No.088
SEPARATION REPORT

TOSOH

Ultra High Speed and Performance Semi-micro GPC Columns: TSKgel H-Type SuperH Series

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1. Introduction

With advancements in polymer science, a wide variety of new polymer substances have been created. Today, with the multifaceted ways in which products engineered from these substances are now used, it is important to thoroughly understand the properties of these polymer substances from the perspective of usage, development and quality control.

It goes without saying that average molecular weight and molecular distribution are among the properties of polymer substances that are most important to understand. A number of methods have been used over the years for determining average molecular weight and molecular weight distribution including ebulliometry, light scattering techniques, osmometry, viscosity, and absolute methods such as ultracentrifugation. Currently, size exclusion chromatography (SEC), a type of liquid chromatography, is widely used instead of these methods because SEC is relatively simple and quick, and has good repeatability.

Gel filtration chromatography (GFC) using polysaccharide gels was first used in the field of biopolymers, as a method of size exclusion in which molecules are separated based on their size. On the other hand, J.C. Moore[1)] used gel permeation chromatography in an organic solvent system with a cross-linked polystyrene gel to analyze synthetic polymers in 1964, and since that time the use of SEC has been rapidly developing.

Since 1971, when TOSOH began developing and marketing the TSKgel S type series of packed columns for GPC in organic solvent systems, as shown in Table 1, the high performance and speed of our columns has been continuously improving.

In 1992, TOSOH developed the HR series of TSKgel H-type columns (hereafter referred to as the H_{HR} series), which had superior durability and an ability to withstand solvent conversion that surpassed that of previous models. Today, the matrix particle size in the H_{HR} series has been reduced to the microparticle level, realizing a series of highly durable, ultra high performance semi-micro GPC columns: the TSKgel H-type SuperH series (hereafter SuperH series) of GPC columns. Moreover, in response to environmental concerns, this series of columns was developed to reduce organic solvent consumption.

In this report, the features and basic characteristics of the TSKgel SuperH Series are introduced together with examples of their application in analyses.

2. Features

The packing material in the TSKgel H_{HR} series has been modified and reduced to 3 μm in the TSKgel SuperH series, and packed into a stainless steel column (6.0 mm I.D. x 15 cm) using advanced packing technology.

Consequently, the SuperH series maintains the same pore characteristics of the H_{HR} series, simplifies the ability to convert between various types of solvents, and realizes a 2-fold improvement over the H_{HR} series in the number of theoretical plates per unit length.

As a result, the SuperH series possesses the following features:

(1) Similar separation performance as the conventional H_{HR} and H_{XL} series can be obtained in half the time.
(2) As an ultra high performance semi-micro column, the relative sensitivity is improved 3- to 4-fold over conventional columns.
(3) Solvent consumption is reduced to 1/3 that of conventional columns, which can play a major role in reducing expensive solvent running costs and waste treatment costs.
(4) Due to the very small particle size of the packing material, separation performance depends very little on flow rate, and especially in the high-flow-rate range, there is very little reduction in separation performance.
(5) Has the same ability to withstand conversion between solvents as the H_{HR} series, with outstanding ability to convert to various organic solvents with stability and robustness.
(6) Mixed bed columns with superior calibration curve linearity are prepared in 4 grades, as the appropriate column can be selected based on the molecular weight and molecular weight distribution of the sample, to allow efficient analysis to be conducted.

Table 2 shows a comparison of the performance characteristics of the SuperH and H_{HR} series. Calibration curves of the TSKgel SuperH series are shown in Figures 1 and 2 in analyses conducted using standard polystyrene in THF solvent.

Table 1 Steps in the development and commercialization of packed columns for GPC using organic solvent systems

Year	Product name	Column length (cm)	Particle size (μm)	Number of theoretical plates (plates/30 cm)
1971	TSKgel S Type	120	40	1,500
1972	TSKgel H Type	60	10 13	8,000 6,00
1983	TSKgel H_{XL} Series	30	5 13	16,000 8,000
1987	TSKgel H_{XL} New Series	30	5 10	16,000 14,000
1992	TSKgel H_{HR} Seriesne	30	5 13	16,000 8,000
1993	TSKgel SuperH Series	15	3	32,000

Table 2 Comparison of performance of TSKgel SuperH and H_{HR} Series

SuperH series			H_{HR} series		
Grade	Particle size (μm)	Guaranteed number of theoretical plates (TP/15 cm)	Grade	Particle size (μm)	Guaranteed number of theoretical plates (TP/30 cm)
SuperH1000	3	16,000	G1000 H_{HR}	5	16,000
SuperH2000	3	16,000	G2000 H_{HR}	5	16,000
SuperH2500	3	16,000	G2500 H_{HR}	5	16,000
SuperH3000	3	16,000	G3000 H_{HR}	5	16,000
SuperH4000	3	16,000	G4000 H_{HR}	5	16,000
SuperH5000	3	16,000	G5000 H_{HR}	5	16,000
SuperH6000	5	10,000	G6000 H_{HR}	5	16,000
SuperH7000	5	10,000	G7000 H_{HR}	5	16,000
SuperHM-L	3	16,000	GMH_{HR}-L	5	16,000
SuperHM-N	3	16,000	GMH_{HR}-N	5	16,000
SuperHM-M	3	16,000	GMH_{HR}-M	5	16,000
SuperHM-H	3	16,000	GMH_{HR}-H	5	16,000

Conditions for measuring number of theoretical plates

Columns: SuperH (6.0 mm I.D. x 15 cm)
H_{HR} (7.8 mm I.D. x 30 cm)

Eluent: Tetrahydrofuran (THF)

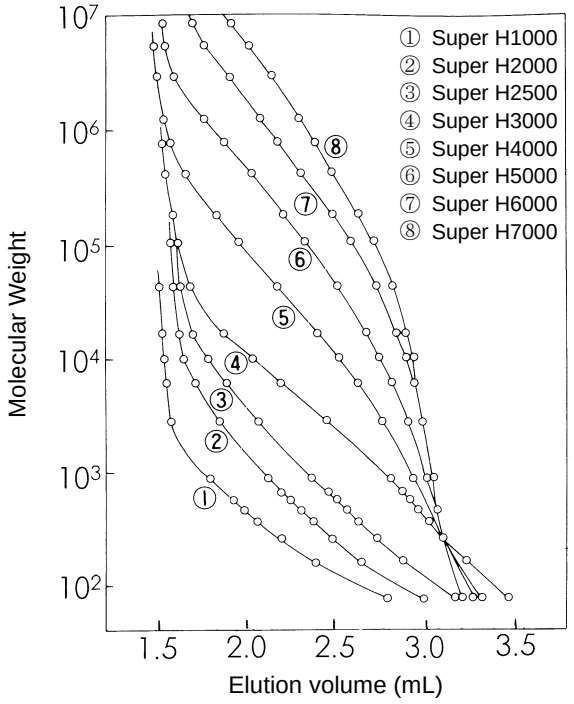
Flow rate: SuperH (0.6mL/min)
H_{HR} (1.0mL/min)

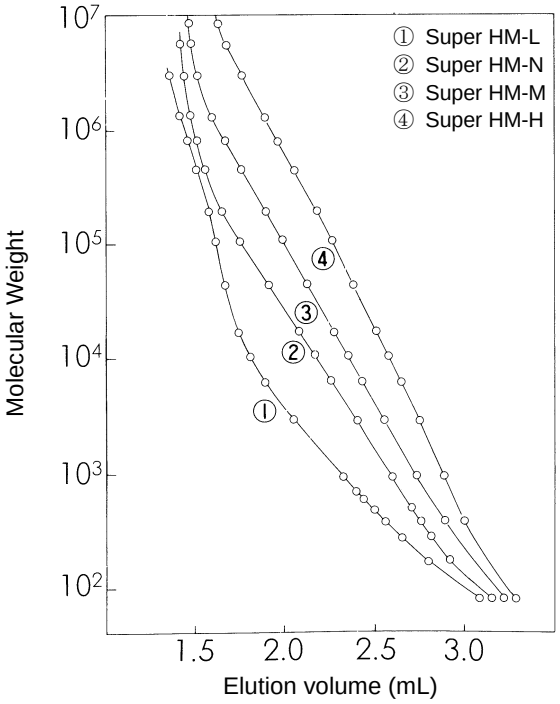
Temperature: 25 ºC

Detection: UV (254 nm)

Sample: SuperH100 (p-hydroxybenzyl alcohol)
SuperH2000 - H7000 and SuperHM (dicyclohexyl phthalate)
G1000 H_{HR}-G2500 H_{HR} (benzene)
G3000 H_{HR}-G4000 H_{HR}, GMH_{HR}-L and -M (n-butylbenzene)
G5000 H_{HR}-G7000 H_{HR}, GMH_{HR}-M and -H (dicyclohexyl phthalate)

Column	:	TSKgel SuperH Series 6.0 mm I.D. x 15 cm
Eluent	:	THF
Flow rate	:	0.6 mL/min
Temperature	:	25 ºC
Detection	:	UV (254 nm)
Sample	:	Standard polystyrene





3. Basic characteristics

3-1. Separation performance

As shown in Table 2, the particle size of the packing material of the SuperH1000-SuperH5000, SuperHM-L, SuperHM-N, SuperHM-M and SuperHM-H columns has been reduced from 5 μm to 3 μm, in comparison to the particle size of the conventional H_{HR} series of columns. This results in a 2-fold increase in the number of theoretical plates per unit length versus the H_{HR} series. Consequently, the SuperH series can achieve the same separation performance as the H_{HR} series in half the analysis time.

Fig. 3 compares chromatograms of epoxy resin separated using the SuperH3000 and G3000 H_{HR} columns, and Fig. 4 compares chromatograms of a mixed standard polystyrene sample separated using the SuperHM-H, GMH_{HR}-H and GMH_{XL} columns. These figures clearly show that the SuperH series can achieve the separation performance of the conventional H_{HR} and H_{XL} series of columns in half the analysis time.

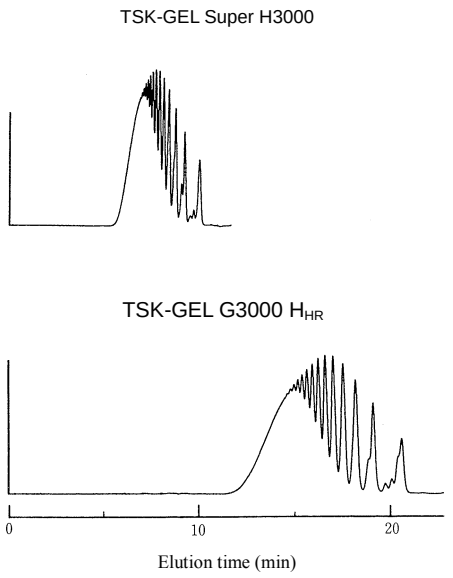


Fig. 3 Separation of epoxy resin using TSKgel SuperH3000 and TSKgel G3000 H_{HR} columns

Columns: TSKgel SuperH3000 (6.0 mm I.D. x 15 cm x 2)
TSKgel G3000 H_{HR} (7.8 mm I.D. x 30 cm x 2)
Eluent: THF
Flow rate: SuperH3000 (0.6 mL/min)
G3000 H_{HR} (1.0 mL/min)
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Epoxy resin

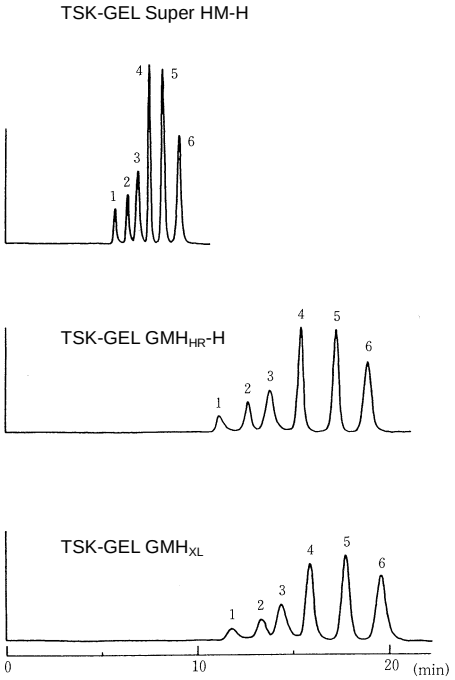


Fig. 4 Separation of polystyrene mixture using TSKgel SuperHM-H and conventional column

Columns: TSKgel SuperHM-H
(6.0 mm I.D. x 15 cm x 2)
TSKgel GMH_{HR}-H, GMH_{XL}
(Both: 7.8 mm I.D. x 30 cm x 2)
Eluent: THF
Flow rate: SuperHM-H (0.6 mL/min)
GMH_{HR}-H, GMH_{XL} (1.0 mL/min)
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene
1. MW 8,420,000 2. MW 1,260,000 3. MW 422,000
4. MW 107,000 5. MW 16,700 6. MW 2,800

3-2. Flow-rate dependence of height equivalent to theoretical plate (HETP)

3-2-1. Flow-rate-dependence of HETP with low molecular weight samples

The effect of flow rate on height equivalent to theoretical plate (HETP) depends a great deal on the particle size of the packing material, sample type and molecule size, solvent type and viscosity, and temperature.

Figure 5 compares the flow-rate dependence of HETP in the SuperHM-H column versus conventional columns using dicyclohexyl phthalate (DCHP) as the sample, and Figure 6 compares the flow-rate dependence of HETP with the SuperH25000 (sample: DCHP) versus the G2500 H_{XL} and G2500 H_{HR} (sample: benzene).

