Characteristics of Linear Type GFC/SEC Semi-Micro Columns for Water-Soluble Polymer Analysis

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Introduction

- Gel filtration chromatography (GFC) is the branch of size exclusion chromatography (SEC) in which an aqueous or aqueous/organic mobile phase is used for the determination of molecular mass distribution and average molecular mass of polymer compounds. SEC plays an important role in not only R&D but also the quality control of synthetic polymers in the chemical industry.

- Tosoh first introduced TSK-GEL multipore columns for organic SEC analysis in 2001. These columns are designed for improving the linearity of the calibration curve over a wide range of molecular weights and removing so-called inflection points on chromatograms. Tosoh's innovative multipore technology resulted in better reproducibility for polymer analysis by SEC.

- Recently we developed TSK-GEL SuperMultiporePW columns: a series of novel multipore columns for aqueous SEC analysis packed with spherical monodisperse polymethacrylate particles. In addition, we also developed a high resolution semi-micro column, the TSK-GEL SuperOligoPW column, for fast analysis of low weight water-soluble polymers.

- In this poster, we report on the basic properties of these new TSK-GEL SEC columns and show elution profile comparisons of synthetic polymers on the newly developed columns and conventional columns.

Experimental

Columns – Tosoh Corporation (Japan)

Semi-micro Multipore:
- TSKgel SuperMultiporePW-N, PW-M, PW-H, 6.0mm ID x 15cm

Semi-micro:
- TSKgel SuperOligoPW, 6.0mm ID x 15cm

Conventional:
- TSKgel G3000PW$_{XL}$, G5000PW$_{XL}$, G-Oligo-PW, 7.8mm ID x 30cm
- TSKgel GMPW$_{XL}$, 7.8mm ID x 30cm, mixed bed column

Instrumentation

Instrument: EcoSEC® GPC system (Tosoh)
Data processing: EcoSEC-WS (Tosoh)

Chemicals and Reagents

Water for eluent and sample preparation was purified with a Milli-Q® water purification system.
PEO, PEG and synthetic polymers were obtained from Tosoh, Wako Pure Chemical (Osaka) and other suppliers.

Preparation of Sample Solution

PEO and PEG standards were dissolved in water at concentrations of 0.5-1.0g/L. Synthetic polymers were dissolved in eluent at concentrations of 3.0-5.0g/L prior to use.

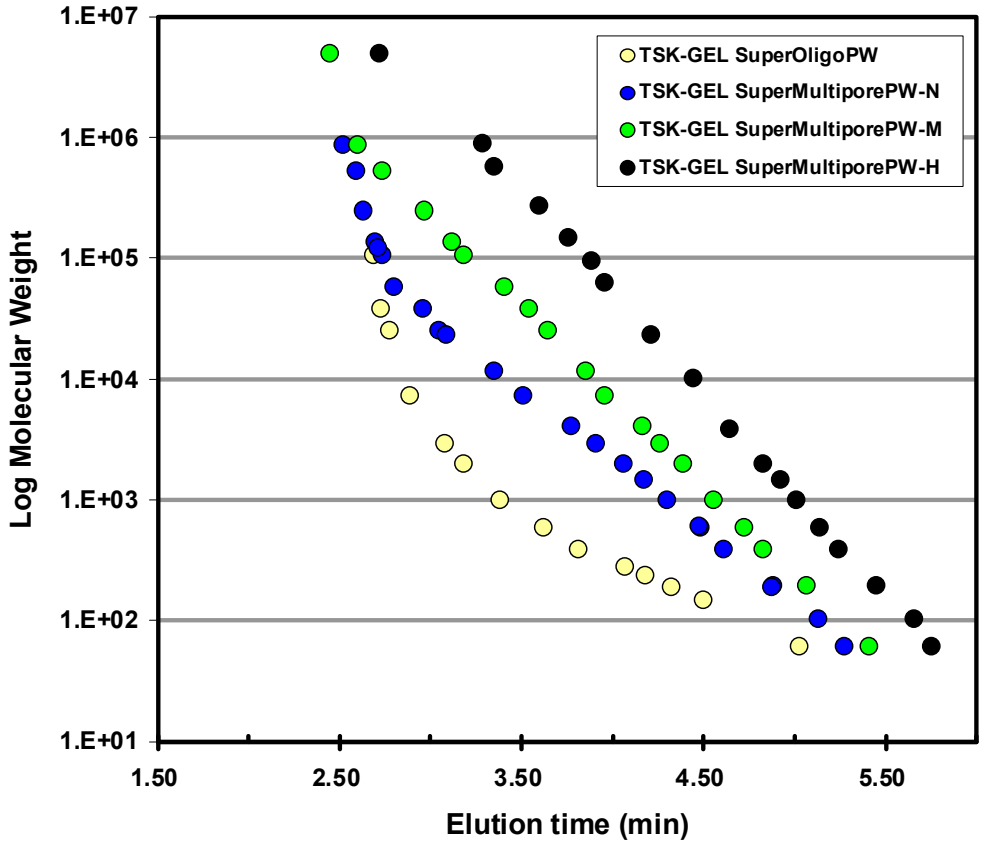
Properties of TSK-GEL SuperOligoPW & SuperMultiporePW columns

Table 1: Specifications

	TSKgel SuperOligoPW	TSKgel SuperMultiporePW-N	TSKgel SuperMultiporePW-M	TSKgel SuperMultiporePW-H
Base material	Polymethacrylate	Polymethacrylate	Polymethacrylate	Polymethacrylate
Particle size (µm)	3 (monodisperse)	4 (monodisperse)	5 (monodisperse)	8 (6 - 10)
Max. exclusion limit MW (PEO,PEG/H_2O)	4,000 - 8,000	100,000 - 150,000	600,000 - 1,500,000	
Range of applicable MW (PEO,PEG/H_2O)	100 - 3,000	300 - 50,000	500 - 1,000,000	1,000 - 10,000,000
Theoretical plates/column	>16,000	>16,000	>12,000	>7,000
Pressure drop (MPa)	4 - 6	4 - 6	2 - 4	1 - 2
Column size	6.0mm ID x 15cm	6.0mm ID x 15cm	6.0mm ID x 15cm	6.0mm ID x 15cm
Guard column	4.6mm ID x 3.5cm	4.6mm ID x 3.5cm	4.6mm ID x 3.5cm	4.6mm ID x 3.5cm

