixed pore sizes	4.0 × 10 ⁵ Da	80 °C
TSKgel G2000H _{HR} (20) HT2	20 µm	2 nm	1 × 10 ⁴ Da	220 °C
GMH _{HR} -H (20) HT2	20 µm	mixed pore sizes	4 × 10 ⁸ Da	220 °C
GMH _{HR} -H (30) HT2	30 µm	mixed pore sizes	4 × 10 ⁸ Da	220 °C
GMH _{HR} -H (S) HT2	13 µm	mixed pore sizes	4 × 10 ⁸ Da	220 °C

Figure 90: Calibration curves of TSKgel H_{HR} columns



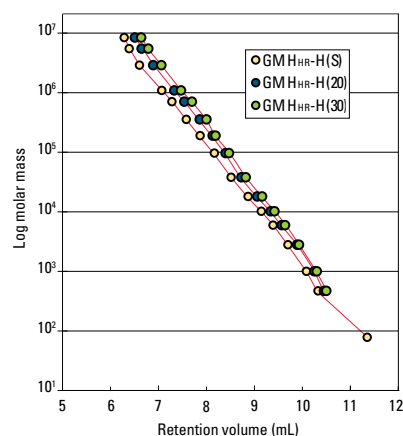
Column: **TSKgel H_{HR} columns, 7.8 mm ID × 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Samples: polystyrene standards

Figure 91: Calibration curves of TSKgel H_{HR} mixed bed columns



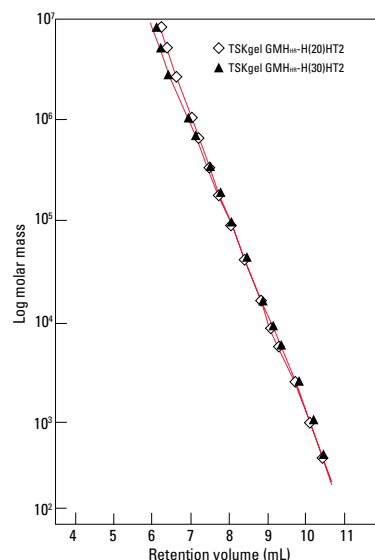
Columns: **TSKgel H_{HR} columns, 7.8 mm ID × 30 cm**
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Samples: polystyrene standards

Figure 92: Calibration curves of TSKgel H_{HR} columns



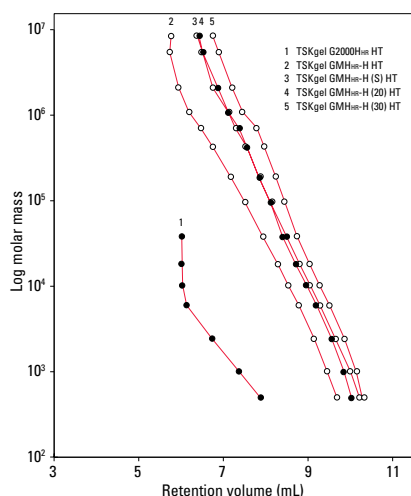
Columns: **TSKgel GMH_{HR}-H (S), 13 µm, 7.8 mm ID × 30 cm**
TSKgel GMH_{HR}-H (20), 20 µm, 7.8 mm ID × 30 cm
TSKgel GMH_{HR}-H (30), 30 µm, 7.8 mm ID × 30 cm
 Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Sample: polystyrene standards

Figure 93: Calibration curves of TSKgel GMH_{HR}-H HT2 columns



Columns: **TSKgel GMH_{HR}-H (20) HT2, 20 µm, 7.8 mm ID × 30 cm**
TSKgel GMH_{HR}-H (30) HT2, 30 µm, 7.8 mm ID × 30 cm
 Mobile phase: ODCB with 0.05% BHT
 Flow rate: 1.0 mL/min
 Detection: RI
 Temperature: 135 °C
 Sample: polystyrene standards

Figure 94: Calibration curves of TSKgel HT columns



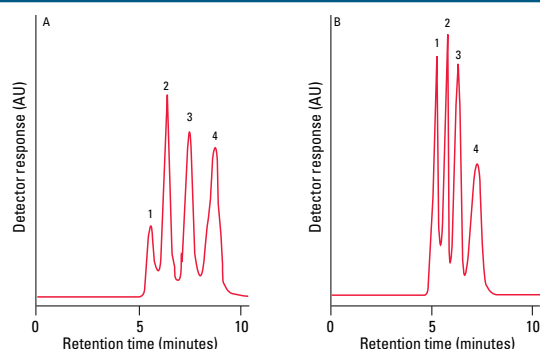
Columns: **TSKgel G2000HHR (20) HT, 20 μ m, 7.8 mm ID \times 30 cm**
TSKgel GMHHR-H HT, 5 μ m, 7.8 mm ID \times 30 cm
TSKgel GMHHR-H (S) HT, 13 μ m, 7.8 mm ID \times 30 cm
TSKgel GMHHR-H (20) HT, 20 μ m, 7.8 mm ID \times 30 cm
TSKgel GMHHR-H (30) HT, 30 μ m, 7.8 mm ID \times 30 cm

Mobile phase: ODCB with 0.05% BHT
 Flow rate: 1.0 mL/min
 Detection: RI (EcoSEC High Temperature GPC System)
 Temperature: 135 $^{\circ}$ C
 Injection vol.: 300 μ L
 Sample: polystyrene

Polymethyl Methacrylate

The effect of different pore size distributions in the mixed beds of TSKgel GMHHR-H and TSKgel GMHHR-M is illustrated in Figure 95. The TSKgel GMHHR-M produces sharper polymethyl methacrylate peaks in the 8.0×10^5 to 1.0×10^4 Da range.

