

Linearity Improvements From Parallel Multiple Pore Size GPC Column Technology

Joe Machamer, GPC Marketing Manager





Outline

Basics of GPC Calibration

Wide Molecular Weight Range Separation

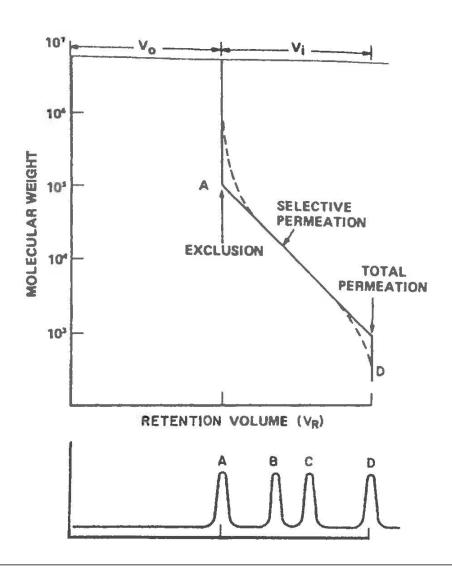
- Serial Approach
- Parallel Approach

Characteristics of TSK-GEL SupermultiporeHZ Columns

The Tosoh EcoSEC® GPC System

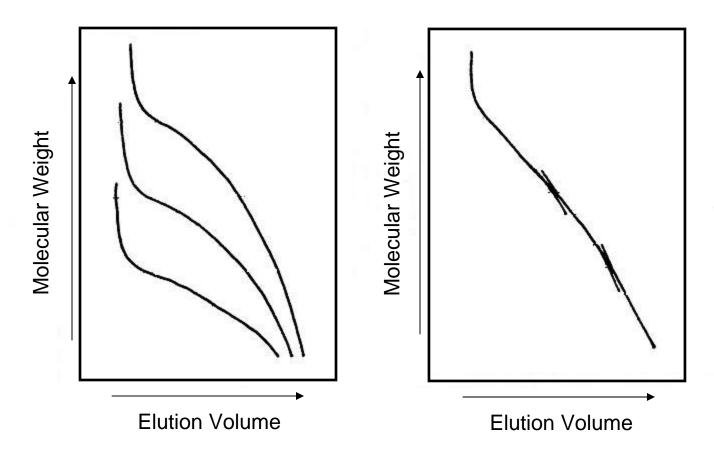


GPC Calibration



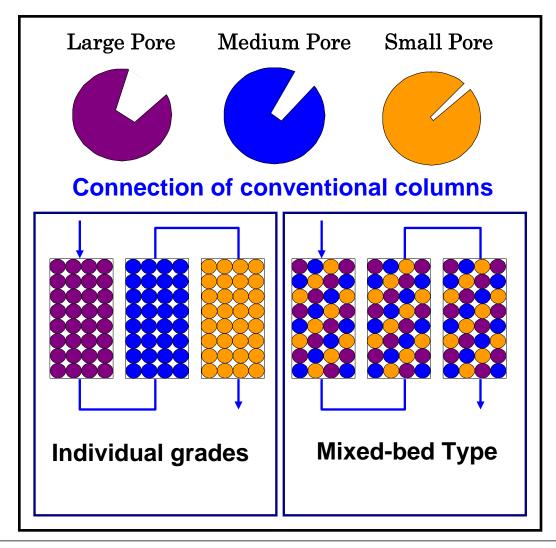


GPC Calibration Curves



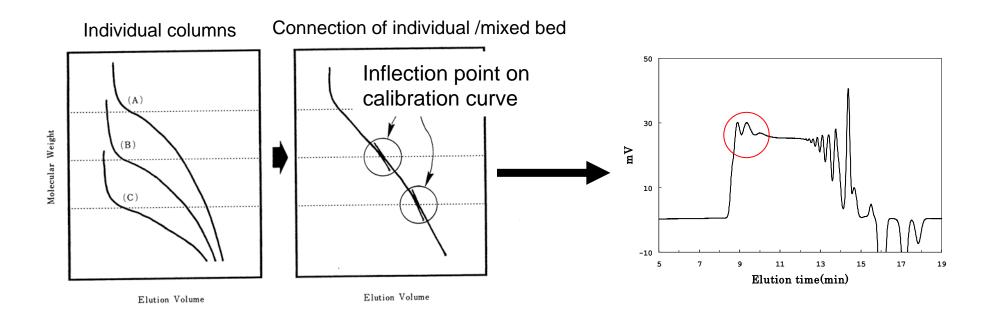


Schematic diagrams of SEC packings





Distortion in chromatogram on conventional columns

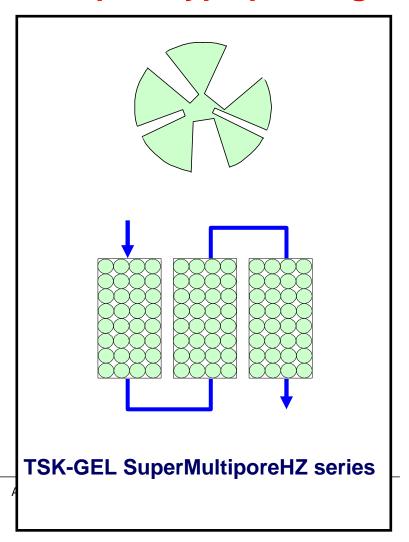