Properties of TSK-GEL SuperOligoPW & SuperMultiporePW columns

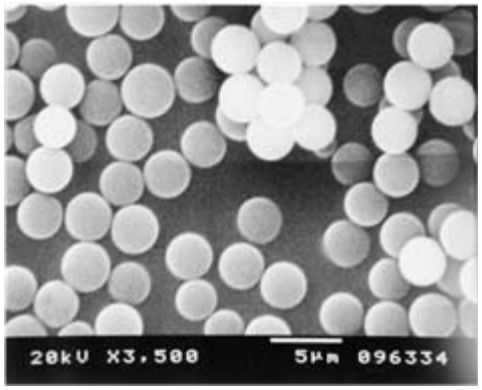
Figure 1: Calibration curves of aqueous SEC columns



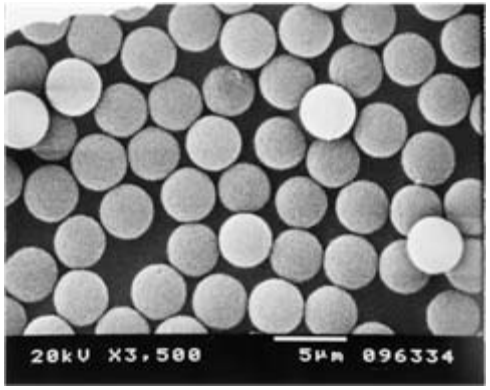
Columns:	TSKgel SuperOligoPW, 6.0mm ID x 15cm
	TSKgel SuperMultiporePW-N, 6.0mm ID x 15cm
	TSKgel SuperMultiporePW-M, 6.0mm ID x 15cm
	TSKgel SuperMultiporePW-H, 6.0mm ID x 15cm
Mobile phase:	H_2O
Flow rate:	0.60mL/min
Detection:	RI
Temperature:	25°C
Samples:	PEO, PEG and ethylene glycol

Properties of TSK-GEL SuperOligoPW & SuperMultiporePW columns

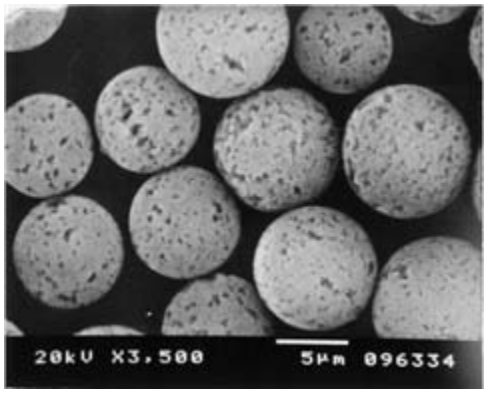
Figure 2: SEM photograph of packing material



