



Semi-Micro Columns for Size-Exclusion Chromatography in Organic Solvent Systems: TSK-GEL SuperMultiporeHZ Series Columns

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In size exclusion chromatography, obtaining calibration curves over a wide range of molecular weights is a difficulty investigators often encounter when analyzing polymers with a broad molar mass distribution. To overcome this problem two procedures are typically used. One option is to use multiple columns of different pore sizes linked together in series. A second is 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 molecular weight standards.

As is shown in Figure 1, a novel approach to solve this problem was developed by Tosoh scientists and is incorporated in TSK-GEL SuperMultiporeHZ Series columns. Using proprietary multi-pore particle synthesis technology, packing material composed of monodisperse particles are synthesized in which a wide range of pore sizes are contained within a single particle. The TSK-GEL SuperMultiporeHZ Series product line further improves performance by reducing column dimensions to the semi-micro level (4.6mm ID × 15cm), which cuts down on solvent consumption costs by one-sixth and running times by one-half.

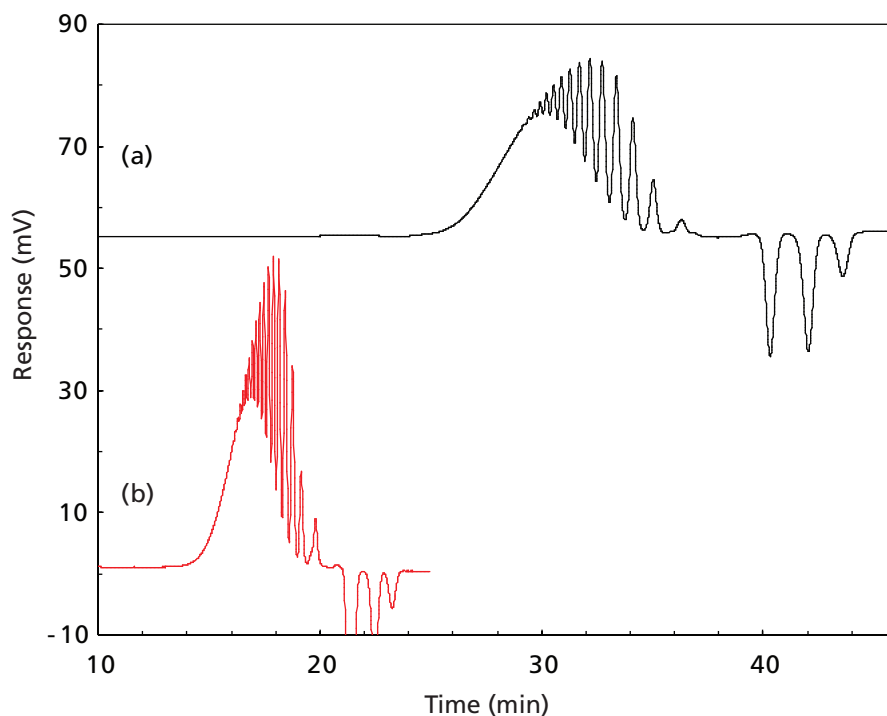


Figure 1: Columns: (A) Conventional columns: TSKgel G1000HXL +3000+2500+2000 (all 5 μm), 7.8 mm ID \times 30 cm \times 4; (B) TSKgel SuperMultiporeHZ-N, 4.6 mm ID \times 15 cm \times 4
 Solvent: THF
 Flow rate: (A) 1.0mL/min
 (B) 0.35mL/min
 Detection: RI
 Temperature: 40°C
 Sample: poly(tetramethylene ether glycol) (PTMEG 650), 10 $\mu\text{g}/\mu\text{L}$
 Detection: RI
 Inj. vol: (A) 50 μL
 (B) 10 μL

Table I: Physical properties of TSK-GEL SuperMultiporeHZ column

Product name	TSKgel SuperMultiporeHZ-N	TSKgel SuperMultiporeHZ-M	TSKgel SuperMultiporeHZ-H
Packing material	poly(styrene/divinylbenzene)	poly(styrene/divinylbenzene)	poly(styrene/divinylbenzene)
Particle size	3 μm	4 μm	6 μm
Plate number (15 cm)	20,000	16,000	11,000
Exclusion limit	120,000	2,000,000	40,000,000*
Column dimensions	4.6 mm ID \times 15 cm	4.6 mm ID \times 15 cm	4.6 mm ID \times 15 cm

* Applicable to polystyrene in THF

Results

Figure 1 compares the separation of oligomers in poly(tetramethylene ether glycol) (PTMEG 650) of four conventional mixed bed GPC columns (A) to the TSKgel SuperMultipore-HZ-N column (B). The TSKgel SuperMultipore-HZ-N column (B) provides the same or higher resolution at a much shorter analysis time than multiple columns linked together.

Table II shows the average molecular weight of silicone resin analyzed on several different lots of TSK-GEL SuperMultipore-HZ-N columns. The data reveals that there is very little difference between lots.

Table II: Reproducibility of different lots of TSK-GEL SuperMultiporeHZ-N columns

Column (Gel Lot)	Mw	Mn	Mz
TSKgel SuperMultiporeHZ-N (A)	3,410	1,340	7,750
TSKgel SuperMultiporeHZ-N (B)	3,400	1,340	7,740
TSKgel SuperMultiporeHZ-N (C)	3,430	1,350	7,850
Avg. (RSD)	3,410 (0.37%)	1,340 (0.35%)	7,780 (0.64%)

Conclusion

TSK-GEL SuperMultiporeHZ Series size exclusion columns represent an innovative type of semi-micro GPC column. These columns have an extended linear MW range with superior reproducibility. Using these products lowers solvent consumption and disposal expenses while offering higher-throughput capabilities. There are three columns available within the TSK-GEL SuperMultiporeHZ Series, each with a different particle size, separation range, and exclusion limit. These columns can separate and characterize polymers within a wide molecular mass range.

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