It is clear that the dependence of HETP on flow rate decreases as the particle size of the packing material becomes smaller. Particularly in conventional columns, the flow-rate dependence of the HETP is very high in the high-flow-rate region (the region in which linear velocity ≥ 0.045 cm/sec and flow rate ≥ 1.2 mL/min). In SuperH columns, however, in which the particle size of the packing material has been reduced, HETP depends very little on flow rate. As a result, when a low molecular weight sample such as DCHP is analyzed, the analysis time can be decreased by increasing the flow rate (linear velocity: 0.07 cm/sec, flow rate: 1.2 mL/min).

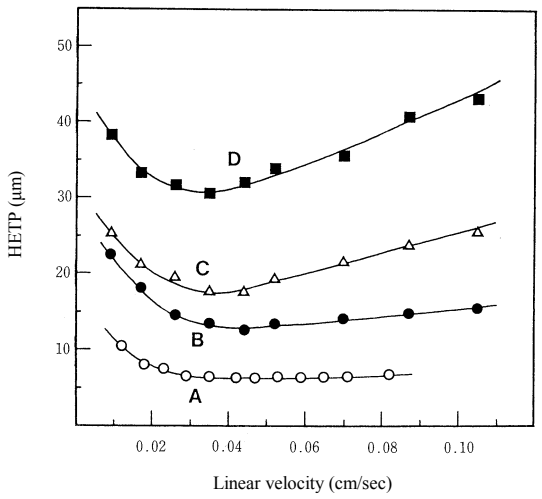


Fig. 5 Relationship between HETP and flow rate in SuperH and conventional columns

Columns: A. TSKgel SuperHM-H 6 mm I.D. x 15 cm
B. TSKgel GMH_{HR}-H 7.8 mm I.D. x 30 cm
C. TSKgel GMH_{XL} 7.8 mm I.D. x 30 cm
D. TSKgel GMH_{HR}-H(S) 7.8 mm I.D. x 30 cm
Eluent: THF Temp.: 25 ºC Detection: UV (254 nm)
Samples: Dicyclohexyl phthalate (DCHP) (0.1%)
A: 3 μL; B, C, and D: 20 μL

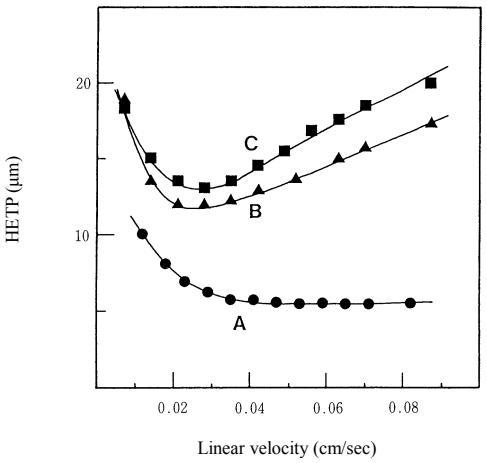


Fig. 6 Relationship between HETP and flow rate in SuperH and conventional columns

Columns: A. TSKgel SuperH2500 6 mm I.D. x 15 cm
B. TSKgel G2500 H_{HR} 7.8 mm I.D. x 30 cm
C. TSKgel G2500 H_{XL} 7.8 mm I.D. x 30 cm
Eluent: THF
Temp.: 25 ºC
Detection: UV (254 nm)
Samples: A: DCHP (0.1%), 3 μL
B, C: Benzene (0.1%), 20 μL

Figure 7 shows the relationship between flow rate and separation of standard polystyrene A-500 in chromatograms produced by SuperH2500 and G2500 H_{XL} columns. With low molecular weight samples such as A-500, the separation performance of the SuperH2500 is essentially independent of flow rate, as a high level of separation is maintained, even in the high-flow-rate region. On the other hand, with the G2500 H_{XL}, separation performance decreases as flow rate increases.

3-2-2. Flow-rate dependence of HETP with polymer samples

Fig. 8 shows the flow-rate dependence of HETP in a SuperHM-H column with standard polystyrenes used as samples.

With low molecular weight samples, no flow rate dependence of HETP was observed in the high flow rate region, but when a polymer sample is used, HETP increases with the flow rate, confirming the dependency of HETP on flow rate. This effect increases with increases in molecular weight.

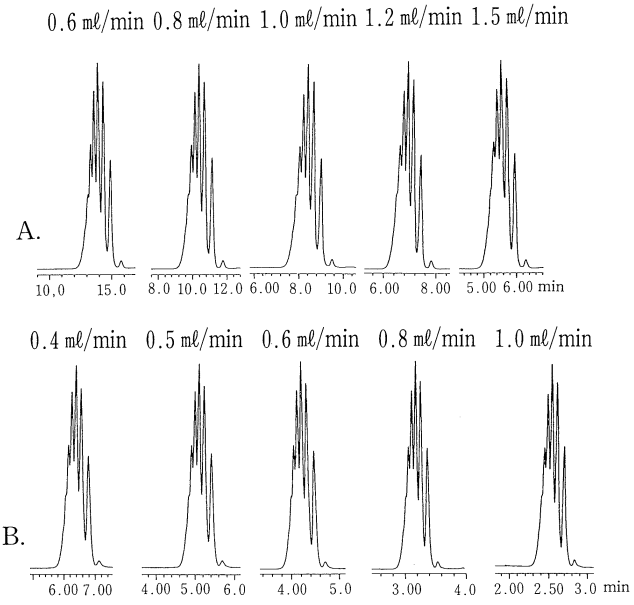


Fig. 7 Flow rate dependence of separation of standard polystyrene in TSKgel SuperH2500 and G2500 H_{XL} columns

Columns: A. TSKgel G2500H_{XL} 7.8 mm I.D. x 30 cm
B. TSKgel SuperH2500 6 mm I.D. x 15 cm
Eluent: THF
Temp.: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene A-500 (0.1%), 10 µL

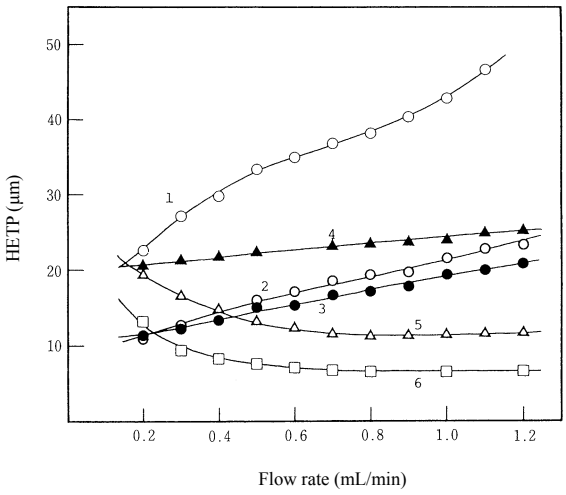


Fig. 8 Relationship between HETP and flow rate at various molecular weights in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Temp.: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene
1. MW 1,260,000 (○)
2. MW 107,000 (○) 3. MW 16,700 (●)
4. MW 2,800 (▲) 5. MW 500 (△)
6. DCHP (□)

Fig. 9 shows the relationship between flow rate and separation performance of standard polystyrene in the SuperHM-H. Separation is clearly dependent on flow rate, as separation performance decreases as the flow rate increases. Thus when polymer samples are analyzed, the lower the flow rate, the better the separation performance.

Fig. 10 shows the flow-rate dependence of separation of epoxy resin in chromatograms from SuperH columns and Fig. 11 shows the flow-rate dependence of separation of standard polystyrenes in chromatograms from the SuperHM-H column. In SuperH columns, separation performance depends little on flow rate in comparison to the conventional H_{HR} and H_{XL} series, making analysis at high flow rates possible. However, better separation performance is obtained at low flow rates when polymer samples are analyzed with this column.

Consequently, when analyzing a polymer sample with the SuperH series, the flow rate should be between 0.3 and 0.6 mL (equivalent to 0.5 to 1.0 mL with conventional H_{HR} and H_{XL} series columns), and when analyzing oligomers and low molecular weight samples, the flow rate should be about 0.6 mL/min.

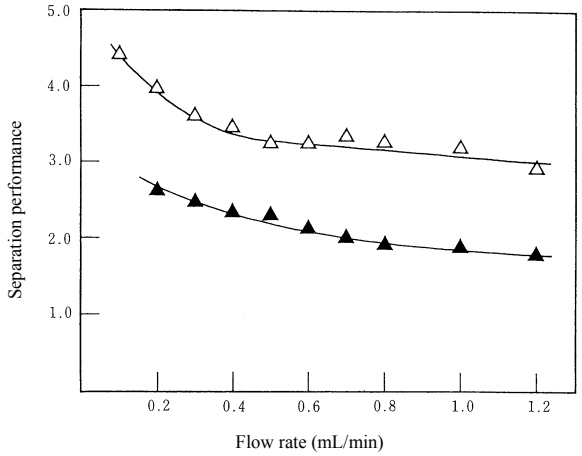


Fig. 9 Flow rate dependence in separation of standard polystyrene with SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm x 2
Eluent: THF
Temp.: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene
Separation performance:
△: F-550 (0.02%)/F-80 (0.022%)
▲: F-40 (0.035%)/F-10 (0.05%)
F-550 (MW: 5,480,000)
F-80 (MW: 706,000)
F-40 (MW: 422,000)
F-10 (MW: 107,000)

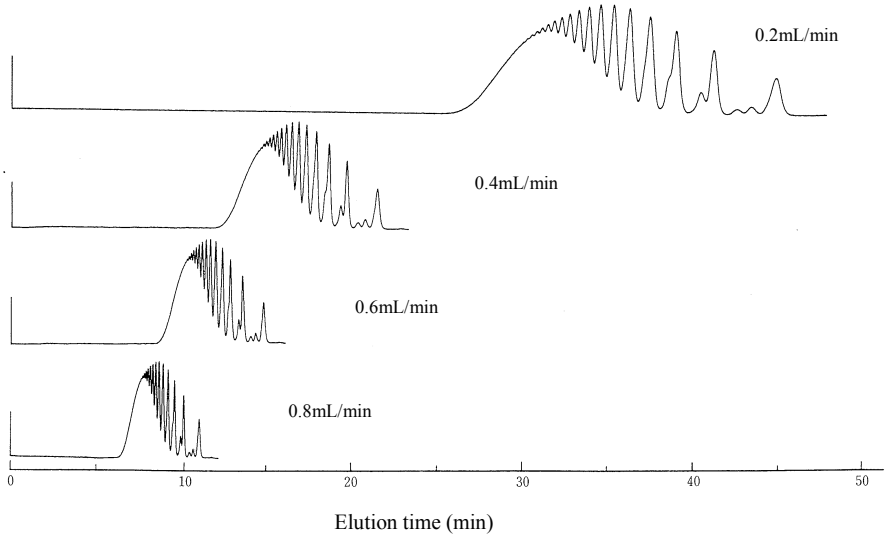


Fig. 10 Flow rate dependence of separation of epoxy resin in SuperH columns

Column: TSKgel SuperH3000 x 2 + TSKgel SuperH2500
6 mm I.D. x 15 cm x 3

Eluent: THF Temp.: 25 ºC Detection: UV (254 nm)

Sample: Epikote 1004 (0.1%), 10 µL

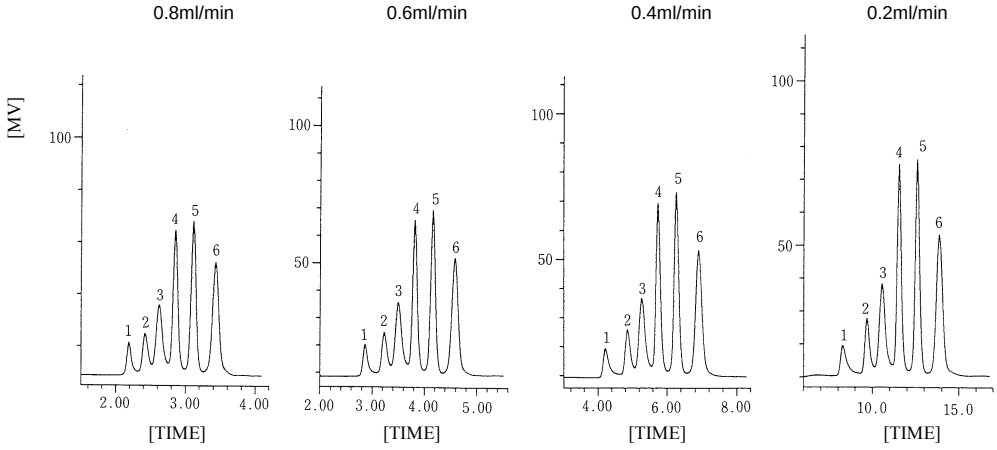


Fig. 11 Flow rate dependence of separation of standard polystyrene in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6mm I.D. x 15 cm

Eluent: THF Temp.: 25 ºC Detection: UV (254 nm)

Sample: Standard polystyrene

1. MW 8,420,000 2. MW 1,260,000 3. MW 422,000
4. MW 107,000 5. MW 16,700 6. MW 2,800

3-3. Peak detection sensitivity

The size of the SuperH series has been reduced from 7.8 mm I.D. x 30 cm to 6.0 mm I.D. x 15, to create a semi-micro column with ultra high performance capability. As a result, the detection sensitivity of the SuperH columns for sample peaks is relatively higher than that of conventional H_{HR} and H_{XL} series of columns.