Schematic diagrams of SEC packings

Conventional packings/columns Multipore type packings

Large Pore Small Pore Medium Pore Connection of conventional columns **Mixed-bed Type Individual grades**



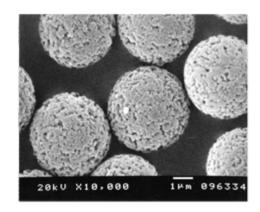


TSK-GEL SuperMultiporeHZ Packing via SEM

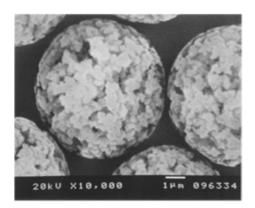
TSKgel SuperMultiporeHZ-N 3µm

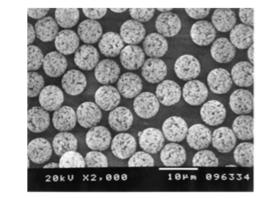
20kU X10,000 1µm 096334

TSKgel SuperMultiporeHZ-M 4µm



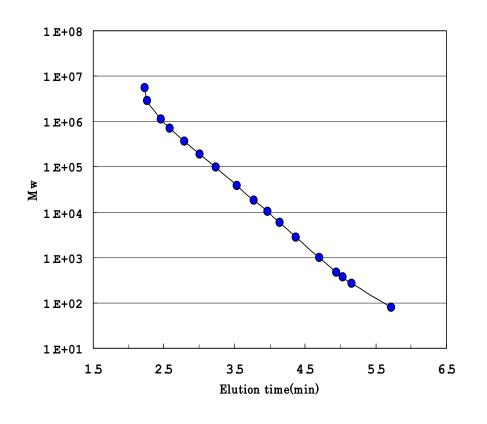
TSKgel SuperMultiporeHZ-H 6µm

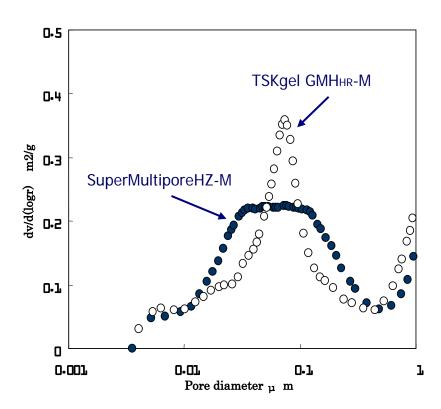






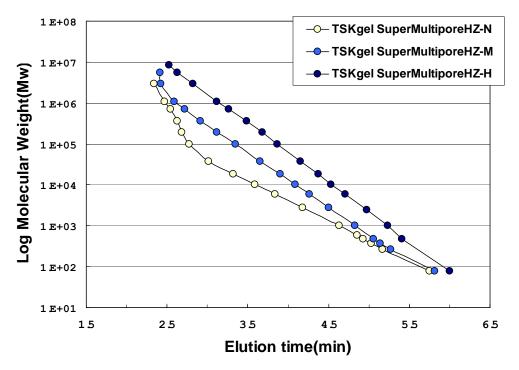
Pore characterization of TSKgel SuperMultiporeHZ-M