Figure 95: Comparison of standard polymethyl methacrylate mixture



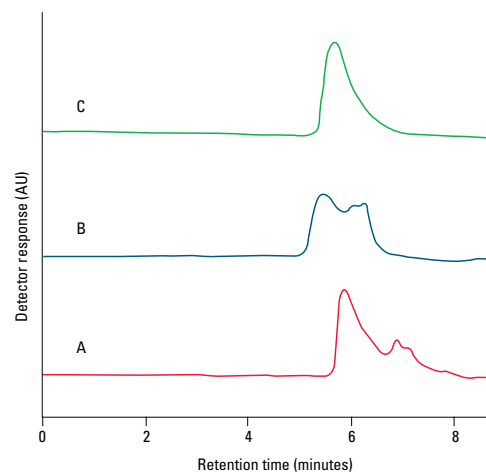
Columns: **A. TSKgel GMHHR-H, 5 μ m, 7.8 mm ID \times 30 cm**
B. TSKgel GMHHR-M, 5 μ m, 7.8 mm ID \times 30 cm

Mobile phase: 5 mmol/L sodium trifluoroacetate in HFIP
 Flow rate: 1.0 mL/min
 Detection: UV @ 220 nm
 Temperature: 40 $^{\circ}$ C
 Sample: standard polymethylmethacrylate
 1. 8.2×10^5 Da
 2. 6.7×10^4 Da
 3. 1.02×10^4 Da
 4. 1,950 Da

Shear Degradation

Shear degradation is observed especially when ultra-high molar mass compounds are analyzed. It tends to occur when analysis is carried out at high flow rates using a micro-particle size packing material. Figure 96 demonstrates the relationship between shear degradation and particle size of the packing material, when TSKgel GMH columns were used. When the flow rate is 1.0 mL/min, normal elution of an ultra-high molar mass sample (2.06×10^7 Da) is only possible with the TSKgel GMHHR-H (S) column, which has a large particle size. However, with the TSKgel GMHXL and GMHHR-H columns, shear degradation does take place and new peaks appear in the chromatogram on the smaller molar mass side.

Figure 96: Shear degradation comparison



Columns: **A: TSKgel GMHHR-H, 5 μ m, 7.8 mm ID \times 30 cm**
B: TSKgel GMHXL, 9 μ m, 7.8 mm ID \times 30 cm
C: TSKgel GMHHR-H (S), 13 μ m, 7.8 mm ID \times 30 cm

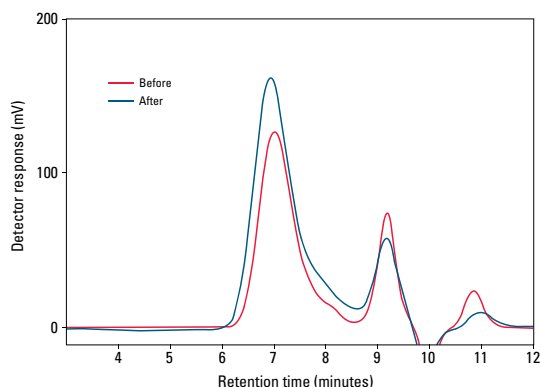
Mobile phase: THF
 Flow rate: 1.0 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 $^{\circ}$ C
 Sample: polystyrene standard F2000 (2.06×10^7 Da)
 20 μ L (0.025%)

Column Durability at 220 °C

Column durability in high temperature GPC polymer analysis is essential as these columns are continuously exposed to harsh organic solvents, extremely elevated temperatures and temperature cycling as GPC systems are turned on and off. The durability of a high temperature GPC column directly influences the quality, applicability and selectivity, or resolution, of the GPC column, thus the accuracy of the molar mass averages obtained. As a high temperature GPC column begins to fail or lose resolution due to the extreme experimental conditions required for high temperature GPC polymer analysis, the number- and z-average molar mass values obtained become inflated and the GPC elution profile begins to shift due to a decrease in multiple factors that affect the ability of the columns to separate species varying in hydrodynamic volume.

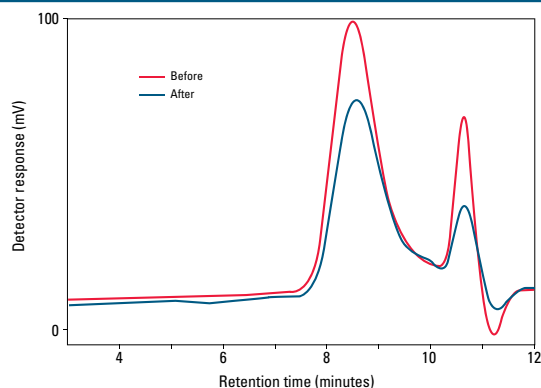
A durability and stability study of a TSKgel GMH_{HR}-H (S) HT high temperature GPC column was performed and the results were compared to another commercially available column for polymer analysis at 220 °C. The deterioration of the commercially available high temperature GPC column is observed in the GPC elution profiles, **Figure 97**, as the resolution between the sample and solvent peaks decreases after the column is exposed to temperature cycling. The GPC elution profiles obtained for the TSKgel GMH_{HR}-H (S) HT column before and after temperature cycling remain superimposable, **Figure 98**.

Figure 97: GPC elution profile for a polymer before and after temperature cycling obtained using a commercially available high temperature GPC column



Column: Commercially available high temperature GPC column,
13 μ m, 7.8 mm ID \times 30 cm
Mobile phase: 1-chloronaphthalene
Flow rate: 1.0 mL/min
Detection: RI
Temperature: 220 °C
Injection vol.: 200 μ L
Sample: synthetic polymer

Figure 98: GPC elution profile for a polymer before and after temperature cycling obtained using a TSKgel GMH_{HR}-H (S) HT column

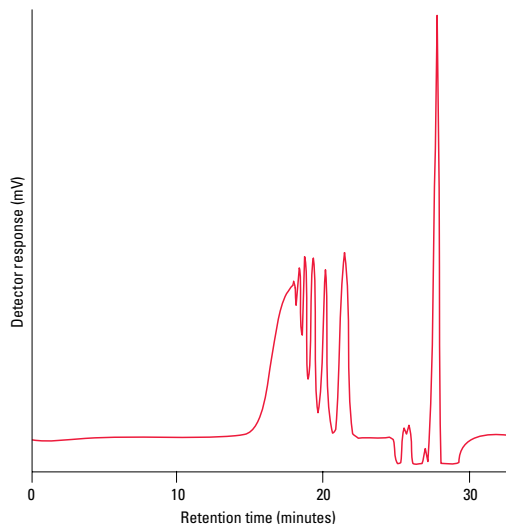


Column: TSKgel GMH_{HR}-H (S) HT, 13 μ m, 7.8 mm ID \times 30 cm
Mobile phase: 1-chloronaphthalene
Flow rate: 1.0 mL/min
Detection: RI
Temperature: 220 °C
Injection vol.: 200 μ L
Sample: synthetic polymer

Dextran T-40 Hydrolysate

The analysis of dextran T-40 hydrolysate is shown using TSKgel G3000H_{HR} and G2500H_{HR} columns in series in **Figure 99** below.