Fig. 12 shows the relationship between the peak height and sample injection volume for standard polystyrene A-500 in the SuperH2500 and G2500 H_{XL} columns. Fig. 13 shows the relationship between peak height and injection volume for polystyrene using the SuperHM-H and GMH_{HR}-H columns.

Fig. 14 compares chromatograms of the separation of standard polystyrene A-500 in the SuperH2500 and G2500 H_{XL} columns. Fig. 15 and Fig. 16 compare the chromatograms of the separation of a standard polystyrene mixture and commercial polystyrene using the SuperHM-H GMH_{HR}-H and GMH_{XL} columns.

It is clear from these figures that the detection sensitivity is increased 3- to 4-fold relative to that of conventional columns. In other words, the sample input volume (absolute load) can be decreased 1/3 to 1/4 compared to the quantity used with conventional columns.

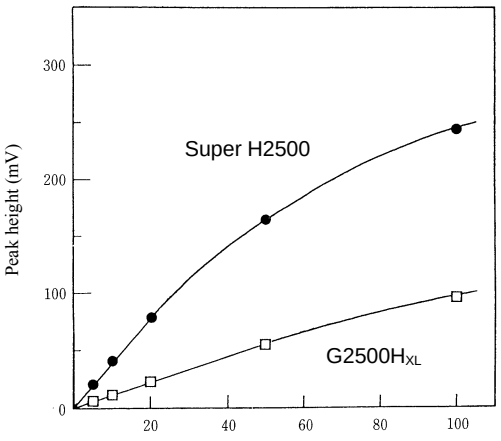


Fig. 12 Relationship between peak height and injection volume of polystyrene using TSKgel SuperH2500 and G2500H_{XL}

Columns: TSKgel SuperH2500
(6.0 mm I.D. x 15 cm x 2)
G2500H_{XL} (7.8 mm I.D. x 30 cm x 2)
Eluent: THF
Flow rate: TSKgel SuperH2500 (0.6 mL/min)
TSKgel G2500H_{XL} (1.0 mL/min)
Sample: Standard polystyrene A-500

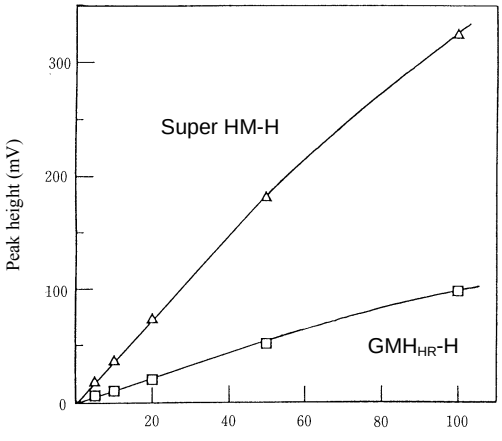


Fig. 13 Relationship between peak height and injection volume of polystyrene using TSKgel SuperHM-H and GMH_{HR} columns

Columns: TSKgel SuperHM-H
(6.0 mm I.D. x 15 cm x 2)
GMH_{HR}-H (7.8 mm I.D. x 30 cm x 2)
Eluent: THF
Flow rate: TSKgel SuperHM-H (0.6 mL/min)
TSKgel GMH_{HR}-H (1.0 mL/min)
Sample: Commercial polystyrene (0.5%)

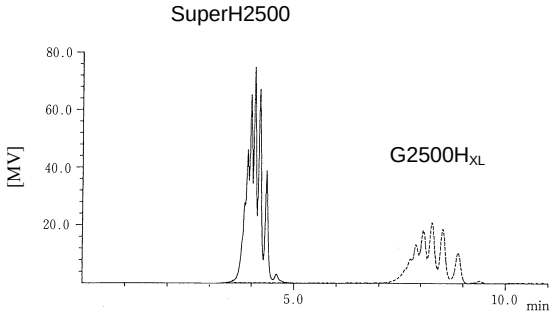


Fig. 14 Comparison of peak heights of standard polystyrene (A-500) produced by TSKgel SuperH2500 and conventional columns (G2500H$_{XL}$)

Columns: TSKgel SuperH2500 6 mm I.D. x 15 cm
TSKgel G2500H$_{XL}$ 7.8 mm I.D. x 30 cm
Eluent: THF
Flow rate: TSKgel SuperH2500 :0.6 mL/min
TSKgel G2500H$_{XL}$:1.0 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene (A-500), 0.1%, 10 µL

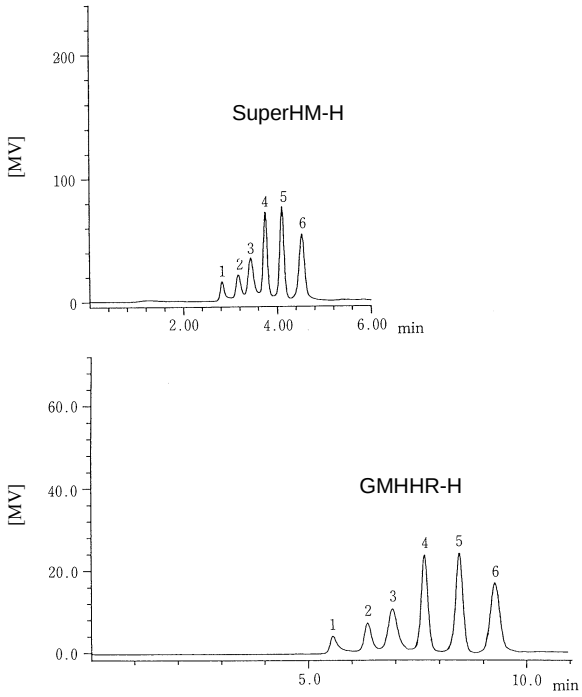


Fig. 15 Separation of standard polystyrenes by TSKgel SuperHM-H and GMH$_{HR}$-H

Columns: TSKgel SuperHM-H 6 mm I.D. x 15 cm
TSKgel GMH$_{HR}$-H 7.8 mm I.D. x 30 cm
Eluent: THF
Flow rate: TSKgel SuperHM-H :0.6 mL/min
TSKgel GMH$_{HR}$-H :1.0 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene, 10 µL

1. MW 8,420,000, (0.02%)
2. MW 1,260,000 (0.035%)
3. MW 422,000 (0.06%)
4. MW 107,000 (0.09%)
5. MW 16,700 (0.1%)
6. MW 2,800 (0.1%)

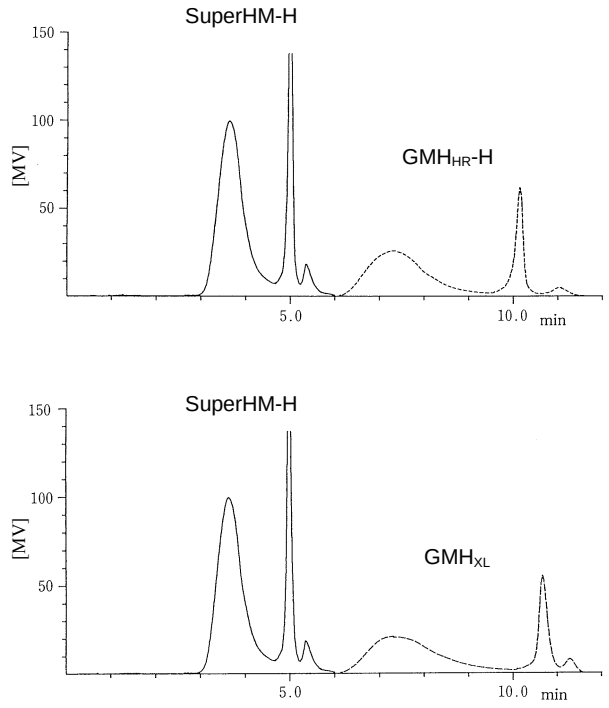


Fig. 16 Comparison of commercial polystyrene peak heights produced by TSKgel SuperHM-H and conventional columns

Columns: TSKgel SuperHM-H 6 mm I.D. x 15 cm
TSKgel GMH$_{HR}$-H, TSKgel GMH$_{XL}$
Both: 7.8 mm I.D. x 30 cm
Eluent: THF
Flow rate: TSKgel SuperHM-H: 0.6mL/min
Others: 1.0 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Commercial polystyrene (0.25%), 20 µl

3-4. Solvent compatibility

Because the SuperH series uses the same smaller size particles for the packing material used in the H_{HR} series, it is expected that shrinkage and swelling of the packing materials occurring with various solvents will be equivalent to the properties of the H_{HR} series.

Table 3 compares shrinkage and swelling properties of the TSKgel SuperH2000, G2000 H_{HR} and G2000H_{XL} with various organic solvents. Based on this data it is clear that the solvent can be converted from the initially loaded solvent (THF) to virtually any of the organic solvents shown in Table 4.

Table 3 Comparison of shrinkage and swelling properties of TSKgel SuperH2000, G2000 H_{HR} and G2000 H_{XL}

Solvent	Shrinkage/swelling		
	SuperH2000	G2000H_{HR}	G2000H_{XL}
Toluene	1.00	1.01	1.06
Benzene	1.01	1.00	——
THF	1.00	1.00	1.00
Dimethylformamide (DMF)	1.00	0.99	0.86
Acetone	0.99	0.99	0.86
Methanol (MeOH)	0.98	0.98	0.67
THF/water = 1/1	0.97	0.98	——
MeOH/water = 1/1	0.92	0.93	——
Water	0.85	0.86	0.52

*Shrinkage/swelling occurring with various organic solvents based on shrinkage volume with THF of 1.00.

Table 4 Solvents that can be converted in TSKgel SuperH series

Toluene, xylene, chloroform ($CHCl_3$), benzene, dichloromethane, dichloroethane, dimethylformamide (DMF), dimethylsulfoxide (DMSO), dioxane, N-methylpyrrolidone (NMP), m-cresol/$CHCl_3$, quinoline, methyl ethyl ketone (MEK), o-dichlorobenzene (ODCB), trichlorobenzene (TCB), hexafluoroisopropanol (HFIP), HFIP/$CHCl_3$, o-chlorophenol (OCP), OCP/$CHCl_3$, pyridine, carbon tetrachloride, ethyl acetate, methanol (MeOH), MeOH/$CHCl_3$, THF/MeOH, acetone, ethanol, dimethylacetamide, n-hexane, dodecane, 1-chloronaphthalene, FC-113, trichloroethane

Figure 17 shows the changes resulting when solvents in SuperH columns (SuperH2000, SuperH3000, and SuperHM-H) were directly converted from THF to an organic solvent (from toluene to ethanol), expressed as the ratio of the number of theoretical plates with THF after solvent conversion versus the number of theoretical plates with THF before solvent conversion. In this test, direct conversion from THF to one of a number of organic solvents was performed. After leaving the new solvent in the columns for one week, the solvent was converted back to THF, and then converted again to a new organic solvent. Specifically, with this test method, the changes to the packing properties of the column (column efficiency) were observed under continuous conversion of the solvent to various other organic solvents.

The results show that in each of the SuperH columns there was no change in the number of theoretical plates after conversion to any other organic solvent, which clearly demonstrates that the SuperH series is a very stable and robust series of columns, possessing the same outstanding ability to withstand conversion between solvents as the TSKgel H_{HR} series.

Figure 18 compares chromatograms of standard polystyrene mixtures separated using the SuperH2500 with various organic solvents (THF, $CHCl_3$, DMF, and CCl_4) and Figure 19 compares chromatograms of standard polystyrene mixtures separated using the SuperHM-H with various organic solvents.

Due to the interaction between the packing material and standard polystyrene occurring with DMF as the solvent,[2] the elution volume of standard polystyrene is greater than it is with good solvents such as THF and $CHCl_3$. This effect is particularly noticeable with SuperH2500, a low molecular grade column. Under these circumstances, polyethylene oxide (PEO) is recommended for the standard sample, as this reacts very little with the packing material.