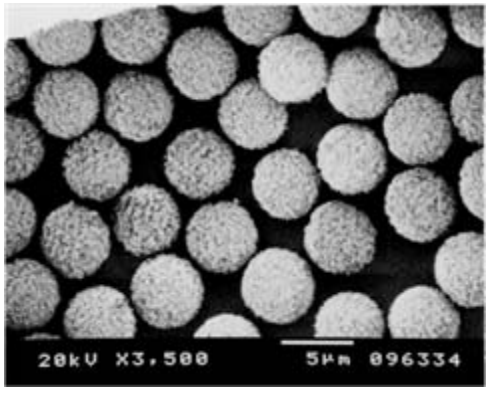
TSKgel SuperOligoPW



TSKgel SuperMultiporePW-N



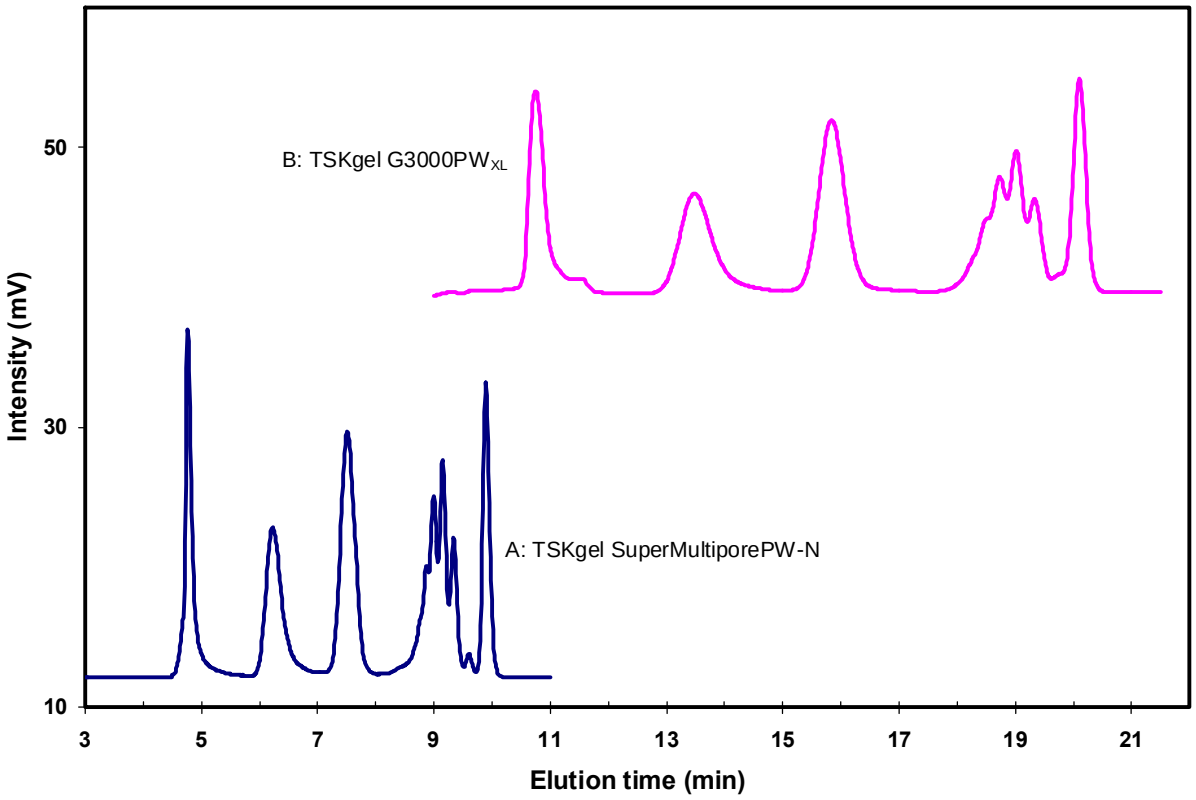
TSKgel SuperMultiporePW-H



TSKgel SuperMultiporePW-M

Evaluation of separation performance

Figure 3: Comparison with conventional columns

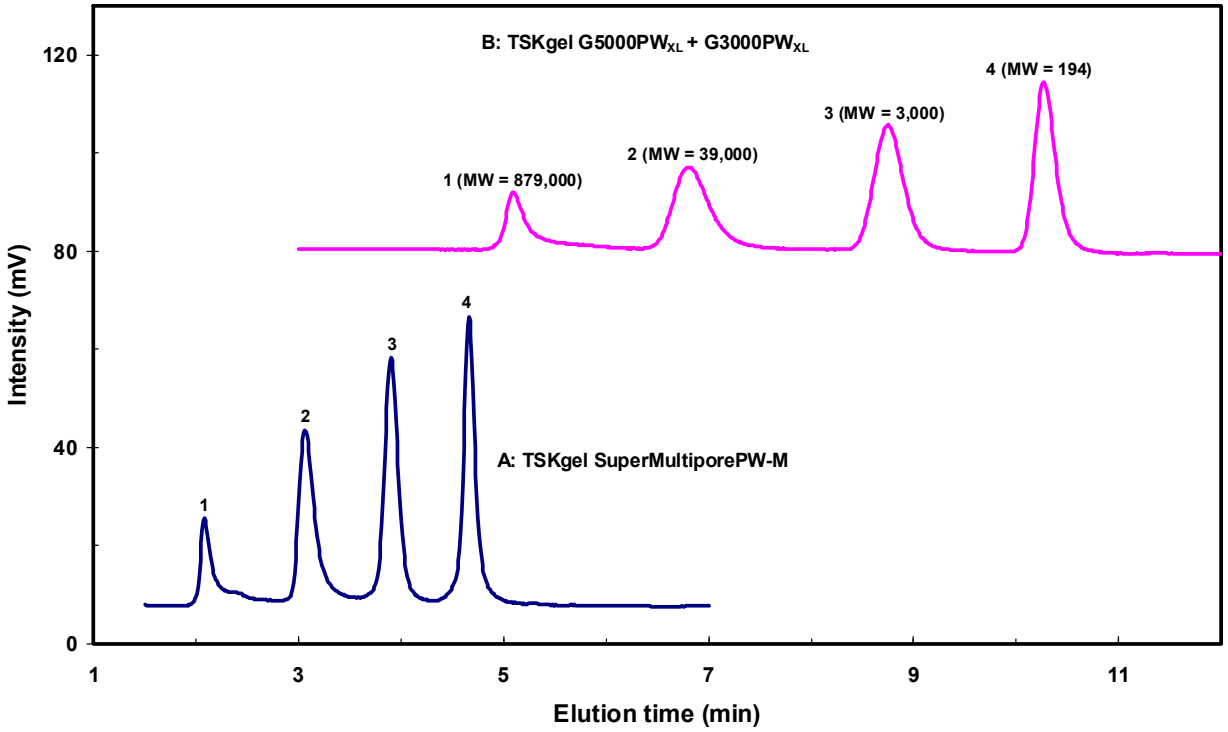


Columns:	TSKgel SuperMultiporePW-N, 6.0mm ID x 15cm x 2 TSKgel G3000PW$_{XL}$, 7.8mm ID x 30cm x 2
Mobile phase:	H_2O
Flow rate:	A: 0.6mL/min B: 1.0mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	A: 20µL B: 100 µL
Samples:	mixture of PEO and PEG

Rs	PW$_{XL}$	SuperMultiporePW-M
Peak 1/Peak 2	3.45	4.25
Peak 2/Peak 3	3.29	3.17
Peak 3/Peak 4	3.30	3.39

Evaluation of separation performance

Figure 3: Comparison with conventional columns, cont.



Columns:	TSKgel SuperMultiporePW-M, 6.0mm ID x 15cm x 2 TSKgel G3000PW$_{XL}$, 7.8mm ID x 30cm x 2 TSKgel G5000PW$_{XL}$, 7.8mm ID x 30cm x 2
Mobile phase:	H_2O
Flow rate:	A: 0.6mL/min B: 1.0mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	A: 20µL B: 100 µL
Samples:	mixture of PEO and PEG