Figure 99: Dextran T-40 hydrolysate analysis

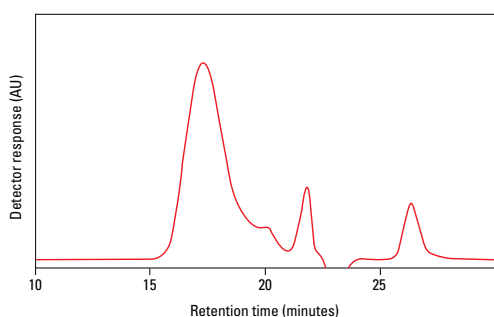


Columns: **TSKgel G3000H_{HR}, 5 μ m, 7.8 mm ID \times 30 cm**
TSKgel G2500H_{HR}, 5 μ m, 7.8 mm ID \times 30 cm
 Mobile phase: 10 mmol/L lithium chloride in N-methylpyrrolidone
 Flow rate: 0.75 mL/min
 Detection: RI
 Temperature: 80 °C
 Sample: dextran T-40 hydrolysate

Polyphenylene Sulfide

The analysis of PPS (polyphenylene sulfide) is shown using two TSKgel GMH_{HR}-H (S) HT2 columns in series in **Figure 100** below.

Figure 100: Polyphenylene sulfide analysis

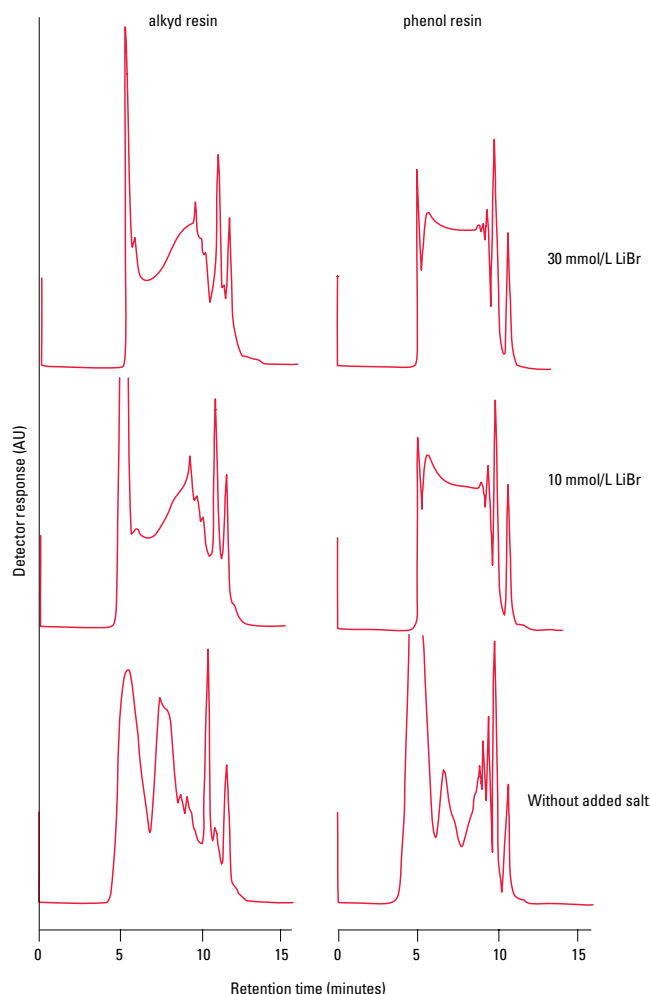


Column: **TSKgel GMH_{HR}-H (S) HT2, 13 μ m, 7.8 mm ID \times 30 cm \times 2**
 Mobile phase: 1-chloronaphthalene
 Flow rate: 1.0 mL/min
 Detection: RI
 Temperature: 220 °C
 Injection vol.: 300 μ L
 Sample: PPS (polyphenylene sulfide), 2 g/L

Effect of Adding Salt

Using the TSKgel G3000H_{HR} column, **Figure 101** shows the elution behavior of alkyd resin and phenol resin analyzed using a DMF solvent, as well as the effects of adding LiBr to DMF. With the DMF solvent, both resins eluted abnormally early from the column due to a static electric interaction. However, by adding LiBr to the DMF solvent, a normal chromatogram is obtained. Normal elution behavior of alkyd resins is possible when the LiBr concentration is about 30 mmol/L, and with phenol resins, when the concentration of the salt is around 10 mmol/L.

Figure 101: Separation of alkyd resin and phenol resin



Column: **TSKgel G3000H_{HR}, 5 μ m, 7.8 mm ID \times 30 cm**
 Mobile phase: DMF (containing LiBr)
 Flow rate: 1.0 mL/min
 Detection: UV @ 270 nm
 Temperature: 25 °C
 Samples: alkyd resin, phenol resin

About: TSKgel SuperH Size Exclusion Columns

TSKgel SuperH columns are conventional GPC columns with dimensions of 6.0 mm ID × 15 cm containing spherical particles composed of PS-DVB. The TSKgel SuperH column line consists of eight columns with different pore sizes, TSKgel SuperH1000 through TSKgel SuperH7000, and four columns with an extended linear range of the calibration curve.

TSKgel SuperH columns are high efficiency/high throughput versions of the conventional TSKgel H_{HR} columns. Both column types are based on the same bead chemistry.

The TSKgel SuperH line consists of the following columns:

- TSKgel SuperH1000
- TSKgel SuperH2000
- TSKgel SuperH2500
- TSKgel SuperH3000
- TSKgel SuperH4000
- TSKgel SuperH5000
- TSKgel SuperH6000
- TSKgel SuperH7000
- TSKgel SuperHM-H mixed bed
- TSKgel SuperHM-L mixed bed
- TSKgel SuperHM-M mixed bed
- TSKgel SuperHM-N mixed bed

The TSKgel SuperH product line contains four linear or mixed bed columns, in which particles with different pore sizes are blended to provide an extended linear calibration curve. The mixed bed columns feature increasing linear calibration ranges, from TSKgel SuperHM-L, SuperHM-M, SuperHM-N, to SuperHM-H.

The volume of a 6 mm ID × 15 cm TSKgel SuperH column is 3.4 times smaller than that of a conventional 7.8 mm ID × 30 cm column. As a result, peak volumes will be proportionally smaller on TSKgel SuperH columns compared to a corresponding TSKgel H_{HR} column. Thus, your HPLC system may require optimization of components that can give rise to extra-column band broadening, such as connecting tubing, injector, injection volume, detector cell volume, and detector time constant.