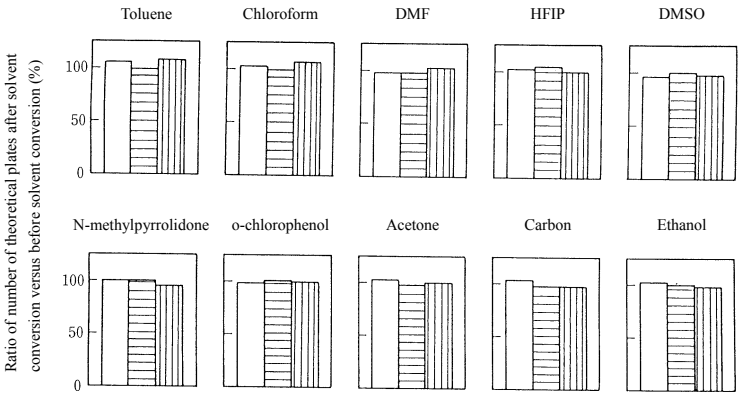


Fig. 17 Solvent compatibility of TSKgel SuperH Series

<Solvent conversion conditions>
Flow rate for conversion to test solvent: 0.2 mL/min
Temperature during conversion to test solvent: 25 ºC
Duration of conversion from THF to test solvent: 16 hours
Time left at rest with test solvent: 1 week
Flow rate, temperature and time elapsed during conversion from test solvent to THF: 0.2 mL/min, 25 ºC and 8 hours

<Conditions for measuring number of theoretical plates>

Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: DCHP (0.1%), 2 µL
TSKgel SuperH2000: □
TSKgel SuperH3000: ▤
TSKgel SuperHM-H: ▥

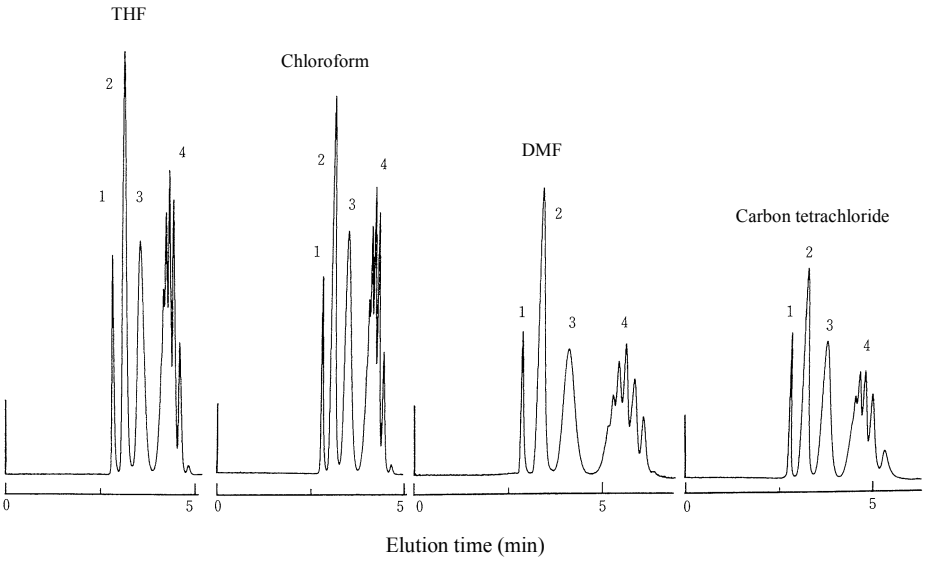


Fig. 18 Separation of standard polystyrenes compared using various solvents with TSKgel SuperH2500

Column: 6.0 mm I.D. x 15 cm
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm or 270 nm)
Sample: 1. MW 190,000
2. MW 9,100
3. MW 2,800
4. A-500

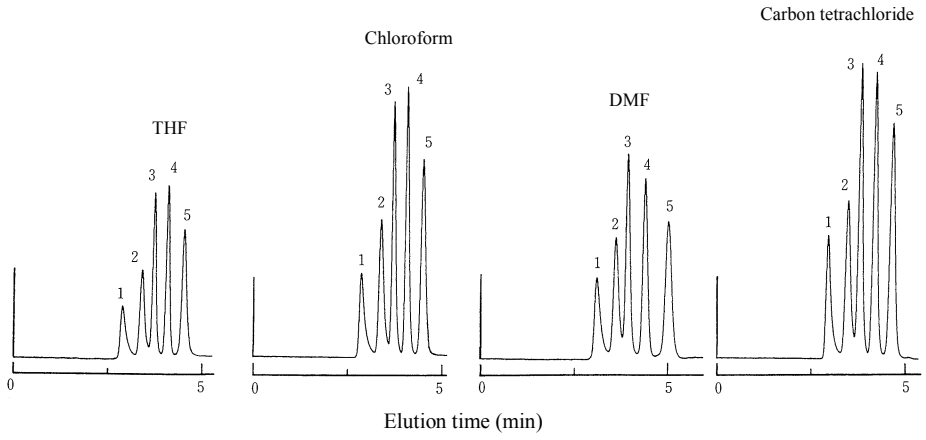


Fig. 19 Separation of standard polystyrenes compared using various solvents with TSKgel SuperHM-H

Column: 6.0 mm I.D. x 15 cm
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm or 270 nm)
Sample: 1. MW 2,890,000 2. MW 422,000
3. MW 107,000 4. MW 16,700
5. MW 2,800

3-5. Sample load

Analysis conditions include some factors involved in maximizing performance of ultra high performance semi-micro columns represented by the SuperH series, and the most important of these conditions is the sample load (sample injection volume and sample concentration).

Sample load depends on sample molecular weight and type, mobile phase, flow rate, temperature, column size, and particle size of packing material. In particular, sample load decreases as the viscosity and molecular weight of the sample increases, and in high performance columns the sample load decreases with particle size. Consequently, to analyze molecular weight distribution with good repeatability at a high degree of separation, it is important to thoroughly understand the sample load in the column being used.

3-5-1. Sample concentration

Fig. 20 shows the relationship between sample concentration, sample load and HETP of various standard polystyrenes using the SuperHM-H. Fig. 21 shows the relationship between sample concentration, sample load and elution volume of various standard polystyrenes using the SuperHM-H. The maximum sample concentration and sample load will differ depending on the standard polystyrene used, and the dependence of HETP and elution volume on sample concentration and sample load will increase as the molecular weight of the sample increases. Table 5 shows the maximum sample concentration and maximum sample load for various standard polystyrenes, based on the results shown in Figures 20 and 21.

Table 5 Maximum sample concentrations and sample loads in SuperH columns

Molecular weight	Maximum sample concentration (%)	Maximum sample load (μg)
～ 10,000	2.0	200
10,000～ 50,000	1.0	100
50,000～ 200,000	0.2	20
200,000～ 500,000	0.1	10
500,000～ 1,000,000	0.05	5
1,000,000～ 5,000,000	0.02	2
5,000,000～	0.01	1

Column: 6.0 mm I.D. x 15 cm
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample: Standard polystyrene, 10 μL

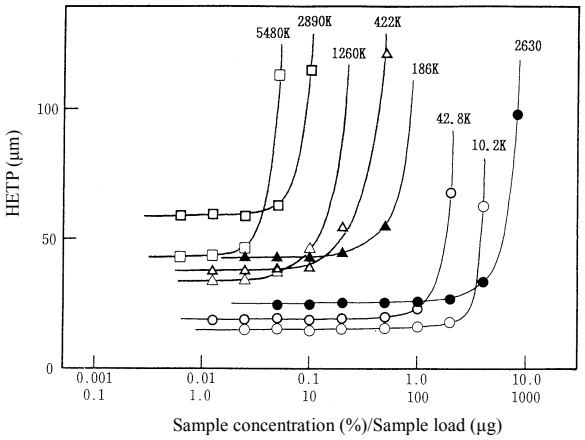


Fig. 20 Relationship between sample concentration (sample load) and HETP for standard polystyrenes in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample: Standard polystyrene, 10 μL

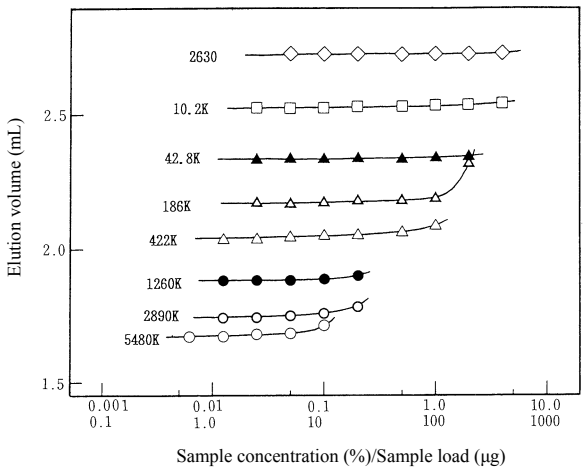


Fig. 21 Relationship between sample concentration (sample load) and elution volume for standard polystyrenes in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample: Standard polystyrene, 10 μL

3-5-2. Sample injection volume

Fig. 22 shows the relationship between sample injection volume, sample load and HETP for various standard polystyrenes using the SuperHM-H and Fig. 23 shows the relationship between sample injection volume, sample load and elution volume for various standard polystyrenes using the SuperHM-H. The dependence of HETP and elution volume on sample concentration and sample load increases with the injection volume. However, unlike sample concentration, there is essentially no correlation between the sample injection volume and molecular weight, and it is understood that the maximum injection volume is 20 µL (20 µg).

Moreover, in this column, the maximum sample injection volume is essentially unrelated to the molecular weight, since as the sample injection volume reaches 20 µL (2 µg), HETP will depend largely on the sample injection volume. One conventional method proposed for increasing the sample load is to decrease the sample concentration while increasing the sample injection volume.[3,4] However, the method used to increase the maximum sample load in SuperH columns is to decrease the sample injection volume (≤ 10 µL) and increase the sample concentration up to the maximum concentration (see Table 5).

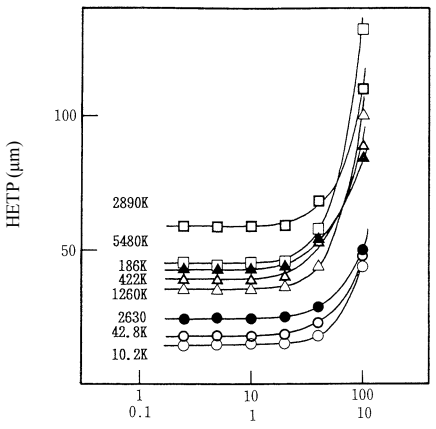


Fig. 22 Relationship between sample injection volume (sample load) and HETP for standard polystyrenes in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene, 10 µL
Sample concentration: 0.01%

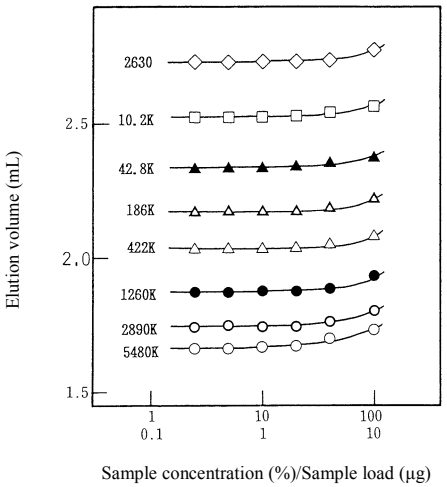


Fig. 23 Relationship between sample concentration (sample load) and elution volume for standard polystyrenes in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene (10 µL)
Sample concentration: 0.01%

Fig. 24 shows the relationship between sample injection volume and separation performance for standard polystyrenes with molecular weights of 8,420,000 and 1,260,000 using 1 to 4 SuperHM-H columns. It is clear that the effect of the standard injection volume on separation performance decreases as the number of columns is increased.

Figs. 25 to 27 show how separation performance is dependent on the sample injection volume in chromatograms of a standard polystyrene mixture produced with 1, 2, and 4 SuperHM-H columns.

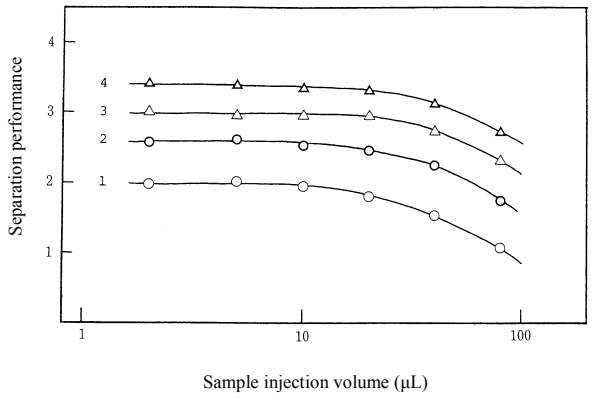


Fig. 24 Relationship between separation performance and sample load of standard polystyrenes in TSKgel SuperHM-H columns

Columns: TSKgel SuperHM-H 6 mm I.D. x 15 cm (○)
6 mm ID x 15 cm x 2 (○)
6 mm ID x 15 cm x 3 (△) 6 mm ID x 15 cm x 4 (△)
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Samples: Standard polystyrene MW 8,420,000 (0.02%)
MW 1,260,000 (0.035%) 10μL

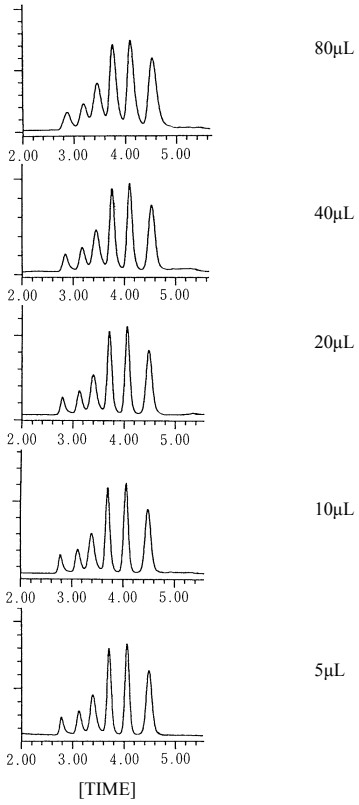


Fig. 25 Effect of sample load on separation of standard polystyrene using TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene mixture

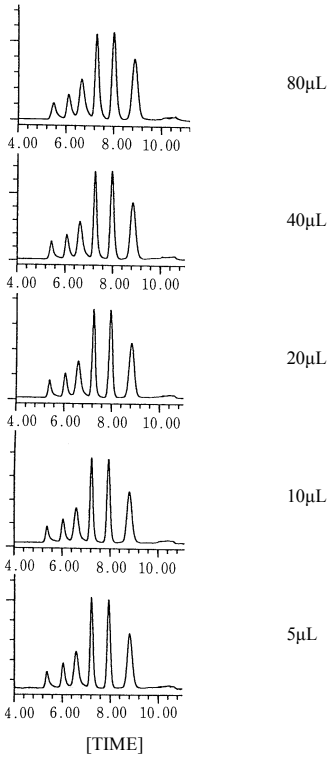


Fig. 26 Effect of sample load on separation of standard polystyrene using TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm x 2
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene mixture

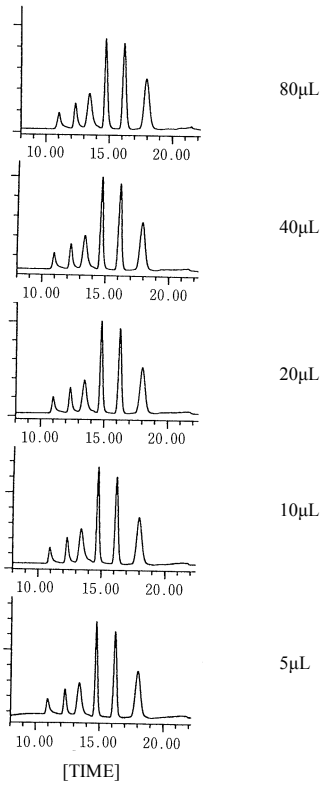


Fig. 27 Effect of sample load on separation of standard polystyrene using TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm x 4
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene mixture

3-6. Shear degradation

Shear degradation is frequently observed, particularly when analyzing very high molecular weight samples, using packing materials composed of very small particles, and when conducting analysis at high flow rates.[5)]

Fig. 28 shows the dependence of shear degradation of standard polystyrene F-2000 (MW: 20,600,000) on flow rate in the SuperHM-H column. With the SuperHM-H column, shear degradation is observed at each flow rate, and elution does not proceed normally.