Calibration curves of TSK-GEL SuperMultiporeHZ columns



Column: TSK-GEL SuperMultiporeHZ

(4.6mmID x 15cm)

Eluent: THF

Flow rate: 0.35 mL/min Detector: UV254nm

Temp. : 25

Inj. Volume: 5µL

Samples: Std. Polystyrene



Features of TSK-GEL® SuperMultiporeHZ

Packing materials with broad pore size distribution

- No inflection point in calibration curve
- No distortion in chromatogram

Real linear calibration curve

- Lower error between actual MW and calibration curve
- More accurate MW measurement

Large porosity

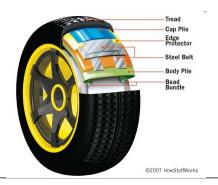
High resolution especially in oligomeric region

Lower lot-to-lot variation

- Better reproducibility of MW measurement
- 3 types of columns with different separation ranges
 - Applicable to wide MW range

Small particle size packed in semi-micro columns

High-speed, high-resolution, low solvent consumption





Semi-micro vs. Conventional Columns

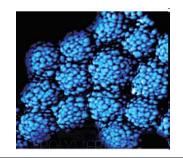
	Conventional	Semi-micro
Diameter (mm)	7.8	4.6-6.0
Length (cm)	30	15
Flow Rate (mL/min.)	1.0	0.35
Run Time (Arbitrary)	100	50

Semi-micro columns reduce run times by 50% and flow rate by 2/3. Throughput is doubled and solvent costs drop by 1/6.



Properties of TSK-GEL SuperMultiporeHZ columns

	SuperMultiporeHZ-N	SuperMultiporeHZ-M	SuperMultiporeHZ-H
Base	PS/DVB	PS/DVB	PS/DVB
Particle Size (µM)	3	4	6
Exclusion Limit (MW)	120,000	2,000,000	40,000,000
Mean Pore Dia. (nM)	8	14	
Separation Range (MW)	300-50,000	500-1,000,000	1,000-10,000,000
Theoretical Plates	20,000TP/15cm	16,000TP/15cm	11,000TP/15cm
Column Size	4.6mm I.D. x 15 cm	4.6mm I.D. x 15 cm	4.6mm I.D. x 15 cm

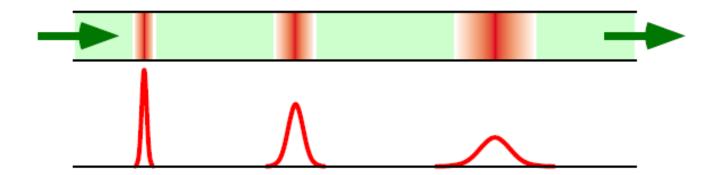




Effect Of System Volume On Resolution

Why Do Bands Spread?

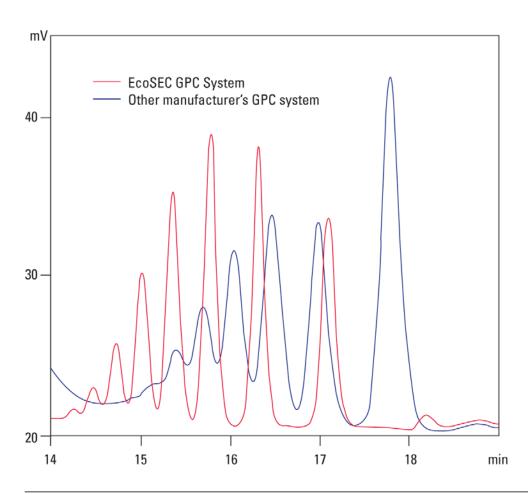
- a. Eddy diffusion
- b. Mobile phase mass transfer
- c. Stagnant mobile phase mass transfer
- d. Stationary phase mass transfer
- e. Longitudinal diffusion