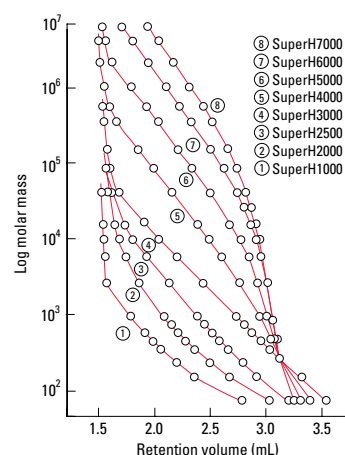
Attributes and Applications:

Table 26 shows product attributes of TSKgel SuperH columns. The maximum operating temperature for TSKgel SuperH columns is 140 °C. All TSKgel SuperH columns are shipped in THF, which can be exchanged for a wide variety of organic solvents. See the table within the TSKgel H series column overview for a listing of these solvents. Figures 102 and 103 show the calibration curves for the TSKgel SuperH columns.

Table 26: Product attributes

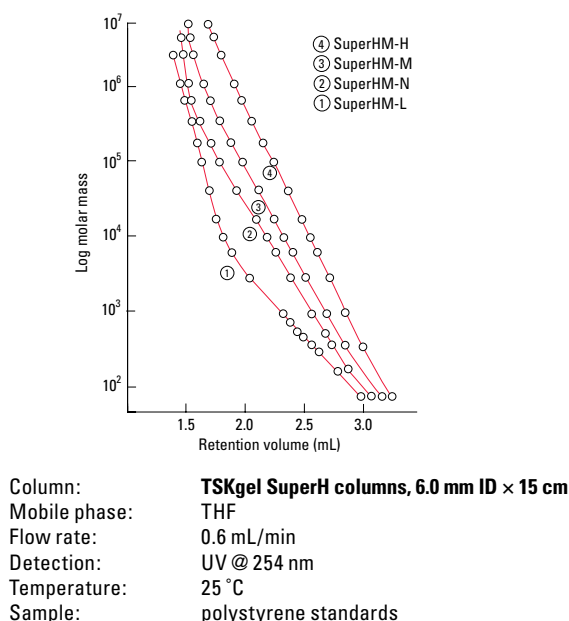
TSKgel column	Particle size (mean)	Pore size (mean)	Exclusion limit
SuperH1000	3 µm	1.5 nm	1,000 Da
SuperH2000	3 µm	2 nm	1.0 × 10 ⁴ Da
SuperH2500	3 µm	3 nm	2.0 × 10 ⁴ Da
SuperH3000	3 µm	7.5 nm	6.0 × 10 ⁴ Da
SuperH4000	3 µm	20 nm	4.0 × 10 ⁵ Da
SuperH5000	3 µm	65 nm	4.0 × 10 ⁶ Da
SuperH6000	5 µm	>65 nm	4.0 × 10 ⁷ Da
SuperH7000	5 µm	>65 nm	4.0 × 10 ⁸ Da
SuperHM-H	3 µm	mixed pore sizes	4.0 × 10 ⁸ Da
SuperHM-L	3 µm	mixed pore sizes	4.0 × 10 ⁶ Da
SuperHM-M	3 µm	mixed pore sizes	4.0 × 10 ⁶ Da
SuperHM-N	3 µm	mixed pore sizes	4.0 × 10 ⁵ Da

Figure 102: Calibration curves for TSKgel SuperH columns



Column: **TSKgel SuperH columns, 6.0 mm ID × 15 cm**
 Mobile phase: THF
 Flow rate: 0.6 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Sample: polystyrene standards

Figure 103: Calibration curves for TSKgel SuperH mixed bed columns

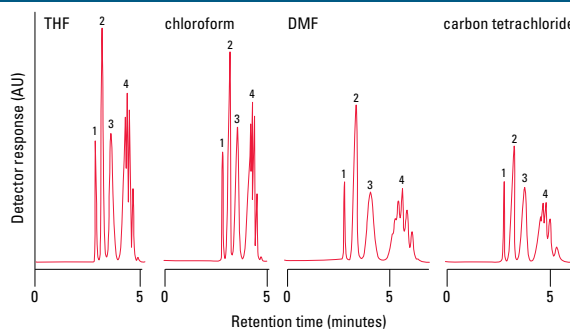


Polystyrene Mixtures

Figure 104 compares chromatograms of standard polystyrene mixtures separated using a TSKgel SuperH2500 column with various organic solvents (THF, CHCl₃, DMF, and CCl₄) and Figure 105 compares chromatograms of standard polystyrene mixtures separated using a TSKgel SuperHM-H column with various organic solvents.

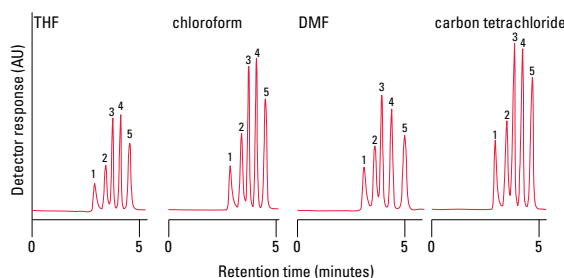
Due to the interaction between the packing material and standard polystyrene when using DMF as the mobile phase, the elution volume of standard polystyrenes is greater than it is with “good” solvents such as THF and CHCl₃. This effect is particularly noticeable with TSKgel SuperH2500, a column for the analysis of low molar mass samples. Under these circumstances, polyethylene oxide (PEO) is recommended as the standard sample, as this reacts very little with the packing material.

Figure 104: Separation of standard polystyrenes using a TSKgel SuperH2500 column



Column: **TSKgel SuperH2500, 3 μm, 6 mm ID × 15 cm**
 Mobile phase: THF, chloroform, DMF, carbon tetrachloride
 Flow rate: 0.6 mL/min
 Temperature: 25 °C
 Detection: UV/VIS @ 254 nm or 270 nm
 Samples: 1) polystyrene (1.9 × 10⁵ Da)
 2) polystyrene (9.1 × 10⁴ Da)
 3) polystyrene (2,800 Da)
 4) polystyrene A-500

Figure 105: Separation of standard polystyrenes using a TSKgel SuperHM-H column



Column: **TSKgel SuperHM-H, 3 μm, 6 mm ID × 15 cm**
 Mobile phase: THF, chloroform, DMF, carbon tetrachloride
 Flow rate: 0.6 mL/min
 Temperature: 25 °C
 Detection: UV/VIS @ 254 nm
 Sample: 1. polystyrene (2.89 × 10⁶ Da)
 2. polystyrene (4.22 × 10⁵ Da)
 3. polystyrene (1.07 × 10⁵ Da)
 4. polystyrene (1.67 × 10⁴ Da)
 5. polystyrene (2,800 Da)

Band Broadening in the Detector

Table 27 compares the number of theoretical plates for a low molar mass sample (DCHP) using a TSKgel SuperH2500 column with various types of UV detectors and different flow cell volumes. **Figure 106** compares the separation performance of each of these using standard polystyrene A-500 and epoxy resin samples. Based on these results, it is clear that the number of theoretical plates and the separation performance of the TSKgel SuperH column are significantly affected by band broadening in the detector, including the size of the flow cell. In analyses performed with a TSKgel SuperH column, a UV-8020 microcell (or an equivalent device) with reduced dead volume must be used in the detector.