Therefore to conduct normal GPC analysis of very high molecular weight samples such as F-2000, analysis at flow rates of 0.8 mL/min or less is recommended, using a TSKgel GMH_{HR}-H (S) column (particle size: 13 μm), as shown in Fig. 29.

Fig. 30 shows the dependence of shear degradation of standard polystyrene F-850 (MW: 8,420,000) on flow rate in the SuperHM-H column and Fig. 31 shows the dependence of elution volume on flow rate of various standard polystyrenes in the SuperHM-H. F-850 can be analyzed normally at 0.6 mL/min or less, but shear degradation occurs at high flow rates of 0.8 mL/min and above.

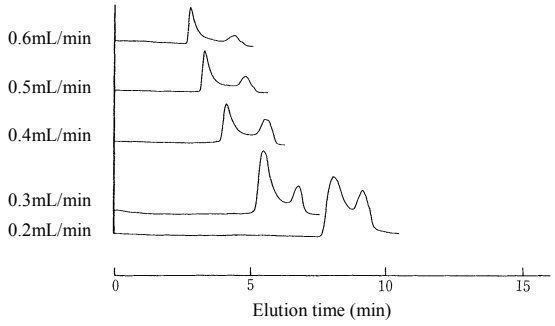


Fig. 28 Dependence of shear degradation on flow rate in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene
F-2000 (MW 20,600,000, 0.015%)

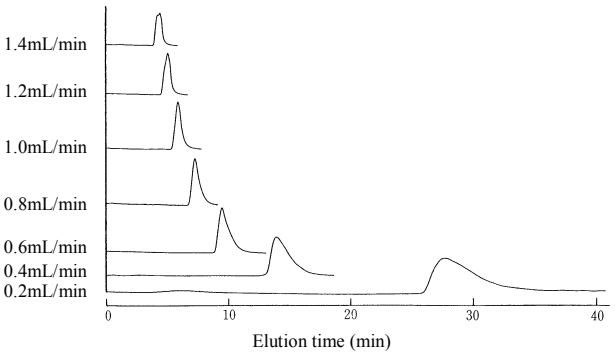


Fig. 29 Dependence of shear degradation on flow rate in TSKgel GMH_{HR}-H (S)

Column: TSKgel GMH_{HR}-H (S) 7.8 mm I.D. x 30 cm
Eluent: THF
Temperature: 25 ºC Detection: UV (254 nm)
Sample: Standard polystyrene
F-2000 (MW 20,600,000, 0.015%)

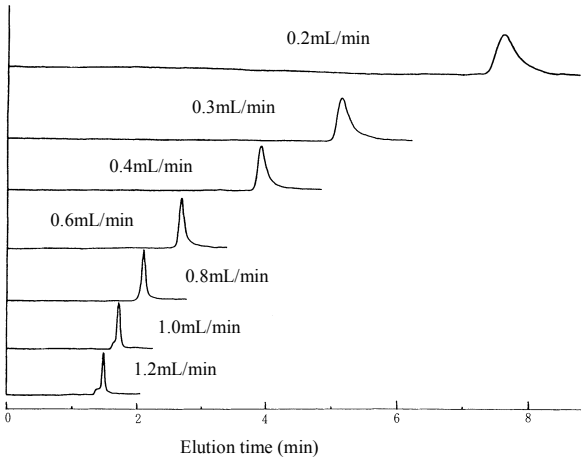


Fig. 30 Dependence of shear degradation on flow rate of standard polystyrene (F-850) in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Temperature: 25 ºC Detection: UV (254 nm)
Sample: Standard polystyrene F-850 (0.01%), 10 μL

Fig. 32 compares the shear degradation of standard polymer F-850 (MW: 8,420,000) in SuperHM-H, SuperHM-M and SuperHM-N columns. As is evident here, shear degradation depends on sample pore size, as this effect becomes stronger the smaller the pore size, and is strongest with the SuperHM-N. Consequently, it is crucial to be aware of the potential for shear degradation when using SuperHM-M and SuperHM-N grade columns (see Table 8).

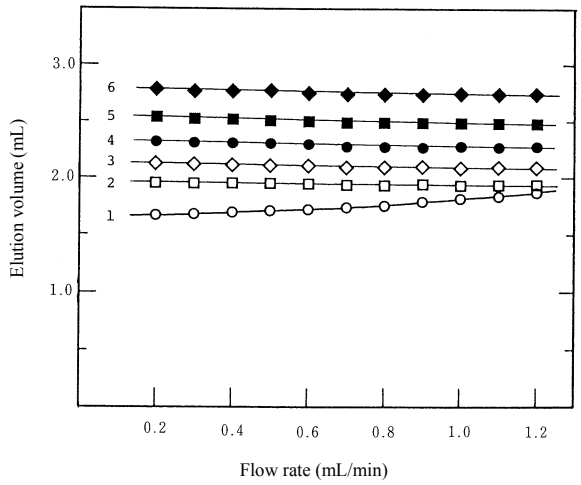


Fig. 31 Relationship between elution volume and flow rate of standard polystyrene in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene
1. MW 8,420,000 (F-850, ○)
2. MW 1,260,000 (F-128, □)
3. MW 422,000 (F-40, ◇)
4. MW 107,000 (F-10, ●)
5. MW 16,700 (F-2, ■)
6. MW 2,800 (A-2500, ◆)

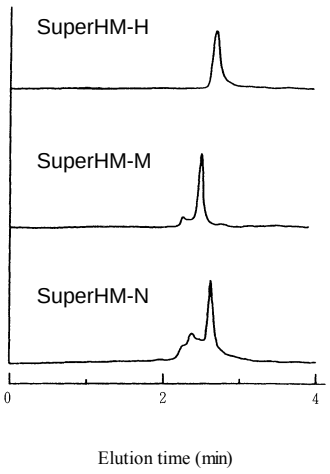


Fig. 32 Comparison of shear degradation of standard polystyrene F-850 using the SuperHM series

Column size: 6.0 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene F-850 (0.01%), 10 µL

3-7. Column temperature

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

(1) Peaks become sharp and separation performance is increased. This is especially noticeable in the high-flow-rate region.

(2) Sample elution volume decreases, shortening analysis time.

(3) 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, and HFIP, etc.

Figures 33 and 34 show the temperature dependence of the separation of epoxy resin and a standard polystyrene mixture in SuperH columns.

Fig. 35 shows the temperature dependence of the separation of standard polystyrenes at various flow rates in the SuperHM-H. Moreover, although shear degradation of the sample occurs at flow rates of 0.8 mL/min and above (see 3-6), shear degradation occurs less readily as the temperature increases.

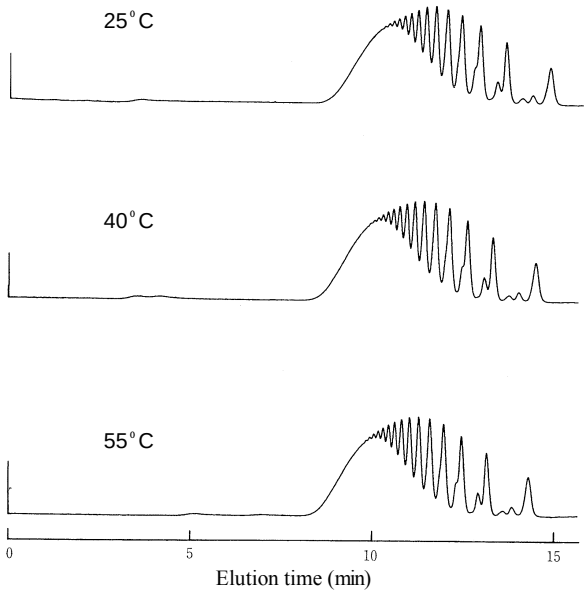


Fig. 33 Temperature dependence of separation of epoxy resin using SuperH columns

Columns: TSKgel SuperH3000 x 2 + TSKgel SuperH2500
6 mm I.D. x 15 cm x 3
Eluent: THF
Flow rate: 0.6 mL/min
Detection: UV (254 nm)
Sample: Epikote 1004 (0.01%), 10 µL

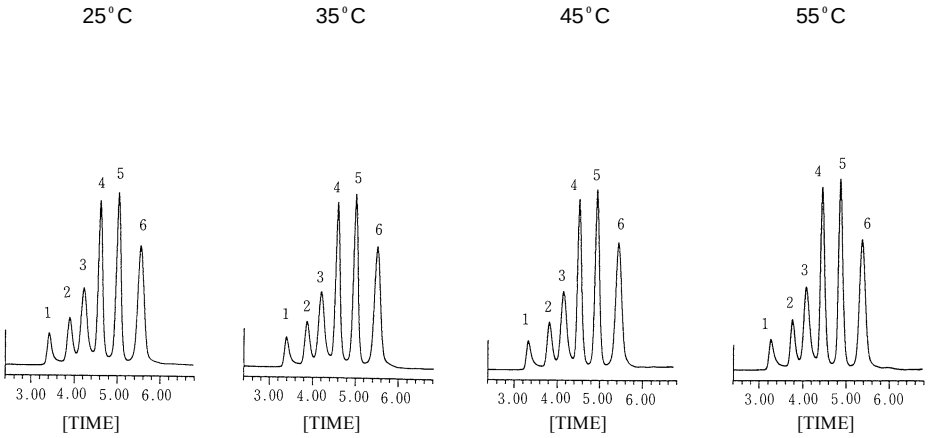


Fig. 34 Temperature dependence of separation of standard polystyrene in TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.5 mL/min
Detection: UV (254 nm)
Sample: Standard polystyrene

1. MW 8,420,000
2. MW 1,260,000
3. MW 422,000
4. MW 107,000
5. MW 16,700
6. MW 2,800

3-8. Optimization of hardware (system)

To maximize column performance, it is extremely important to optimize the analysis conditions, including the solvent and the software, as discussed above. In addition to optimizing these conditions, in ultra high performance SuperH columns in particular, it is important to minimize spreading of the sample peak outside the column. Specifically, suppressing band spreading in the detector, sample injector and tubing is the most important problem with these columns.

MacDonald[6] uses the following equation to express the sample band spreading detected that occurs in actual GPC analysis.

$$\omega t^2 = \omega i^2 + \omega a^2 + \omega j^2 + \omega f^2 + \omega c^2$$

where ωt^2 = total band spreading; ωi^2 = spreading in sample injector; ωa^2 = spreading between sample injector and column inlet and between column outlet and detector inlet; ωj^2 = spreading at joints between columns; ωf^2 = spreading in flow cells (detectors); and ωc^2 = spreading within the column.

From this equation it is clear that while band spreading within the column occurs, it is also significantly affected by components outside the column.

3-8-1. Spreading in the detector

Table 6 compares the number of theoretical plates for a low molecular weight sample (DCHP) using a SuperH2500 column with various types of UV detectors and different flow cell volumes. Fig. 36 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 SuperH column are significantly affected by spreading in the detector, including the size of the flow cell. As a result, in analyses performed with a SuperH column, a UV-8020 microcell with reduced dead volume (or an equivalent device) must be used as the detector.