Resolution: Semi-micro vs. Conventional System

 Semi-micro system offers better resolution for semimicro columns



TSKgel SuperHZ2000, 4.6mm ID x 15cm, × 4

Mobile phase: THF

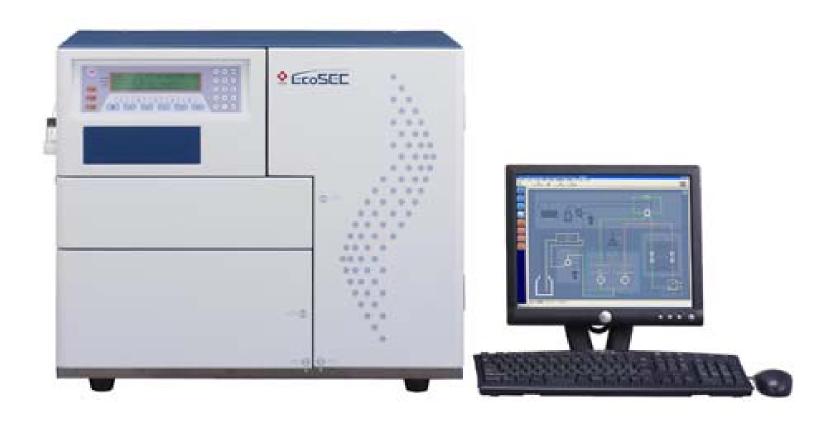
Flow rate: 0.35mL/min

Detection: RI Temperature: 40°C

Injection vol.: 10µL (0.2mg/mL)



EcoSEC GPC System





Save Time and Money

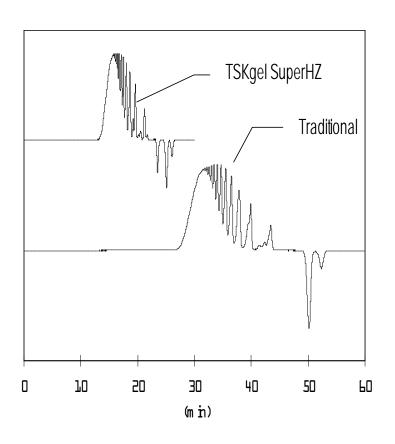
- Low dead volume design and system layout
 - 7.5μL Stroke Volume
 - 2.5μL RI Cell Volume
 - Use of 0.2mm and 0.4mm tubing





Save Time and Money

Reduce run times by 50%



Flow rate: TSKgel SuperHZ(0.35mL/min)

TSKgel H_{HR} (1.0mL/min)

Dimensions: 4.6mm ID x 15cm*4 (TSKgel SuperHZ)

7.8mm ID x 30cm*4 (TSKgel H_{HR})

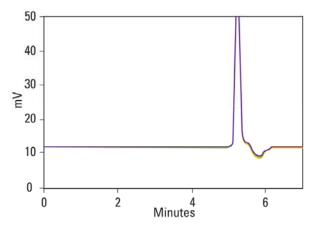


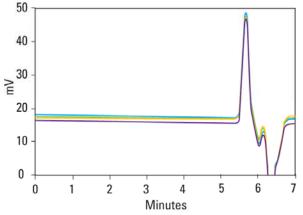
Save Time and Money

Reduce solvent usage by 85%

Solvent	Competitor GPC (solvent + disposal)	EcoSEC GPC System (solvent + disposal)	<u>Savings</u>
NMP (\$30/L)	\$3082	\$1312	\$1,770.00
Chloroform (\$17/L)	\$1830	\$779	\$1,051.00
DMF (\$25/L)	\$2600.50	\$1117	\$1,463.50
HFIP (\$1000/L)	\$96,493	\$41,193	\$55,300.00