Rs	PW$_{XL}$	SuperMultiporePW-M
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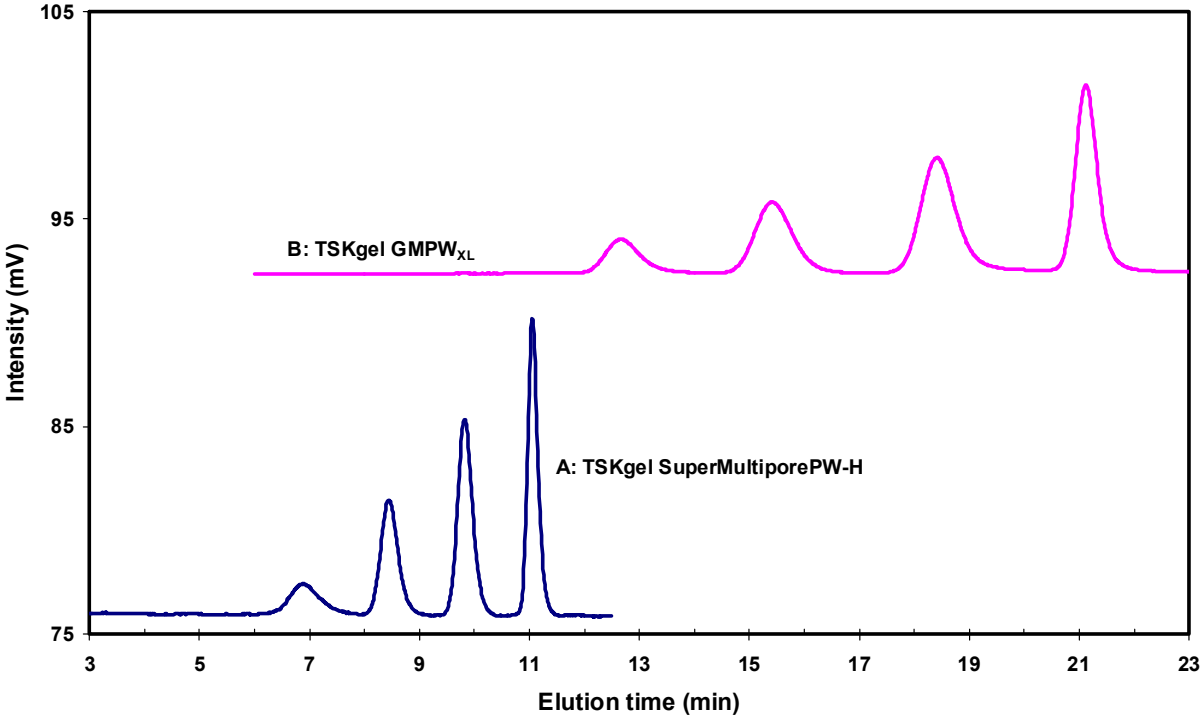
Evaluation of separation performance

Figure 3: Comparison with conventional columns, cont.

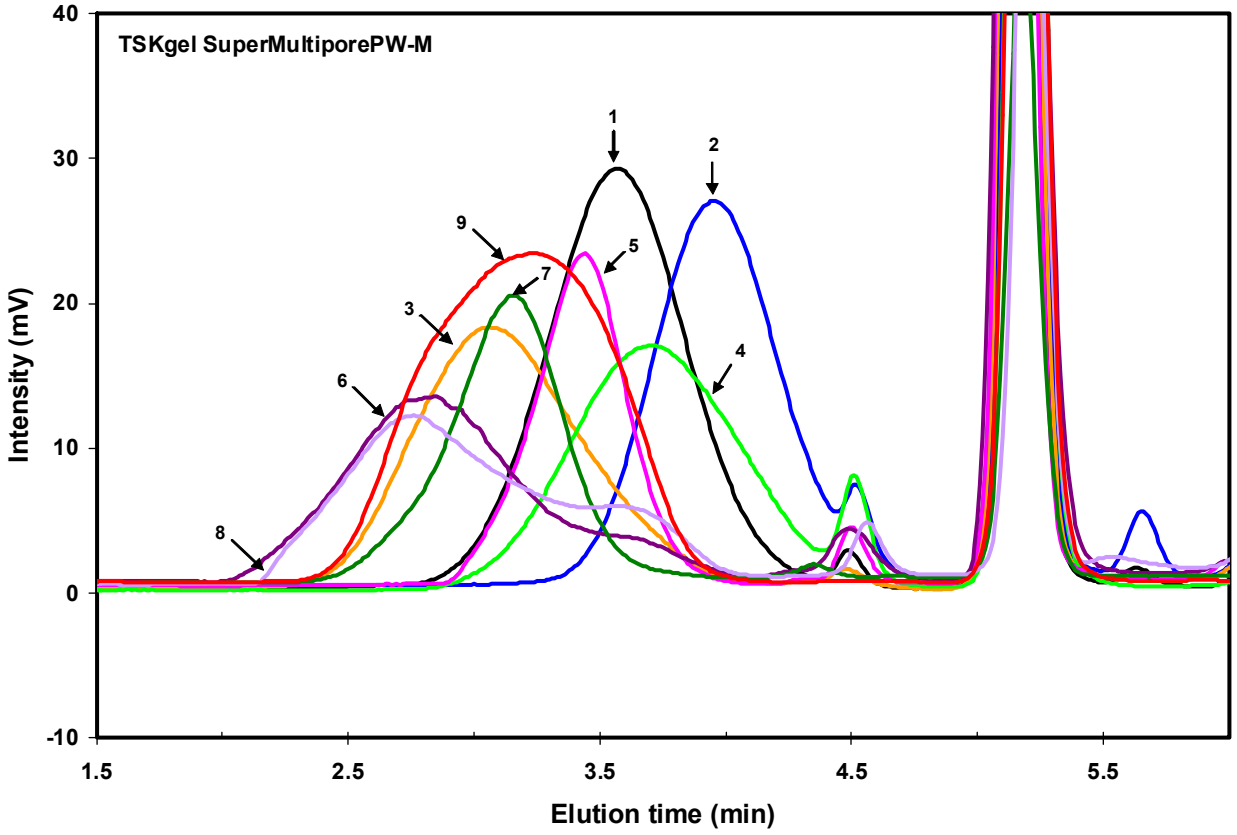


Columns:	TSKgel SuperMultiporePW-H, 6.0mm ID x 15cm x 2 TSKgel GMPW$_{XL}$, 7.8mm ID x 30cm x 2
Mobile phase:	H_2O
Flow rate:	A: 0.6mL/min B: 1.0mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	A: 20µL B: 100 µL
Samples:	mixture of PEO and PEG

Rs	PW$_{XL}$	SuperMultiporePW-M
Peak 1/Peak 2	3.45	4.25
Peak 2/Peak 3	3.29	3.17
Peak 3/Peak 4	3.30	3.39