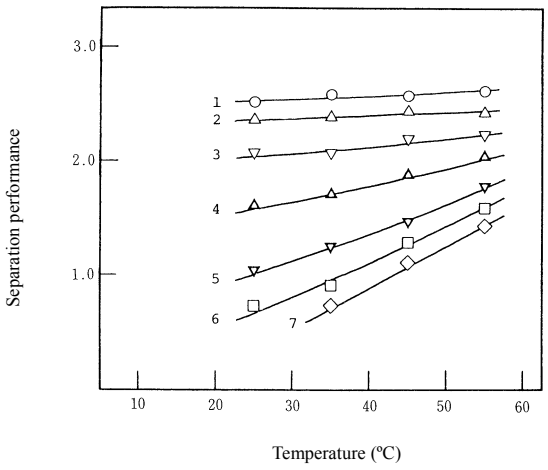


Fig. 35 Relationship between separation performance and temperature at various flow rates using the TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 1. 0.2 mL/min 2. 0.4 mL/min
3. 0.6 mL/min 4. 0.8 mL/min
5. 1.0 mL/min 6. 1.1 mL/min
7. 1.2 mL/min
Temperature: 25 ºC – 55 ºC
Detection: UV (254 nm)
Samples: Standard polystyrene 0.02%, 10 µL
MW 8,420,000 and MW 1,260,000

Table 6 Comparison of number of theoretical plates with various TSKgel SuperH2500 detectors

Number of theoretical plates (TP/15 cm)		
UV-8020*1	UV-8010*2	UV-8010*3
28100	23860	17890

Column: 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: DCHP 0.1%, 2µL

*1 Flow cell volume: 2 µL microcell
*2 Flow cell volume: 10 µL low dead volume type of cell
*3 Flow cell volume: 10 µL

Table 7 compares the number of theoretical plates resulting from differences in the RESPONSE of the UV detector (UV-8020 microcell specifications) in the SuperH2500. When the response time is SLOW, the number of theoretical plates decreases to less than half compared to when it is set to FAST. Thus by setting the time constant to FAST, adequate performance can be obtained in analyses conducted using SuperH columns.

Table 7 Effect of time constant of detector on number of theoretical plates of SuperH2500

Number of theoretical plates (TP/15 cm)		
Time constant (RESPONSE)		
FAST	STD	SLOW
28100	21960	12400

Column: TSKgel SuperH2500 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV-8020 (microcell), 254 nm
Sample: DCHP (0.1%), 2 µL

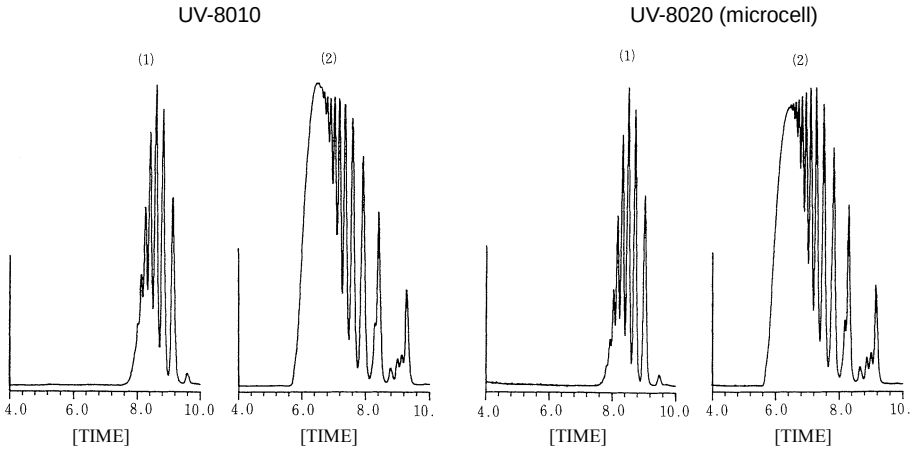


Fig. 36 Dependence of separation performance on band spreading in detector in TSKgel SuperH2500

Column: TSKgel SuperH2500 6 mm I.D. x 15 cm x 2
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Samples: (1) Standard polystyrene A-500 (0.1%), 10 µL
(2) Epikote 1004 (0.1%), 10 µL

3-8-2. Spreading in tubing at column inlet and outlet

According to Scott,[7] band spreading of the sample that occurs within the connecting tubing (Vi) can be expressed by the following equation:

$$Vi^2 = \frac{\pi d^4 FL}{24Dm}$$

where d = inside diameter of tubing; F = flow; L = length of tubing; and Dm = diffusion coefficient of sample in mobile phase.

It is clear from this equation that band spreading depends on the length and inside diameter of the connecting tubing. In particular, the larger the inside diameter of the tubing, the greater the band spreading of the sample.

Using a SuperH2500 column, Fig. 37 shows how the number of theoretical plates is affected by the length of the connecting tubing between the sample injector and the column inlet (inside diameter 0.2 mm). Fig. 38 shows the effect of the length of the connecting tubing between the column outlet and the detector (inside diameter 0.2 mm) on the number of theoretical plates.

In addition, Fig. 39 shows the effect of the length of the connecting tubing between the sample injector and the column inlet on the number of theoretical plates on the inside diameter.

When tubing with an inside diameter of 0.2 mm is used, the number of theoretical plates is affected if the length of the tubing between the sample injector and the column inlet or between the column outlet and detector is longer than 60 cm. Tubing with an inside diameter of 0.1 mm is not linked to a decreased number of theoretical plates up to a length of 80 cm. On the other hand, if the diameter is increased to 0.3 mm, column performance is markedly affected, and it is clear that tubing of 20 cm or longer would not be feasible.

Consequently, although the narrower and shorter the connecting tubing the better, for practical applications, the recommended tubing dimensions are 0.2 mm I.D. x 40 to 50 cm.

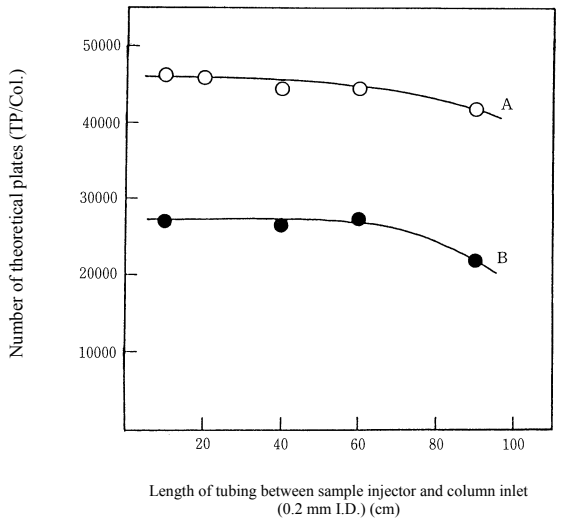


Fig. 37 Relationship between number of theoretical plates and length of column inlet tubing in TSKgel SuperH2500

Column: TSKgel SuperH2500
A. 6 mm I.D. x 15 cm x 2 B. 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min Temperature: 25 ºC
Detection: UV (254 nm), UV-8020 (microcell)
Sample: DCHP (0.1%), 10 µL

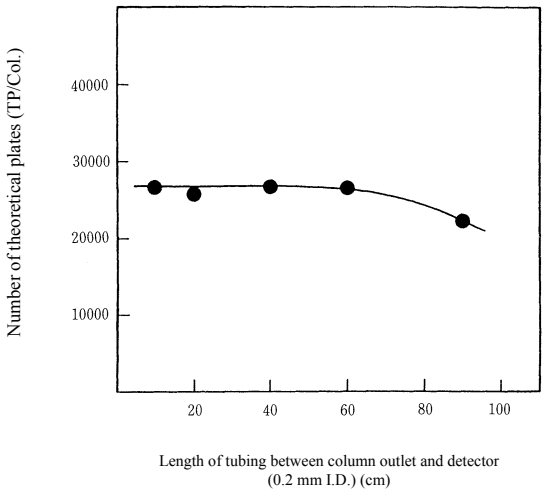


Fig. 38 Relationship between number of theoretical plates and length of column outlet tubing in TSKgel SuperH2500

Column: TSKgel SuperH2500 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min Temperature: 25 ºC
Detection: UV (254 nm), UV-8020 (microcell)
Sample: DCHP (0.1%), 10 µL

3-8-3. Spreading in connecting tubing between columns

Fig. 40 shows the effects of the dimensions of the connecting tubing between columns on the number of theoretical plates using two SuperH2500 columns. Normally, the length of connecting tubing between columns must be around 10 cm, and at this length the column performance is not affected if the tubing has an inside diameter of 0.1 or 0.2 mm. However, if the inside diameter is 0.3 mm, column performance decreases even when the length of the tubing is 10 cm.

3-8-4. Spreading in other connecting components

Band spreading of the sample will also occur for the following reasons, in addition to those discussed in sections 3-8-1 to 3-8-3, above:

(1) Spreading from joints between tubing (connecting joints).
(2) Spreading due to gaps (dead volume) between all connecting components.
(3) Spreading due to machining defects at the ends of the tubing.

Thus, as explained above, spreading outside the column is a major problem for the SuperH series (ultra high performance columns). For optimal use, please consult this Separation Report and the column User's Guide.

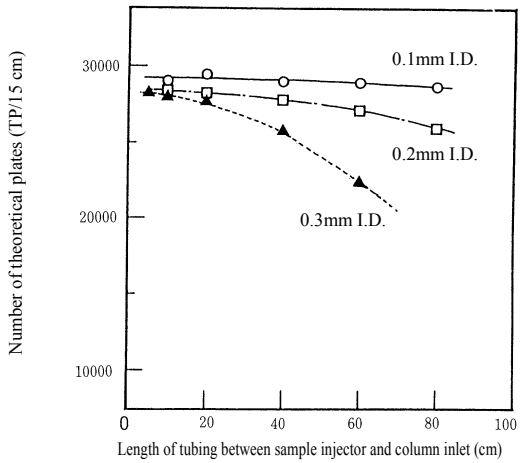


Fig. 39 Relationship between number of theoretical plates and tubing inside diameter and length of column inlet tubing in TSKgel SuperH2500

Column: TSKgel SuperH2500 6 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min Temperature: 25 ºC
Detection: UV (254 nm), UV-8020 (microcell)
Sample: DCHP (0.1%), 2 µL

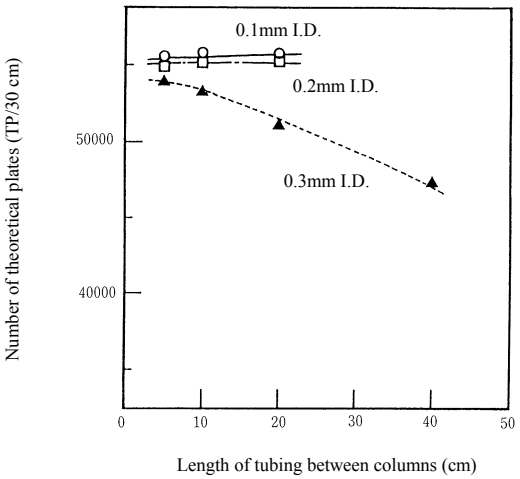


Fig. 40 Relationship between number of theoretical plates and inside diameter of tubing and length of connecting tubing between columns in TSKgel SuperH2500

Column: TSKgel SuperH2500 6 mm I.D. x 15 cm x 2
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm), UV-8020 (microcell)
Sample: DCHP (0.1%), 2 µL

3-9. Mixed bed columns (linear)

The TSKgel SuperH series is a linear mixed-bed series of columns prepared in four grades. The SuperHM-H and SuperHM-M were developed to analyze the molecular weight distribution of polymers, and the SuperHM-N for GPC analysis of samples that have a relatively low molecular weight. The SuperHM-L was developed to analyze oligomers and low molecular weight samples, and also was optimally designed for pattern analysis of samples from the high molecular weight range to the oligomer range.

Table 8 lists the molecular weight ranges and linear range of the calibration curve in each of the grades. Figure 2 shows the calibration curve when THF was used as the solvent, and standard polystyrene is used as the sample.

Figures 41 to 44 compare the elution curves of various types of standard polystyrene in the SuperHM series.

Figures 45 and 46 compare chromatograms of standard polystyrene separated with columns in the SuperHM series.

Figures 47 and 48 compare the separation of epoxy resin with columns in the SuperHM series.

Figure 49 compares the separation of phenol resin with columns in the SuperHM series.

4. Applications

Figures 50 to 56 show examples of analysis performed with various samples.

Figure 57 shows an example of a report (print out) produced when Epikote 1004 was separated using a high speed GPC instrument (HLC-8120PC).