TSKgel SuperMultiporeHZ-M, 4.6mm ID x 15cm

Mobile Phase: THF

Flow rate: 0.35mL/min

Detection: RI
Temperature: 40°C
Injection vol.: 10µL

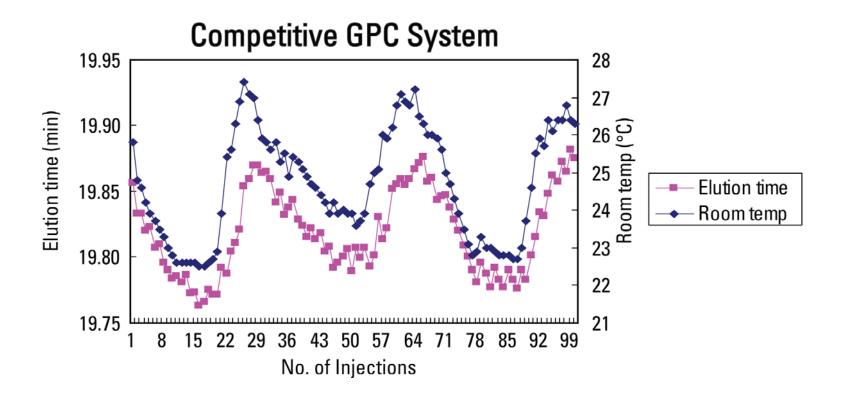
Sample: dicyclohexyl phthalate

EcoSEC GPC System

Other GPC manufacturer

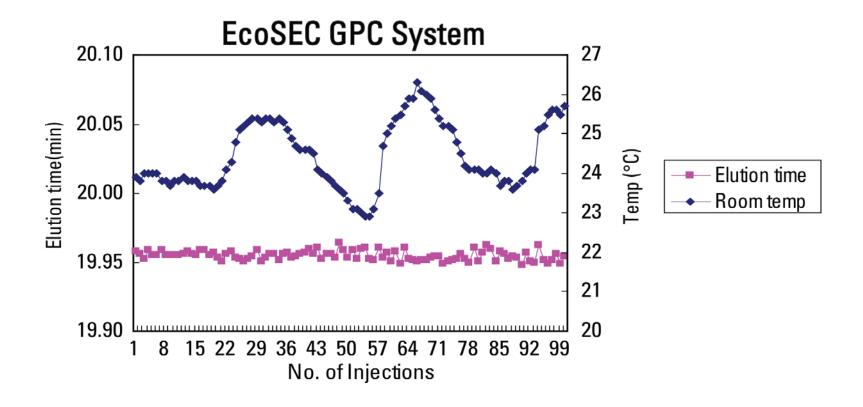


- Room Temperature Affect On Retention Time
 - Conventional Instrument-No Pump Temperature Control



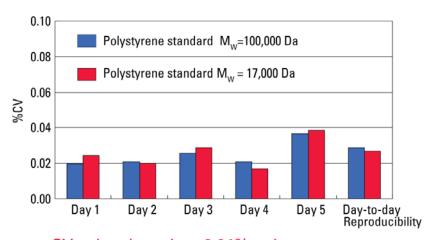


- Room Temperature Affect On Retention Time
 - EcoSEC GPC System-With Temperature Controlled Pumps





- Temperature Controlled Pumps
- Excellent Retention Time Precision



CV value less than 0.04% a day. CV value less than 0.03% on different days.

TSKgel SuperMultiporeHZ-M, 4.6mm ID x 15cm, × 2

Mobile phase: THF

Flow rate: 0.35mL/min

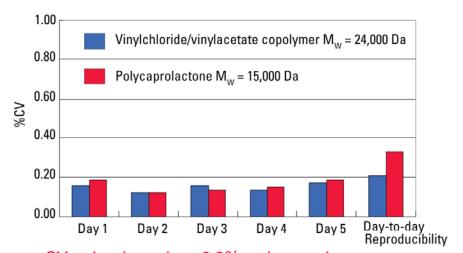
Temperature: 40°C Injection vol.: 10µL

Samples: polystyrene standards

of injections: 10/day



- Temperature Controlled Pumps
- Excellent Molecular Weight Precision



CV value less than 0.2% or less a day. CV value less than 0.4% on different days.