Applications: Analysis of synthetic water-soluble polymers

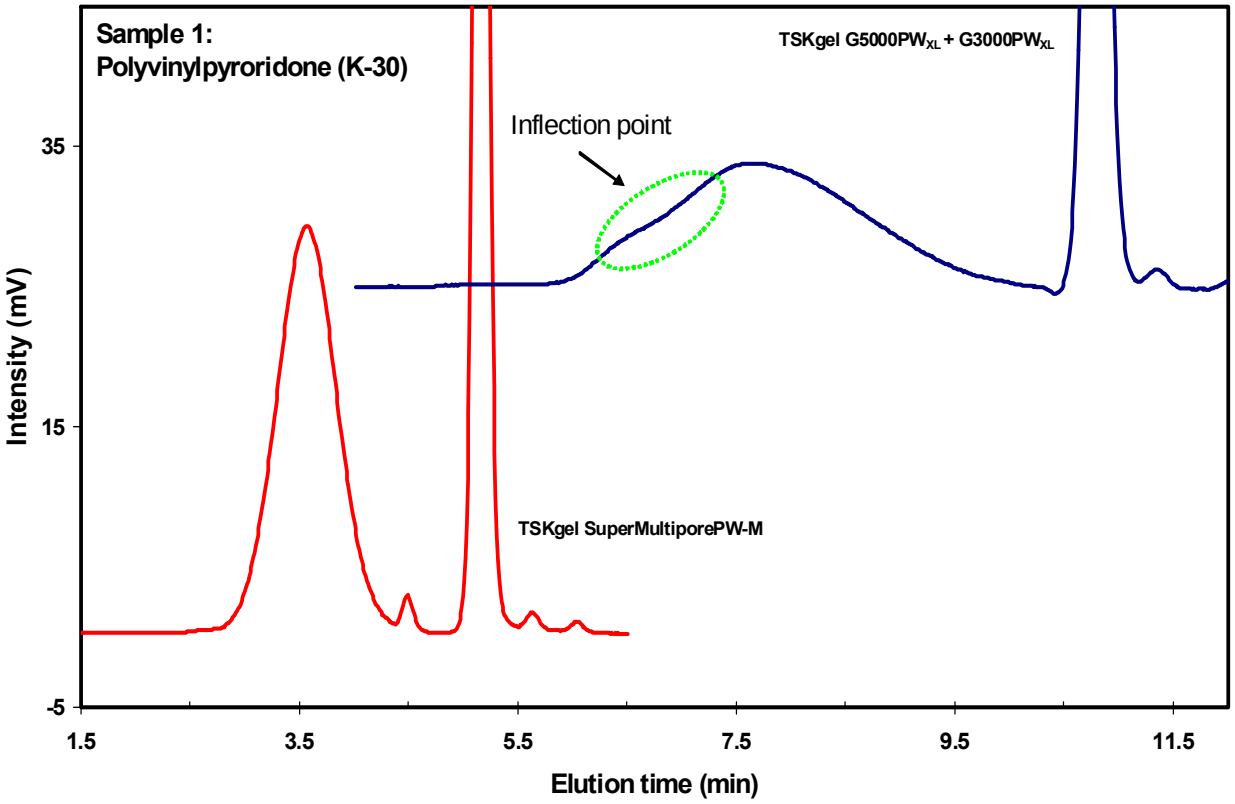
Figure 4: Separation of water-soluble synthetic polymers



Column: TSKgel SuperMutiporePW-M, 6.0mm ID x 15cm x 1
Mobile phase: 0.1mol/L $NaNO_3$
Flow rate: 0.6mL/min
Detection: RI
Temperature: 25°C
Injection vol.: 35µL
Samples:
1. polyvinylpyroridone (K-30)
2. polyvinylpyroridone (K-15)
3. hydroxypropylcellulose (MW 100K)
4. hydroxypropylcellulose (MW 100K)
5. chondroitin sulfate sodium salt
6. carboxymethyl cellulose
7. arabic gum
8. poly(sulfopropyl methacrylate-co-acrylic acid)
9. dextran (200K)

Applications: Analysis of synthetic water-soluble polymers

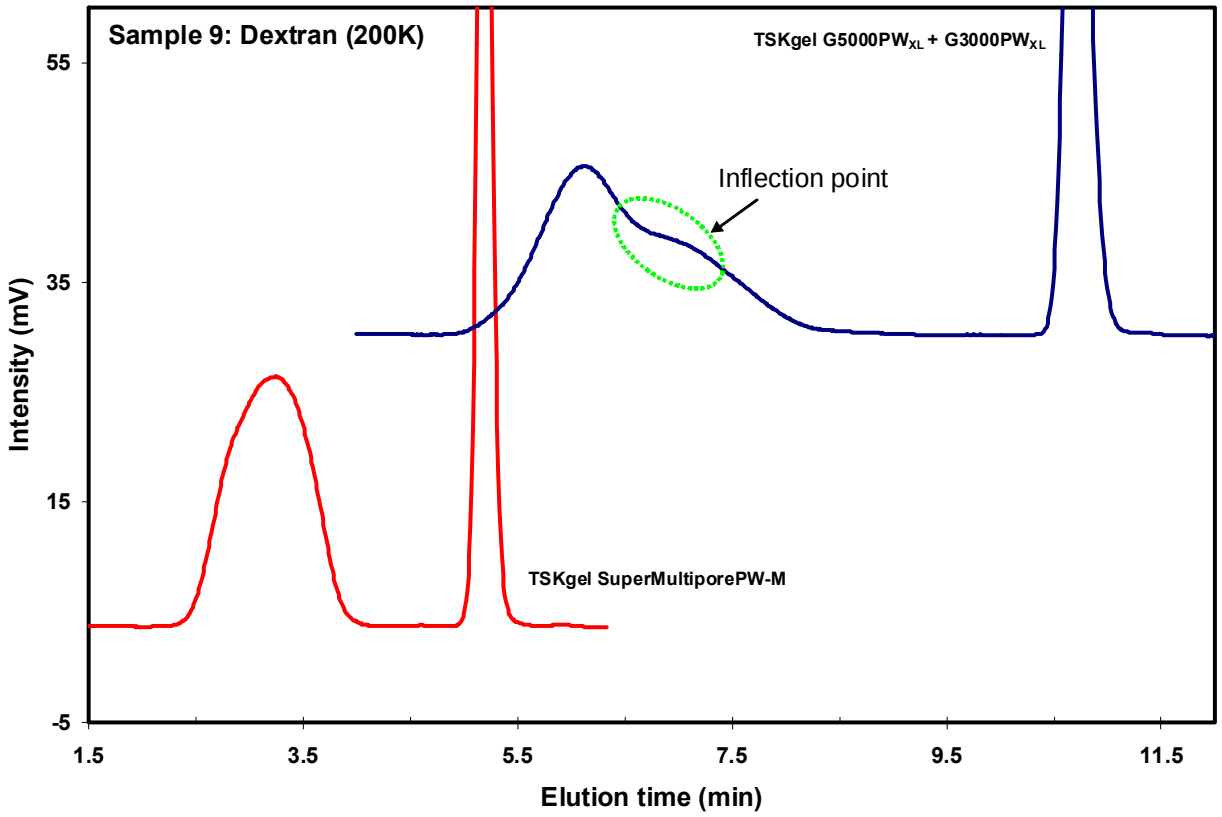
Figure 4: Separation of water-soluble synthetic polymers, cont.