Table 8 Molecular weight fractionation range of mixed-bed columns

Grade	Molecular weight fractionation range	Linear component of calibration curve
SuperHM-L	100～ 3,000,000	200～ 10,000
SuperHM-N	100～ 1,000,000	300～ 200,000
SuperHM-M	300～ 3,000,000	300～ 1,000,000
SuperHM-H	500～ 10,000,000	1,000～ 8,000,000

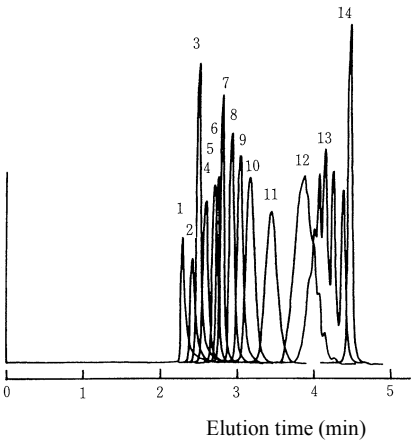


Fig. 41 Elution curve of standard polystyrene using TSKgel SuperHM-L

Column: TSKgel SuperHM-L 6.0 mm I.D. x 15 cm
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample:

1. MW 2,890,000	2. MW 1,260,000	3. MW 775,000
4. MW 422,000	5. MW 186,000	6. MW 107,000
7. MW 42,800	8. MW 16,700	3. MW 10,200
10. MW 6,200	11. MW 2,800	12. A-1,000
13. A-500	14. DCHP	

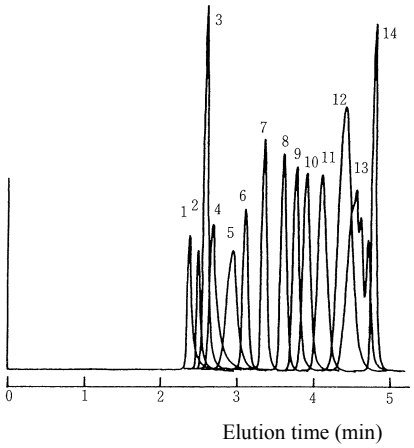


Fig. 42 Elution curve of standard polystyrene using TSKgel SuperHM-N

Column: TSKgel SuperHM-N 6.0 mm I.D. x 15 cm
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample:

1. MW 2,890,000	2. MW 1,260,000	3. MW 775,000
4. MW 422,000	5. MW 186,000	6. MW 107,000
7. MW 42,800	8. MW 16,700	3. MW 10,200
10. MW 6,200	11. MW 2,800	12. A-1,000
13. A-500	14. DCHP	

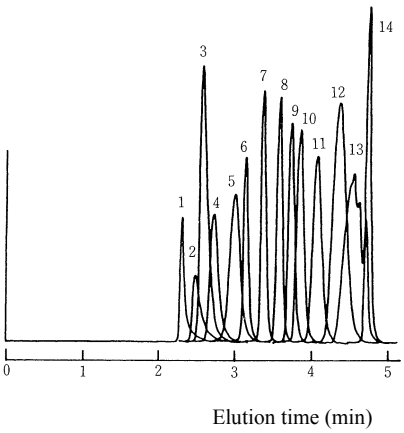


Fig. 43 Elution curve of standard polystyrene using TSKgel SuperHM-M

Column: TSKgel SuperHM-M 6.0 mm I.D. x 15 cm
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample:

1. MW 2,890,000	2. MW 1,260,000	3. MW 775,000
4. MW 422,000	5. MW 186,000	6. MW 107,000
7. MW 42,800	8. MW 16,700	3. MW 10,200
10. MW 6,200	11. MW 2,800	12. A-1,000
13. A-500	14. DCHP	

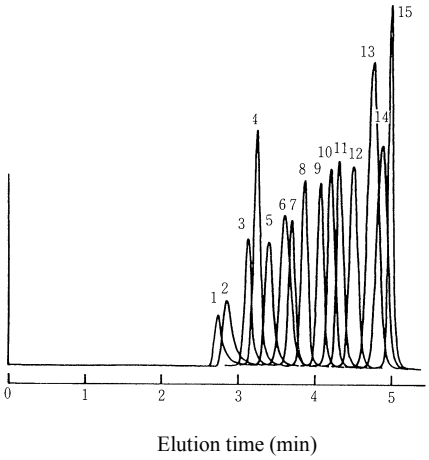


Fig. 44 Elution curve of standard polystyrene using TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6.0 mm I.D. x 15 cm
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample:

1. MW 5,480,000	2. MW 2,890,000	3. MW 1,260,000
4. MW 775,000	5. MW 422,000	6. MW 186,000
7. MW 107,800	8. MW 42,800	3. MW 16,700
10. MW 10,200	11. MW 6,200	12. MW 2,800
13. A-1,000	14. A-500	15. DCHP

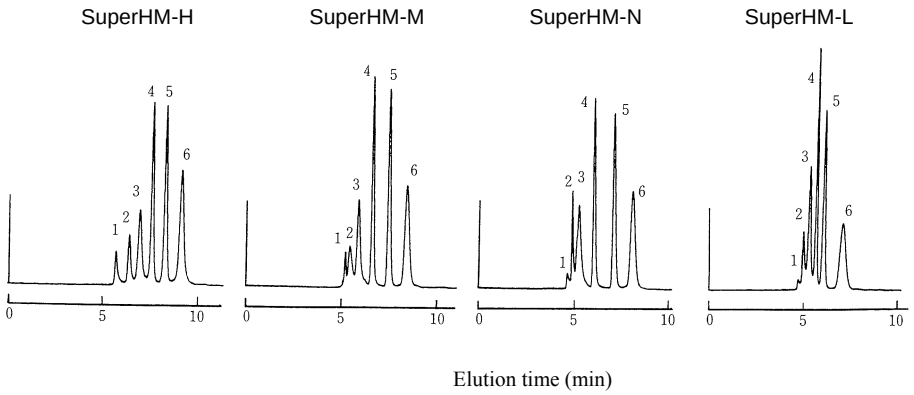


Fig. 45 Comparison of separations of standard polystyrenes with the TSKgel SuperHM series (1)

Columns: TSKgel SuperHM-H, SuperHM-M, SuperHM-N, SuperHM-L
Both: 6 mm I.D. x 15 cm x 2
Eluent: THF Flow rate: 0.6 mL/min
Temperature: 25 ºC Detection: UV (254 nm)
Sample: Standard polystyrene
1. MW 8,420,000 2. MW 1,260,000
3. MW 422,000 4. MW 107,000
5. MW 16,700 6. MW 2,800

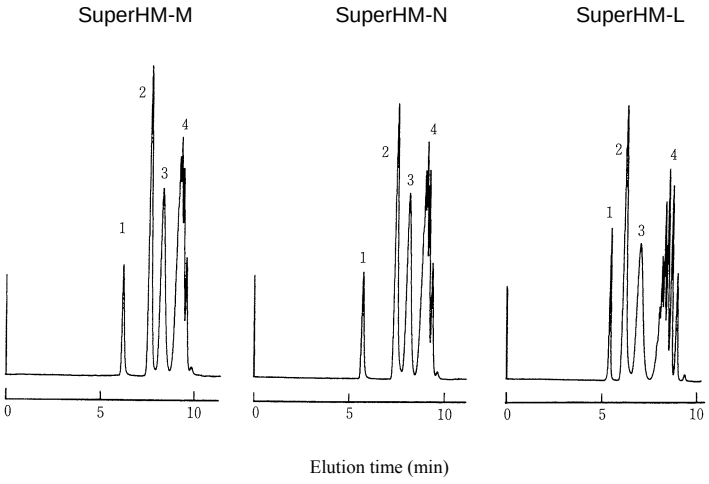


Fig. 46 Comparison of separations of standard polystyrenes with the TSKgel SuperHM series (2)

Column: TSKgel SuperHM-M, SuperHM-N, SuperHM-L
Both: 6 mm I.D. x 15 cm x 2
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene
1. MW 190,000 2. MW 9,100
3. MW 2,800 4. A-500

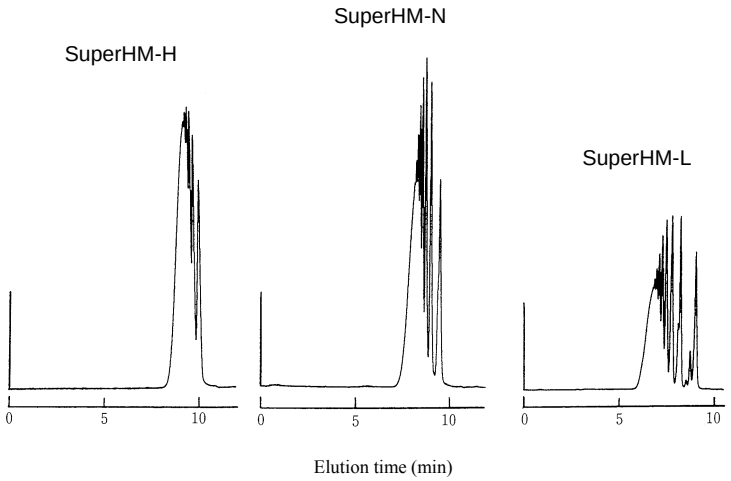


Fig. 47 Comparison of separations of epoxy resin with the TSKgel SuperHM series (1)

Columns: TSKgel SuperHM-H, SuperHM-N, SuperHM-L (6 mm I.D. x 15 cm x 2, respectively)
Eluent: THF
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Epoxy resin (Ep100)

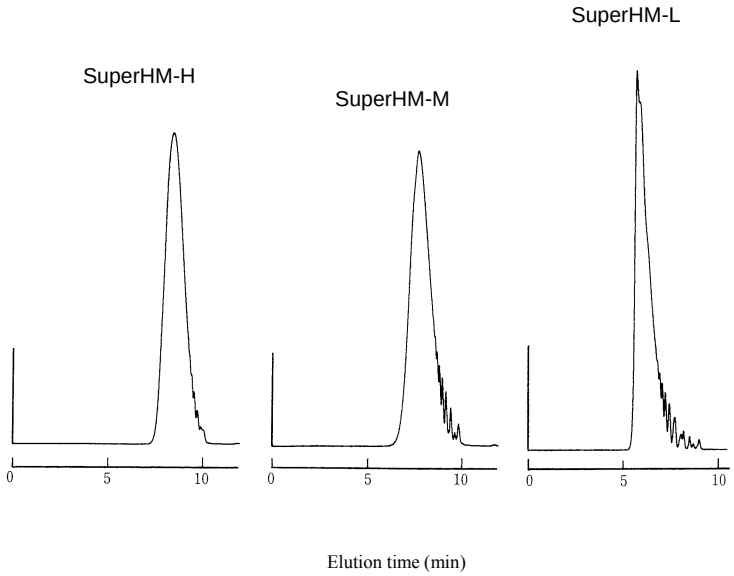


Fig. 48 Comparison of separations of epoxy resin with the TSKgel SuperHM series (2)

Columns: TSKgel SuperHM-H, SuperHM-M, SuperHM-L (6 mm I.D. x 15 cm x 2, respectively)
Eluent: THF
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Epoxy resin (Ep100)

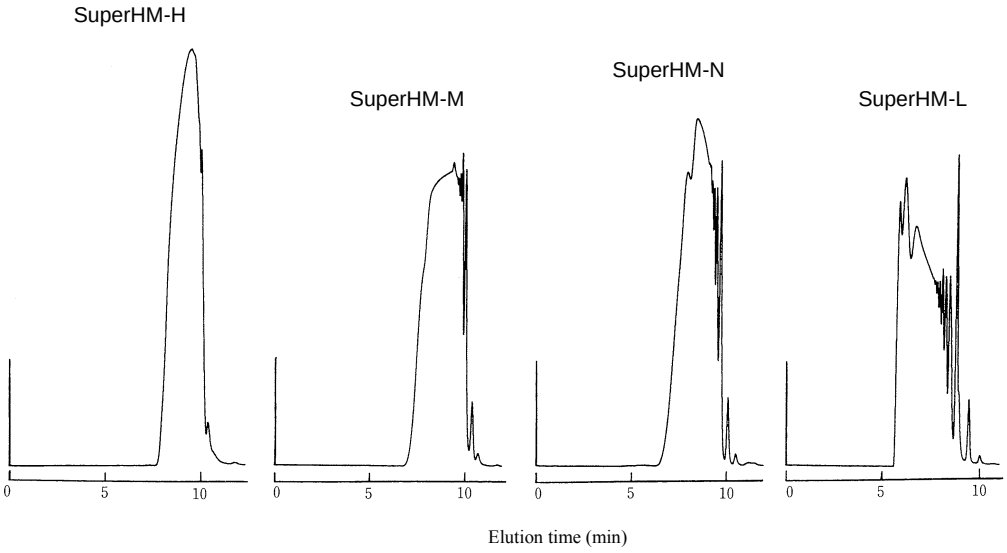


Fig. 49 Comparison of separations of phenol resin with the TSKgel SuperHM series

Columns: TSKgel SuperHM-H, SuperHM-M,
SuperHM-N, SuperHM-L
(6 mm I.D. x 15 cm x 2, respectively)
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Phenol resin

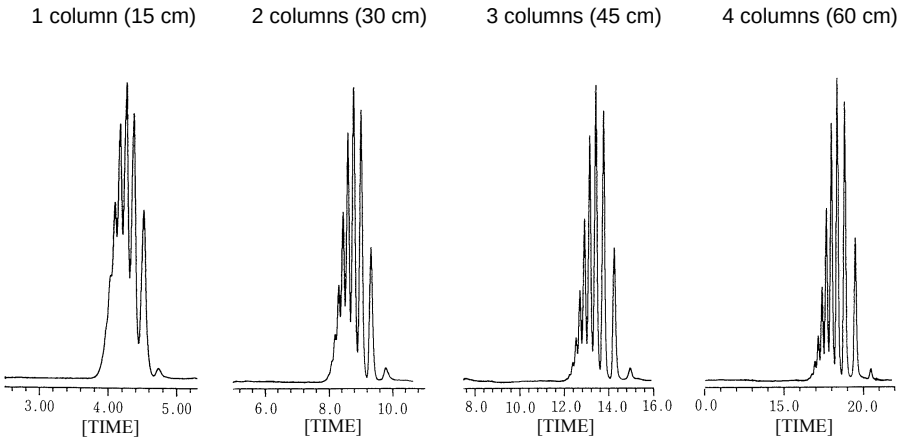


Fig. 50 Dependence of separation of standard polystyrene (A-500) on column length in TSKgel SuperH2500

Column: TSKgel SuperH2500
6 mm I.D. x 15 cm～6 mm I.D. x 15 cm x 4
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene A-500 (0.1%, 10µL)