TSKgel SuperMultiporeHZ-M, 4.6mm ID x 15cm, \times 2

Mobile phase: THF

Flow rate: 0.35mL/min

Temperature: 40°C Injection vol.: 10µL

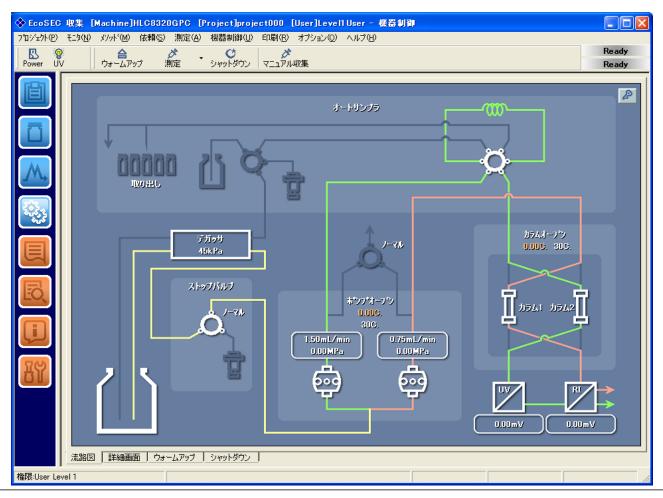
Samples: copolymer and polyester standards

of injections: 10/day



Versatility

Easy to use software





Versatility

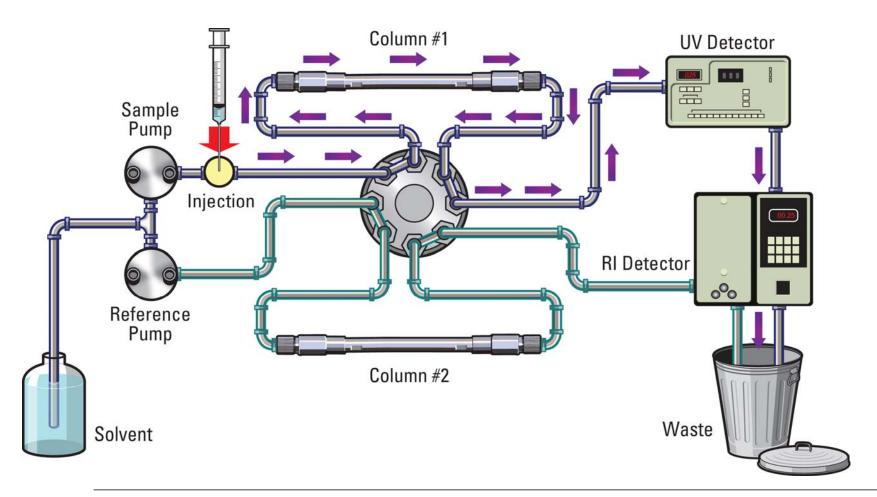
• Column oven holds up to 8, 30cm columns





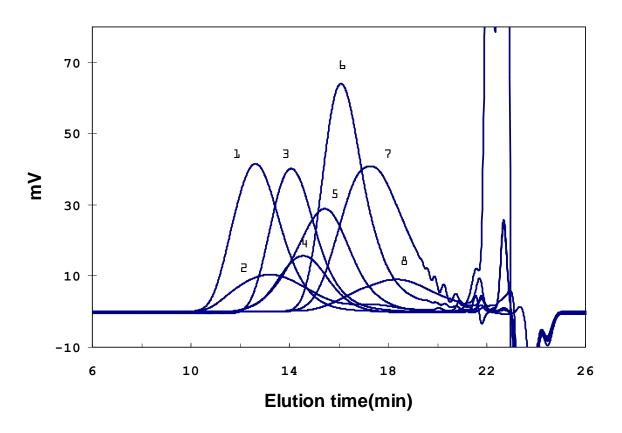
Versatility

Optional Column Switching Valve





Chromatograms of various polymers on SuperMultiporeHZ column



Column: TSK-GEL

SuperMultiporeHZ-H (4.6mmID x 15cm) x 4

Eluent: THF

Flow rate: 0.35 mL/min

Detection: RI Temp.: 40°C

Load: 10µL, 3g/L each Sample: 1. Poly isobutylene

- 2. Acrylic resin #1
- 3. Polystyrene (SRM706)
- 4. Polyvinylchloride
- 5. Polyvinylbutylral
- 6. Polycarbonate
- 7. Epoxy resin
- 8. Acrylic resin #2



Conclusion

- TSK-GEL SupermultiporeHZ columns provide accurate wide linear range separations
- Semi-micro columns
 - Save Time and Money
- Eco-SEC GPC System combined with TSK-GEL SupermultiporeHZ columns
 - Superior Performance
 - Versatility