Table 27: Comparison of number of theoretical plates with various detectors

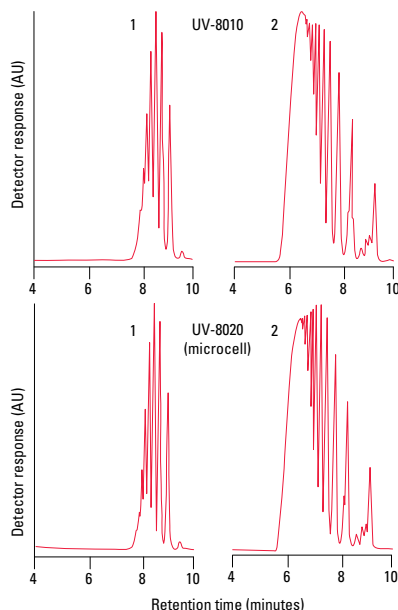
Number of theoretical plates (TP/15 cm)		
UV-8020*1	UV-8010*2	UV-8010*3
28,100	23,860	17,890
Column: TSKgel SuperH2500, 3 μ m, 6 mm ID \times 15 cm		
Mobile phase: THF		
Flow rate: 0.6 mL/min		
Temperature: 25 $^{\circ}$ C		
Detection: UV @ 254 nm		
Sample: DCHP 0.1%, 2 μ L		

*1 flow cell volume: 2 μ L microcell

*2 flow cell volume: 10 μ L low dead volume type

*3 flow cell volume: 10 μ L

Figure 106: Dependence of separation performance on band spreading in detector



Column: TSKgel SuperH2500, 3 μ m, 6 mm ID \times 15 cm \times 2
 Mobile phase: THF
 Flow rate: 0.6 mL/min
 Detection: UV/VIS @ 254 nm
 Temperature: 25 $^{\circ}$ C
 Samples: 1. standard polystyrene A-500 (0.1%), 10 μ L
 2. Epikote 1004 (0.1%), 10 μ L

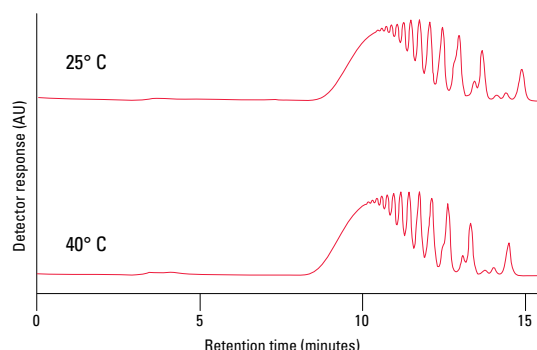
Column Temperature

The following advantages are gained by conducting analysis at high temperature:

- Peaks become sharper as separation performance is increased. This is especially noticeable at higher flow rates.
- Viscosity of the mobile phase is lowered and operating pressure is decreased. This is an especially effective method with high-viscosity solvents such as DMSO, DMF, HFIP, etc.

Figure 107 demonstrates the temperature dependence of the separation of epoxy resin and a standard polystyrene mixture in TSKgel SuperH3000 and SuperH2500 columns.

Figure 107: Temperature dependence of separation on epoxy resin



Columns: TSKgel SuperH3000, 3 μ m, 6 mm ID \times 15 cm \times 2 +
 TSKgel SuperH2500, 3 μ m, 6 mm ID \times 15 cm \times 3
 Mobile phase: THF
 Flow rate: 0.6 mL/min
 Detection: UV @ 254 nm
 Sample: Epikote 1004 (0.1%), 10 μ L



About: TSKgel SuperHZ Size Exclusion Columns

The TSKgel SuperHZ column line consists of five columns of 4.6 mm ID and 6.0 mm ID × 15 cm containing spherical particles composed of PS-DVB, TSKgel Super HZ1000 – 4000. Each column consists of a different pore size packing material. Subsequently, a unique separation range for each column exists, allowing researchers to choose a column that is designed for the sample type being analyzed.

The TSKgel SuperHZ column line also contains three linear, or mixed bed columns in which particles with different pore sizes are blended to provide an extended linear calibration curve. The mixed bed columns feature increasing linear calibration ranges, from TSKgel SuperHSM-M to SuperHSM-N to SuperHSM-H. The mixed bed columns are also available in 4.6 mm ID and 6.0 mm ID × 15 cm.

The following eight columns are available within the TSKgel SuperHZ column line:

- TSKgel SuperHZ1000
- TSKgel SuperHZ2000
- TSKgel SuperHZ2500
- TSKgel SuperHZ3000
- TSKgel SuperHZ4000
- TSKgel SuperHSM-H mixed bed
- TSKgel SuperHSM-M mixed bed
- TSKgel SuperHSM-N mixed bed

TSKgel SuperHZ column dimensions are 6 mm ID × 15 cm and 4.6 mm ID × 15 cm versus 7.8 mm ID × 30 cm for conventional GPC columns. The smaller column dimensions translate to a reduction of peak volume by a factor of 3.4 (6 mm ID) and a factor of 5.8 (4.6 mm ID) versus the same component eluting from a corresponding TSKgel HxL column. Thus, your HPLC system may require optimization of components that can give rise to extra-column band broadening, such as connecting tubing, injector, injection volume, detector cell volume, and detector time constant.

Attributes and Applications:

TSKgel SuperHZ columns have been developed for high throughput, high efficiency GPC applications such as those encountered in combinatorial chemistry experiments. These columns feature ultra-low sample adsorption, i.e., the columns show true size exclusion behavior for most polymers.