Columns:	TSKgel SuperMutiporePW-M, 6.0mm ID x 15cm x 1 TSKgel G5000PW$_{XL}$ + G3000PW$_{XL}$, 6.0mm ID x 15cm x 2
Mobile phase:	0.1mol/L $NaNO_3$
Flow rate:	0.6mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	35µL
Samples:	1. polyvinylpyroridone (K-30) 2. polyvinylpyroridone (K-15) 3. hydroxypropylcellulose (MW 100K) 4. hydroxypropylcellulose (MW 100K) 5. chondroitin sulfate sodium salt 6. carboxymethyl cellulose 7. arabic gum 8. poly(sulfopropyl methacrylate-co-acrylic acid) 9. dextran (200K)

Applications: Analysis of synthetic water-soluble polymers

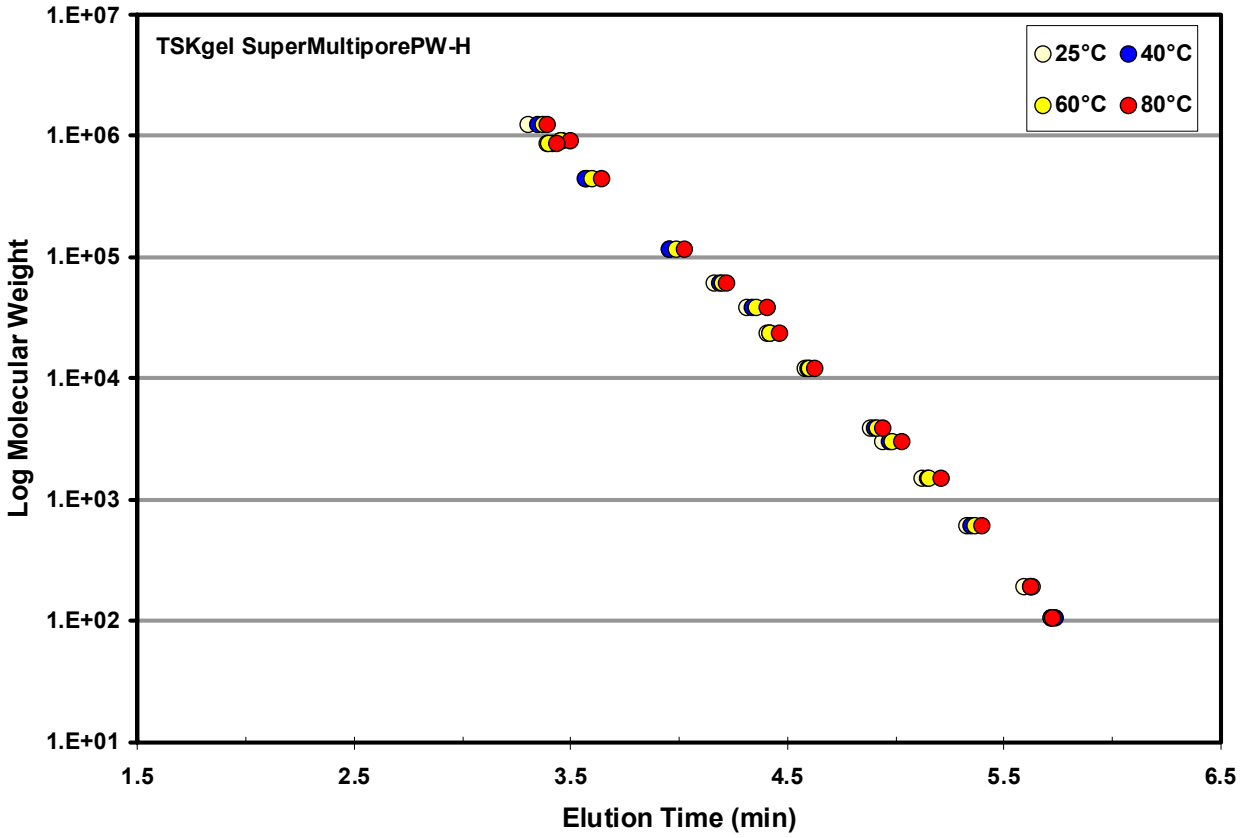
Figure 4: Separation of water-soluble synthetic polymers, cont.