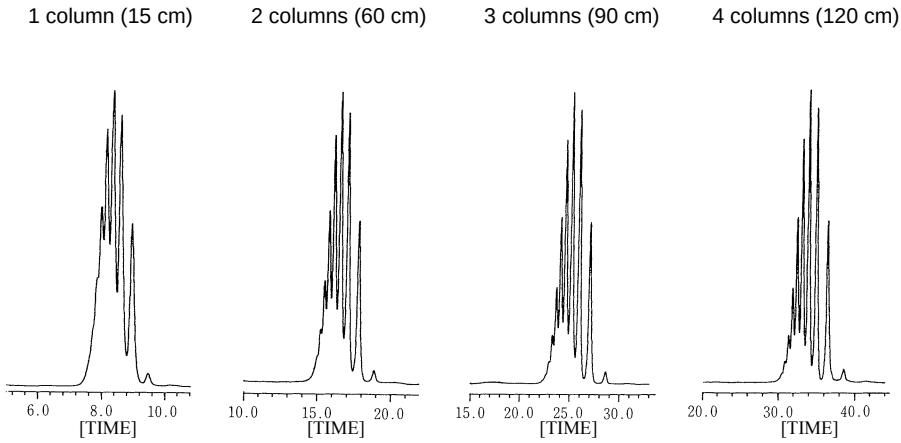


Fig. 51 Dependence of separation of standard polystyrene (A-500) on column length in TSKgel G2500 H_{XL}

Column: TSKgel G2500H_{XL}
7.8 mm I.D. x 30 cm～7.8 mm I.D. x 30 cm x 4
Eluent: THF
Flow rate: 1.0 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene A-500 (0.1%), 20 µL

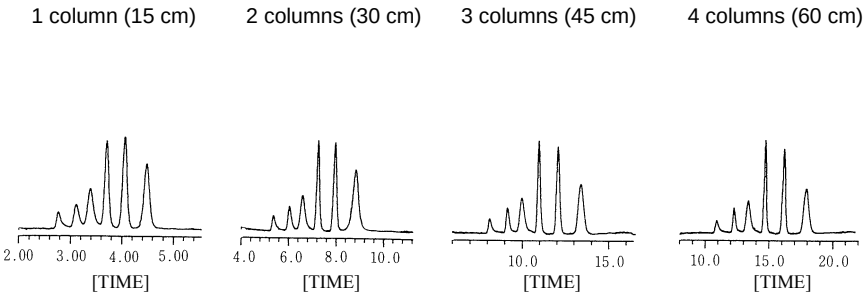


Fig. 52 Dependence of separation of standard polystyrene on column length in TSKgel SuperHM-H

Column: TSKgel SuperHM-H
6 mm I.D. x 15 cm～6 mm I.D. x 15 cm x 4
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Standard polystyrene

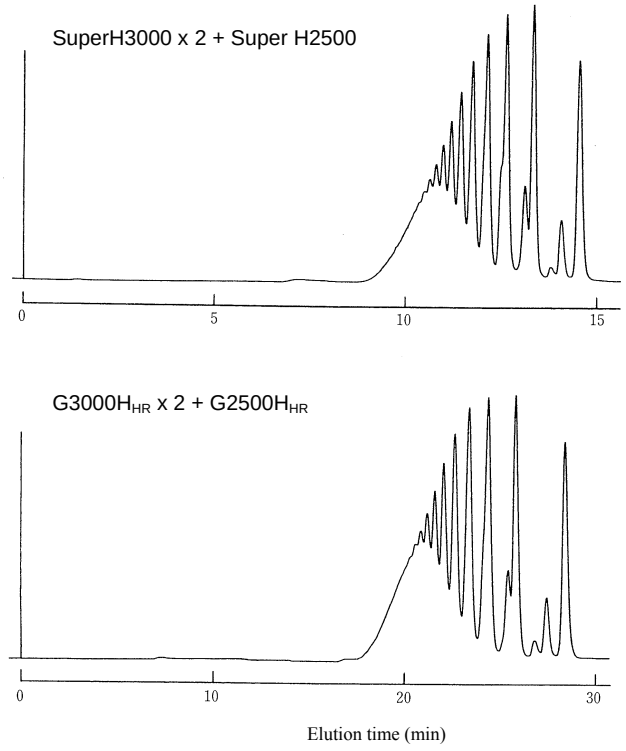


Fig. 53 Comparison of separation performance of SuperH and H_{HR}

Columns: TSKgel SuperH3000 x 2 + SuperH2500
6 mm I.D. x 15 cm x 3
TSKgel G3000H_{HR} x 2 + G2500H_{HR}
7.8 mm I.D. x 30 cm x 3
Eluent: THF
Flow rate: SuperH: 0.6 mL/min
H_{HR}: 1.0 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Epikote 1001 (0.1%), 10 µL

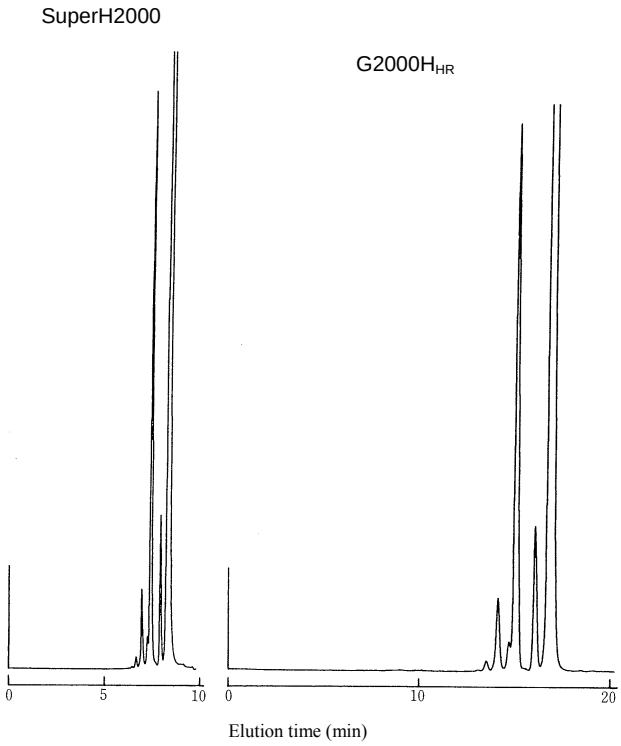


Fig. 54 Comparison of separation of epoxy resin in TSKgel SuperH2000 and G2000 H_{HR} columns

Columns: TSKgel SuperH2000 (6 mm I.D. x 15 cm x 2)
TSKgel G2000H_{HR} (7.8 mm I.D. x 30 cm x 2)
Eluent: THF
Flow rate: SuperH2000: 0.6 mL/min
G2000 H_{HR}: 1.0 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Epikote 828 (0.1%), 10 µL

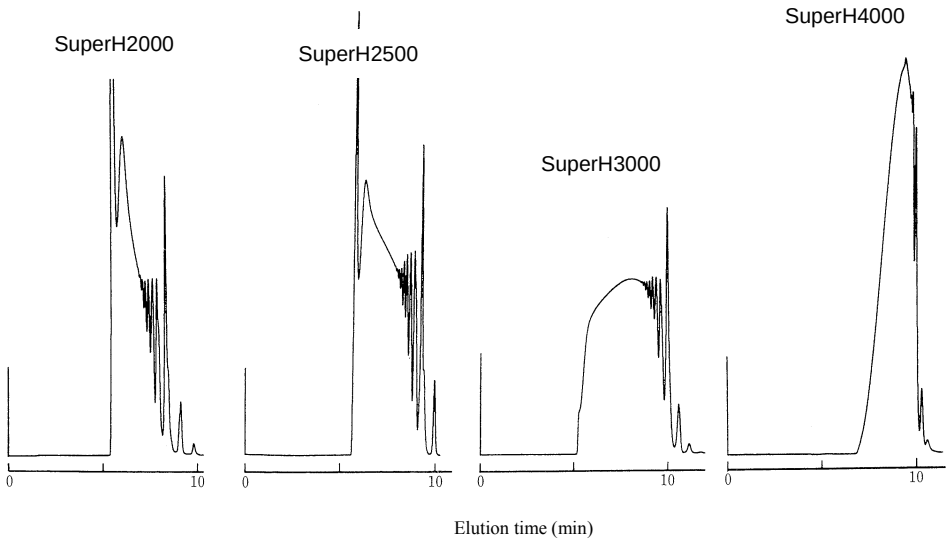


Fig. 55 Comparison of separation of phenol resin in TSKgel SuperH series

Columns: TSKgel SuperH2000～SuperH4000
(6 mm I.D. x 15 cm x 2, respectively)
Eluent: THF
Flow rate: 0.6 mL/min
Temperature: 25 ºC
Detection: UV (254 nm)
Sample: Phenol resin (0.1%), 5 µL

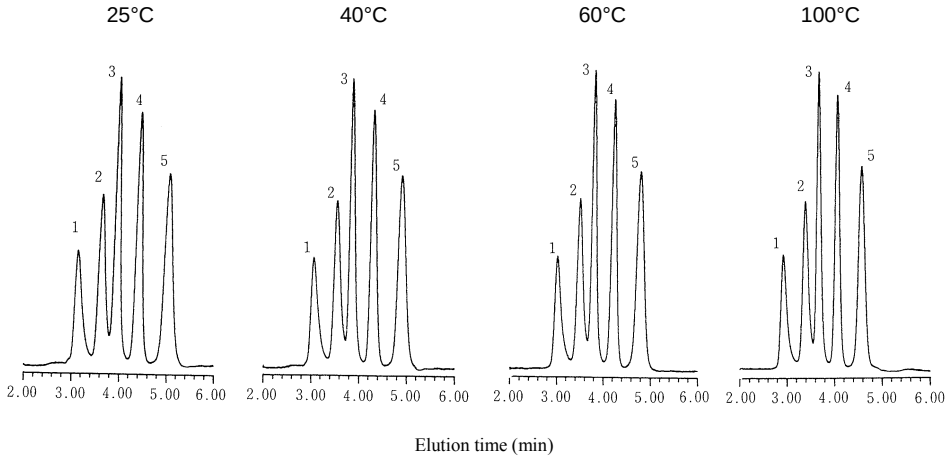


Fig. 56 Temperature dependence of separation of standard polystyrene with the TSKgel SuperHM-H

Column: TSKgel SuperHM-H 6 mm I.D. x 15 cm
Eluent: 10 mM LiBr in DMF
Flow rate: 0.6 mL/min
Detection: UV (270 nm)
Sample: Standard polystyrene

1. MW 2,890,000
2. MW 422,000
3. MW 107,000
4. MW 16,700
5. MW 2,800

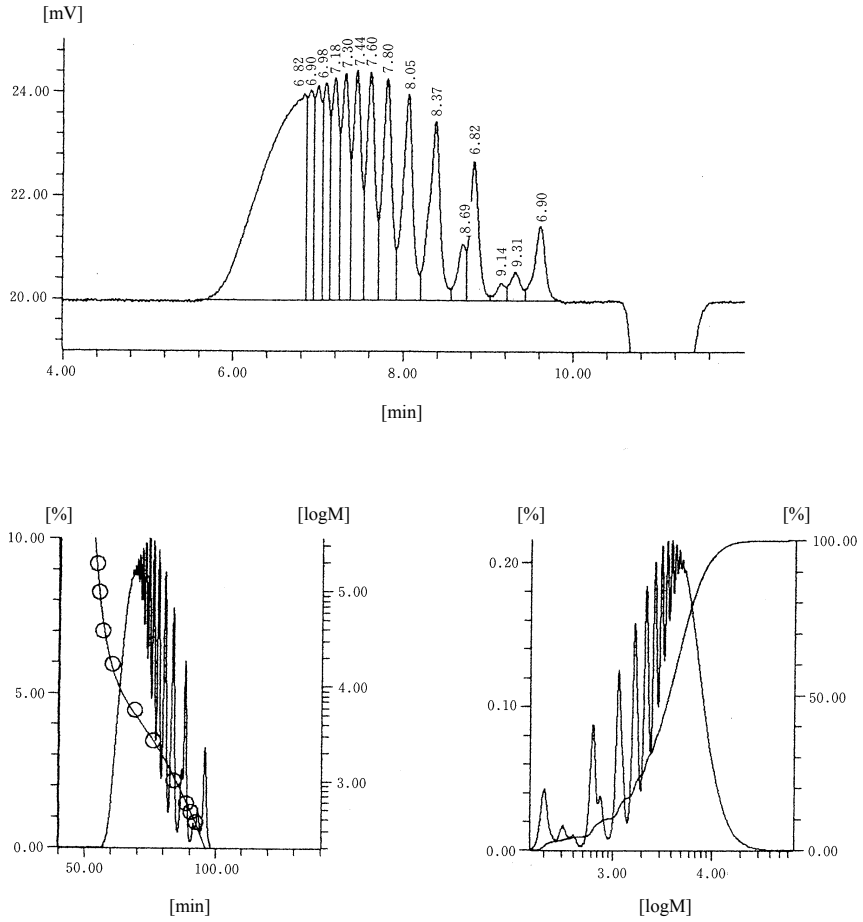


Fig. 57 Separation of Epikote 1004 (E1004) using high speed GPC instrument (HLC-8120PC)

Columns: TSKgel SuperH3000 + TSKgel SuperH2500
6 mm I.D. x 15 cm x 2

Temperature: 25 ºC

Eluent: THF

Flow rate: 0.6 mL/min

Detection: RI

Sample: Epikote 1004 (EP1004) (0.1%), 10 µL

5. Conclusion

With the TSKgel H-Type SuperH Series of ultra high speed and performance semi-micro GPC columns, the separation performance of conventional H_{HR} and H_{XL} series can be achieved in half the analysis time.

The SuperH series was designed to minimize sample band spreading within the column. Consequently, to maximize the performance of these columns, it is important to optimize analysis conditions and decrease dead volume in the system as described above.

As a result, a conventional build up system with reduced dead volume can also be used for analysis, but here the performance of the SuperH series can be exploited by using Tosoh's all solvent type of high performance GPC instrument, the HLC-8120 GPC.

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