TSKgel SuperHZ1000 – 4000 columns are capable of measuring monomers, polymer additives, oligomers and polymers up to a molar mass of several hundred thousand with proper selection of pore size. Ultra-fine particles (3 µm) have been developed to provide high resolution over the entire molar mass range. This is especially important for the separation of low molar mass compounds.

Additionally, the mixed bed columns (TSKgel SuperHSM-N, M-M, and M-H) are capable of measuring oligomers and polymers with molar masses up to tens of millions with proper selection of the pore size. The various particle sizes of the mixed bed packing materials have been optimized to ensure resolution in the low molar mass range while avoiding shear degradation of polymers in the high molar mass region.

The columns are shipped in THF, which can be exchanged for a limited number of organic solvents as shown in the table within the TSKgel H series column overview.

Table 28 shows the product attributes of TSKgel SuperHZ columns, while Table 29 lists the features of the TSKgel SuperHZ column line and the corresponding benefits. The calibration curves for the TSKgel SuperHZ columns are shown in Figures 106 and 109.

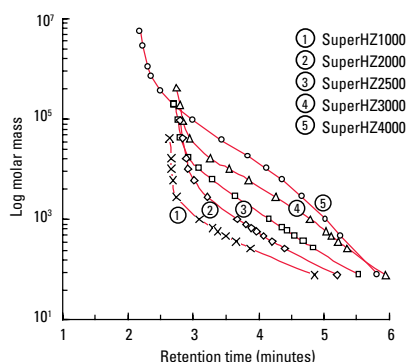
Table 28: Product attributes

TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
SuperHZ1000	3 µm	1.5 nm	1,000 Da	60 °C
SuperHZ2000	3 µm	2 nm	1.0 × 10 ⁴ Da	60 °C
SuperHZ2500	3 µm	3 nm	2.0 × 10 ⁴ Da	60 °C
SuperHZ3000	3 µm	7.5 nm	6.0 × 10 ⁴ Da	60 °C
SuperHZ4000	3 µm	20 nm	4.0 × 10 ⁵ Da	80 °C
SuperHSM-N	3 µm	mixed pore sizes	7.0 × 10 ⁵ Da	80 °C
SuperHSM-M	3 µm	mixed pore sizes	4.0 × 10 ⁶ Da	80 °C
SuperHSM-H	10 µm	mixed pore sizes	4.0 × 10 ⁸ Da	80 °C

Table 29: Features and benefits of TSKgel SuperHZ columns

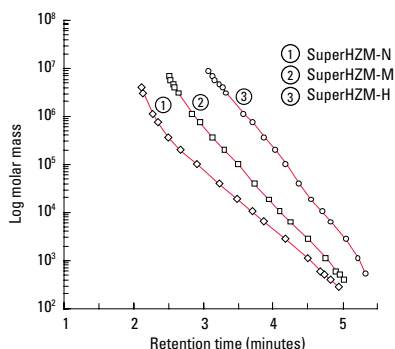
Feature	Benefit
Ultra-fine particles used in packing material	<ul style="list-style-type: none"> • Short measurement time is achieved. • Resolution equivalent to conventional columns (30 cm) can be obtained in ½ measurement time • Resolution does not deteriorate even under a high flow rate.
Semi-micro columns (4.6 mm ID and 6.0 mm ID)	<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
Optimization of particle size in the packing materials	<ul style="list-style-type: none"> • Shear degradation in polymers with high molar mass can be prevented
Adoption of low-adsorption packing materials	<ul style="list-style-type: none"> • Applicable to wide range of samples

Figure 108: Calibration curves for TSKgel SuperHZ columns



Column: **TSKgel SuperHZ columns, 4.6 mm ID × 15 cm**
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Temperature: 25 °C
 Injection vol.: 2 µL
 Samples: polystyrene standards

Figure 109: Calibration curves for TSKgel SuperHZ mixed bed columns

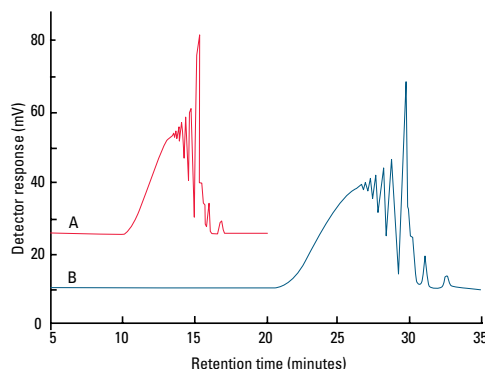


Column: **TSKgel SuperHZ columns, 4.6 mm ID × 15 cm**
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Temperature: 25 °C
 Injection vol.: 2 µL
 Samples: polystyrene standards

Faster Analysis

TSKgel SuperHZ1000-SuperHZ4000 columns are packed with 3 µm particles. The ultra-fine particles allow for high efficiency separations of low molar mass substances such as oligomers. These columns have theoretical plate values (per unit length) which are twice those of the conventional 5 µm columns. As a result, equal resolution can be obtained within half the analysis time. An example showing the analysis of phenolic resin is demonstrated in Figure 110.

Figure 110: Comparison of analysis on TSKgel SuperHZ and TSKgel H_{XL} columns

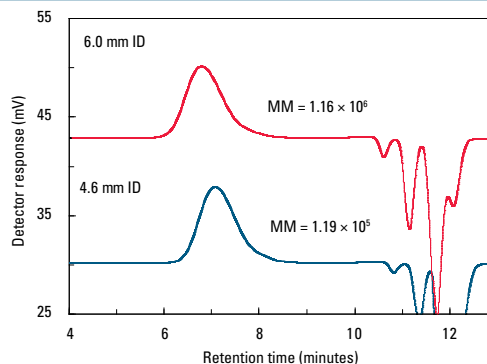


Columns: **A. TSKgel SuperHZ columns (4000, 3000, 2500), 4.6 mm ID × 15 cm × 3**
B. TSKgel H_{XL} columns (4000, 3000, 2500), 7.8 mm ID × 30 cm × 3
 Mobile phase: THF
 Flow rate: A. 0.35 mL/min B. 1.0 mL/min
 Detection: RI
 Temperature: 40 °C
 Injection vol.: A. 5 µL B. 30 µL
 Sample: phenolic resin

Polyisobutylene

The chromatogram in Figure 111 shows the analysis of polyisobutylene using two TSKgel SuperH2M-M columns in series