Columns:	TSKgel SuperMutiporePW-M, 6.0mm ID x 15cm x 1 TSKgel G5000PW$_{XL}$ + G3000PW$_{XL}$, 6.0mm ID x 15cm x 2
Mobile phase:	0.1mol/L $NaNO_3$
Flow rate:	0.6mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	35µL
Samples:	1. polyvinylpyroridone (K-30) 2. polyvinylpyroridone (K-15) 3. hydroxypropylcellulose (MW 100K) 4. hydroxypropylcellulose (MW 100K) 5. chondroitin sulfate sodium salt 6. carboxymethyl cellulose 7. arabic gum 8. poly(sulfopropyl methacrylate-co-acrylic acid) 9. dextran (200K)

Applications: Analysis of synthetic water-soluble polymers

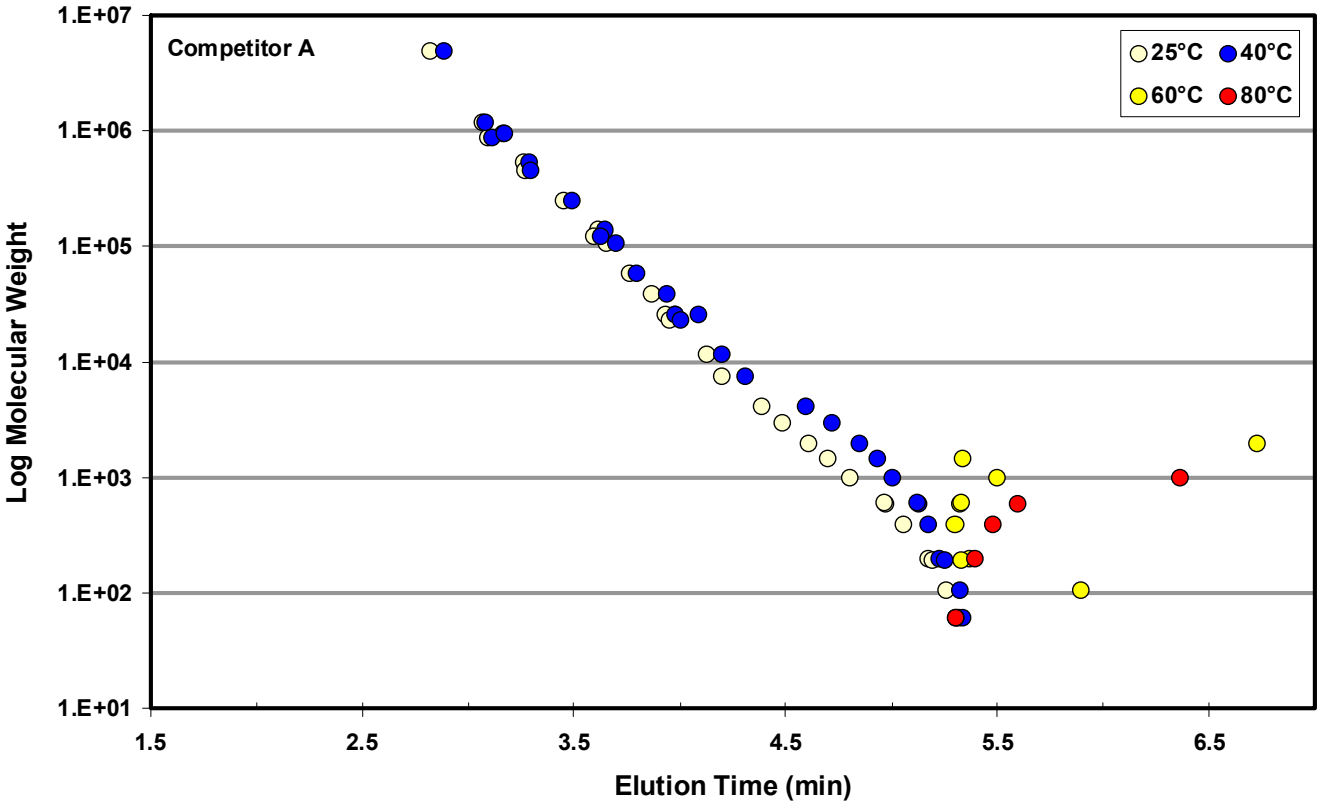
Figure 5: Effect of temperature on elution time



Analysis conditions were the same as in figure 1.

Applications: Analysis of synthetic water-soluble polymers

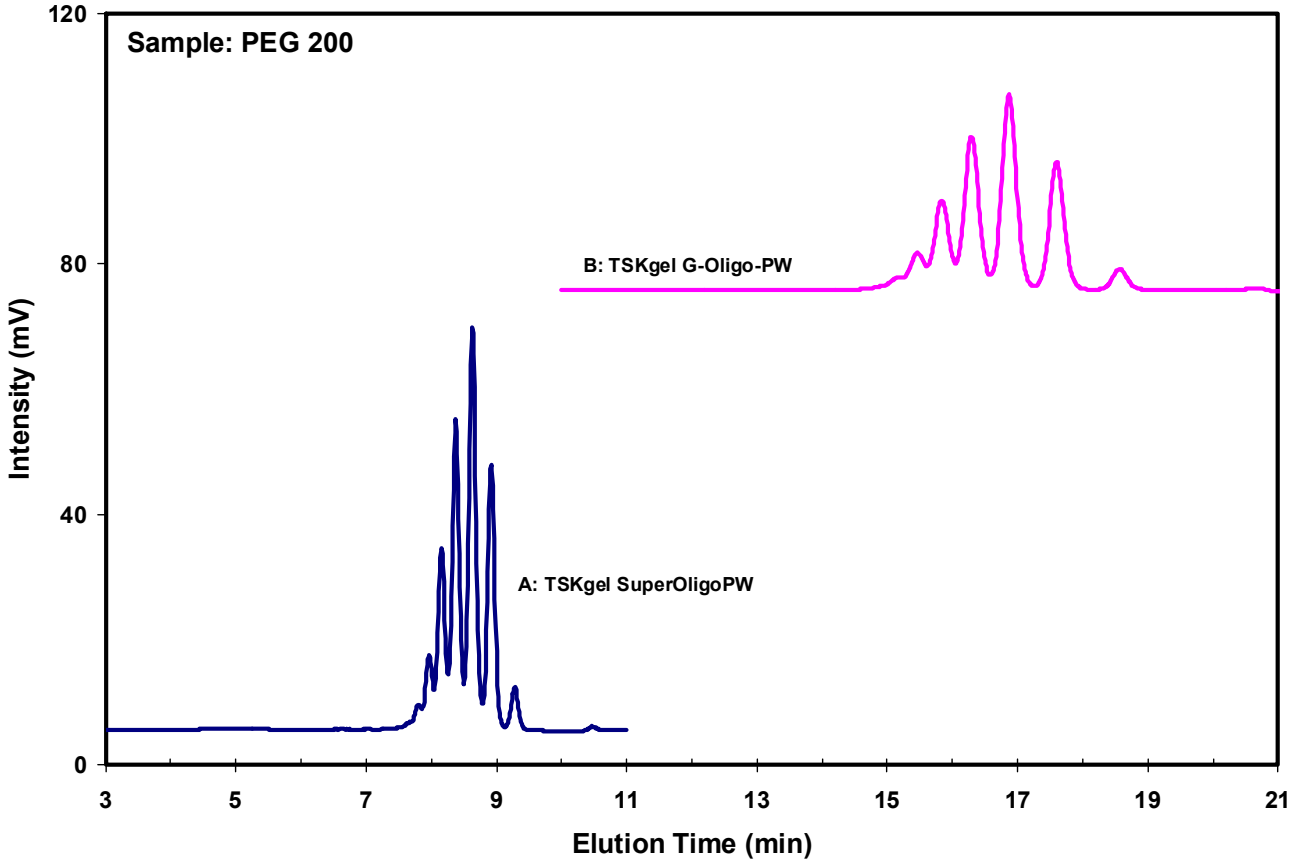
Figure 5: Effect of temperature on elution time, cont.