Figure 111: Analysis of polyisobutylene



Column: **TSKgel SuperH2M-M, 3 µm × 2**
 Mobile phase: THF
 Flow rate: 0.35 mL/min (4.6 mm ID), 0.6 mL/min (6.0 mm ID)
 Detection: RI
 Temperature: 40 °C
 Injection vol.: 10 µL (4.6 mm ID), 17 µL (6.0 mm ID)
 Sample: polyisobutylene (0.5 g/L)



About: TSKgel SuperMultiporeHZ Size Exclusion Columns

TSKgel SuperMultiporeHZ columns represent a new strategy for the separation of polymers with a wide range of molar masses. These columns are packed with particles of a uniform size, with each particle having a very broad pore size distribution. This innovative multi-pore approach, pioneered by Tosoh Bioscience, essentially creates a linear calibration curve within each particle. The spherical monodisperse, 3, 4 or 6 μm particles consist of cross-linked polystyrene/divinylbenzene copolymer. This base material, coupled with the semi-micro column dimensions (4.6 mm ID \times 15 cm), offers users high speed and low solvent consumption analyses with precise results. Three columns are available within the TSKgel SuperMultiporeHZ series, each with a different particle size and separation range.

The TSKgel SuperMultiporeHZ columns offered include:

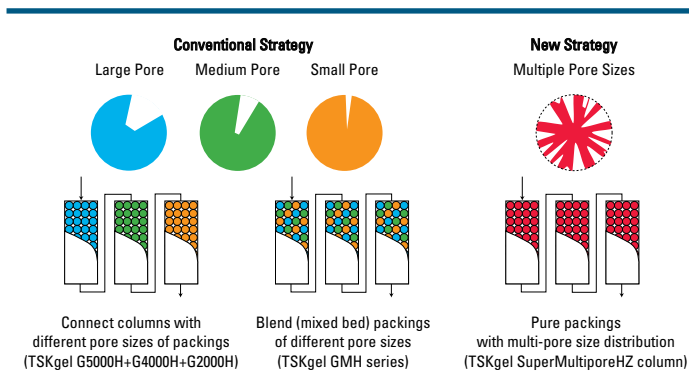
- TSKgel SuperMultiporeHZ-N
- TSKgel SuperMultiporeHZ-M
- TSKgel SuperMultiporeHZ-H

About: Multi-pore Technology

Prior to the introduction of TSKgel SuperMultiporeHZ columns, scientists separating polymers with a wide range of molar masses were left with two options. One option was to use multiple columns of different pore sizes linked together in series. A second was to use a column packed with a mixed bed resin of different pore sizes at an optimized mix ratio. However, problems can occur with both of these methods, which include distortion of the chromatogram or deviations between the actual calibration curve and the calibration curve approximated from data obtained from the molar mass standards.

As is shown in Figure 112, a novel approach to solve this problem was developed by Tosoh scientists and is incorporated in TSKgel SuperMultiporeHZ columns. Small particles of uniform size are synthesized with a broad distribution of pore sizes. This novel approach creates a linear calibration curve within each particle. Therefore, columns with an extended linear calibration curve can now be prepared without mixing particles of different pore sizes.

Figure 112: Graphical representations illustrate the multi-pore particle synthesis technology



Attributes and Applications:

Product attributes for the TSKgel SuperMultiporeHZ columns are listed in Table 30. Table 31 lists features and benefits of these columns. TSKgel SuperMultiporeHZ columns can be utilized for the analysis of polymers with a wide MM distribution range. The columns are shipped in THF, which cannot be replaced for any other organic solvent. Figure 113 shows the calibration curves for the TSKgel SuperMultiporeHZ columns.

Table 30: Product attributes

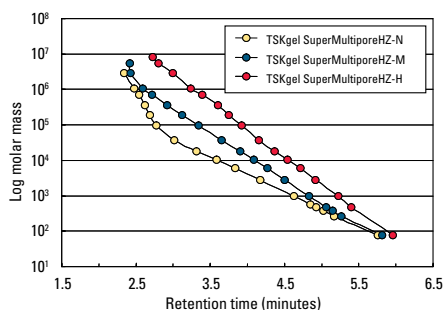
TSKgel column	SuperMultipore HZ-N	SuperMultipore HZ-M	SuperMultipore HZ-H
Base material	PS-DVB	PS-DVB	PS-DVB
Particle size	3 μm^*	4 μm^*	6 μm^*
Pore size	8 nm	14 nm	>14 nm
Exclusion limit (PST/THF)	1.2×10^5 Da	2.0×10^6 Da	4.0×10^7 Da
Separation range	300 ~ 5.0×10^4 Da	500 ~ 1.0×10^6 Da	1,000 ~ 1.0×10^7 Da
Theoretical plates/15 cm column	20,000	16,000	11,000

* Particle size distribution is monodisperse.

Table 31: Features and benefits

Feature	Benefit
Multi-pore packing material (wide range of pores contained in single particle)	<ul style="list-style-type: none"> • Calibration curves with superior linearity • No observable distortion of chromatograms • Improved accuracy and repeatability of molar mass data • Capable of rapid analysis with high separation performance
Smaller particle size (monodisperse particles)	<ul style="list-style-type: none"> • Capable of achieving the same separation performance as conventional columns (30 cm) in half the analysis time • No reduction in separation performance even for analysis at high flow rates • Improved robustness of column performance
Semi-micro column	<ul style="list-style-type: none"> • Reduced solvent consumption • 1/6th the consumption of conventional (30 cm) columns
Low adsorption packing material	<ul style="list-style-type: none"> • Can be used for a wide variety of samples

Figure 113: Calibration curves for TSKgel SuperMultiporeHZ columns

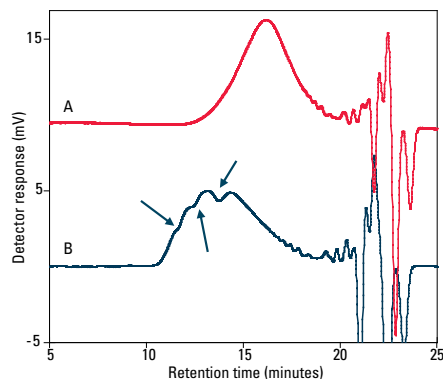


Columns: **TSKgel SuperMultiporeHZ-N, 3 μ m, 4.6 mm ID \times 15 cm**
TSKgel SuperMultiporeHZ-M, 4 μ m, 4.6 mm ID \times 15 cm
TSKgel SuperMultiporeHZ-H, 6 μ m, 4.6 mm ID \times 15 cm
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 $^{\circ}$ C
 Samples: PStQuick polystyrene standards

Acrylic Resin

Figure 114 demonstrates that inflection points are no longer observed with columns packed from particles prepared by multi-pore technology.

Figure 114: Comparison for separation of acrylic resin

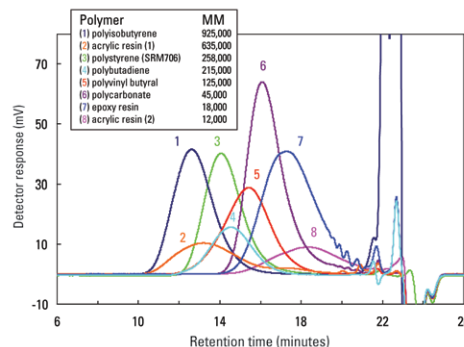


Columns: **A. TSKgel SuperMultiporeHZ-M, 4.6 mm ID \times 15 cm \times 4**
B. TSKgel SuperHZ4000+3000+2500+2000,
4.6 mm ID \times 15cm \times 1
 Mobile phase: THF
 Detection: RI
 Temperature: 40 $^{\circ}$ C
 Injection vol.: 10 μ L
 Samples: acrylic resin

Various Polymers

Various polymers were analyzed on four TSKgel SuperMultiporeHZ-M columns in series. The superimposed chromatograms in Figure 115 clearly demonstrate that these new GPC columns can be utilized for the analysis of polymers with a wide molar mass distribution range.

Figure 115: Separation of various polymers



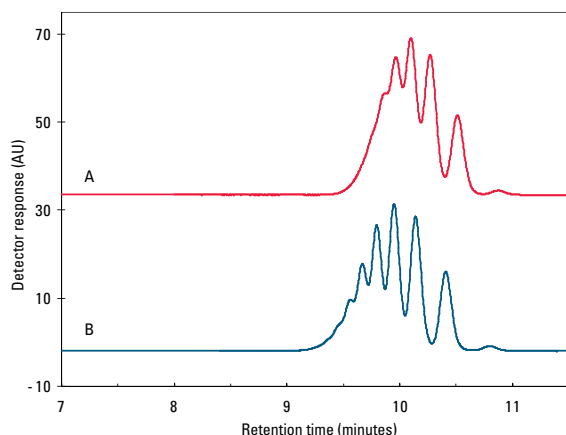
Columns: **SuperMultiporeHZ-M, 4 μ m, 4.6 mm ID \times 15 cm \times 4**
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Detection: RI
 Temperature: 25 $^{\circ}$ C
 Injection vol.: 10 μ L
 Sample conc.: 0.3%



Standard Polystyrene

Figure 116 compares separation on the TSKgel SuperMultiporeHZ-N column versus the TSKgel SuperMultiporeHZ-M column in the low molar mass region (standard polystyrene A-500). The calibration curve for the TSKgel SuperMultiporeHZ-N column is not as steep and better separation is provided in the low molar mass region due to the smaller particle size (higher number of theoretical plates) of the TSKgel SuperMultiporeHZ-N column.

Figure 116: Analysis of standard polystyrene

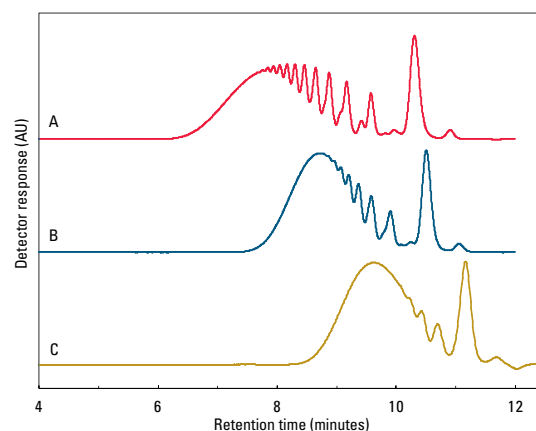


Columns: **A. TSKgel SuperMultiporeHZ-M, 4 μ m, 4.6 mm ID \times 15 cm \times 2**
B. TSKgel SuperMultiporeHZ-N, 3 μ m, 4.6 mm ID \times 15 cm \times 2
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Injection vol.: 5 μ L
 Sample: standard polystyrene oligomer
 (TSKgel standard polystyrene A-500) (5 g/L)

Epoxy Resin

Figure 117 is a chromatogram of an epoxy resin (approximately 6,000 Da) created using the TSKgel SuperMultiporeHZ columns. The best separation performance is shown by the TSKgel SuperMultiporeHZ-N column, the particle size used for low molar mass samples, and it is clear that the TSKgel SuperMultiporeHZ-H column does not provide adequate separation performance.

Figure 117: Analysis of epoxy resin

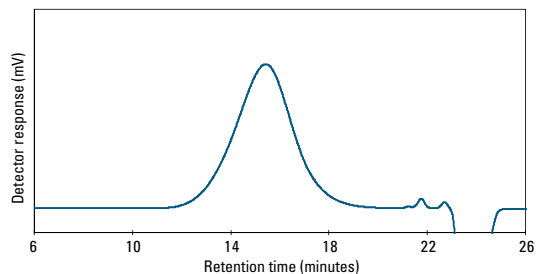


Columns: **A. TSKgel SuperMultiporeHZ-N, 3 μ m, 4.6 mm ID \times 15 cm \times 2**
B. TSKgel SuperMultiporeHZ-M, 4 μ m, 4.6 mm ID \times 15 cm \times 2
C. TSKgel SuperMultiporeHZ-H, 6 μ m, 4.6 mm ID \times 15 cm \times 2
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Detection: UV @ 254 nm
 Temperature: 25 °C
 Injection vol.: 10 μ L
 Sample: epoxy resin (3 g/L)

Polyvinylbutyral

The analysis of polyvinylbutyral using a TSKgel SuperMultiporeHZ-H column is shown in **Figure 118**. A smooth chromatogram without any distortion is obtained.

Figure 118: Analysis of polyvinylbutyral



Column: **TSKgel SuperMultiporeHZ-H, 6 μ m, 4.6 mm ID \times 15 cm \times 4**
 Mobile phase: THF
 Flow rate: 0.35 mL/min
 Detection: RI
 Temperature: 40 $^{\circ}$ C
 Injection vol.: 10 μ L
 Sample: polyvinylbutyral (3 g/L)