Analysis conditions were the same as in figure 1.

Applications using TSKgel SuperOligoPW column

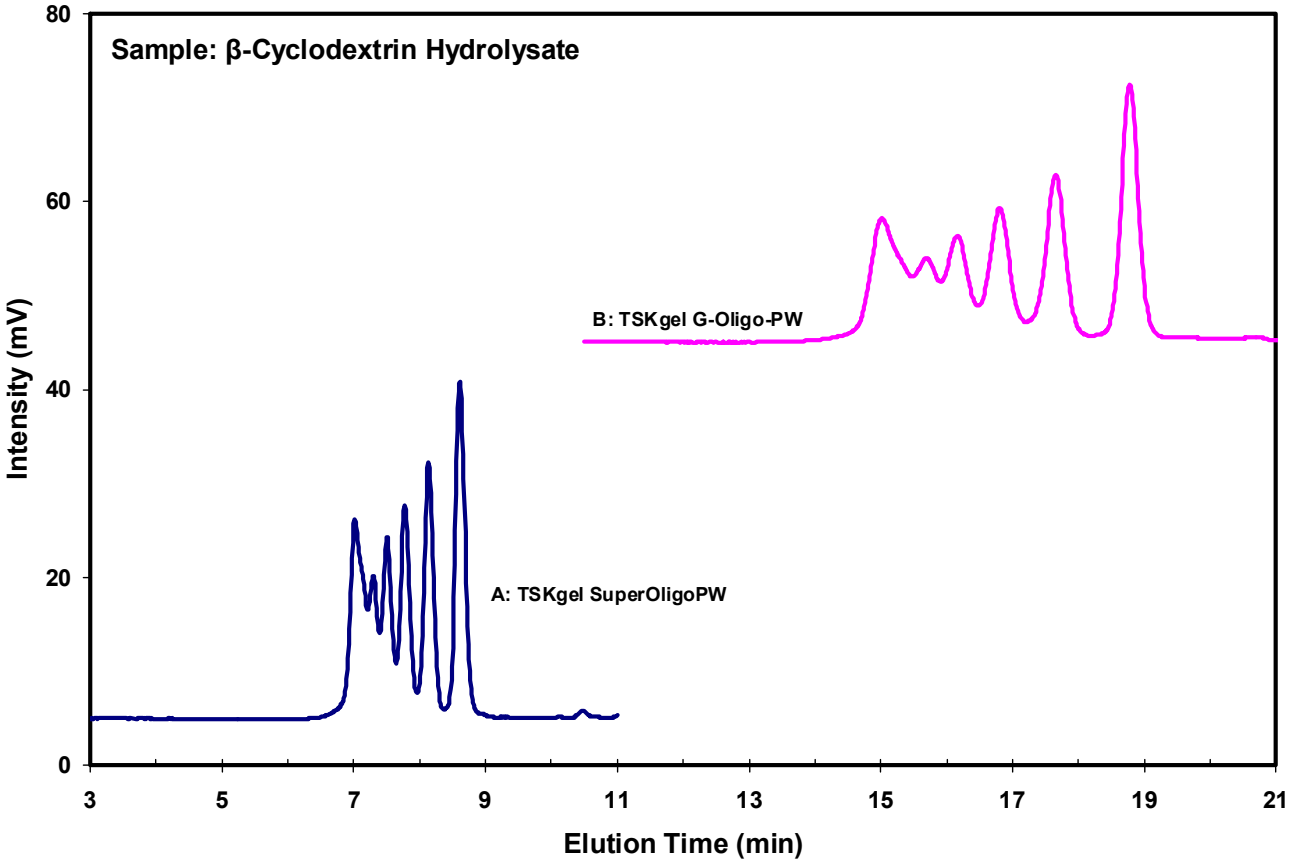
Figure 6: TSKgel SuperOligoPW column applications



Columns: TSKgel SuperOligoPW, 6.0mm ID x 15cm x 2
TSKgel G-Oligo-PW, 7.8mm ID x 30cm x 2
Mobile phase: H_2O
Flow rate: A: 0.6mL/min B: 1.0mL/min
Detection: RI
Temperature: 25°C
Injection vol.: A: 20µL B: 100 µL

Applications using TSKgel SuperOligoPW column

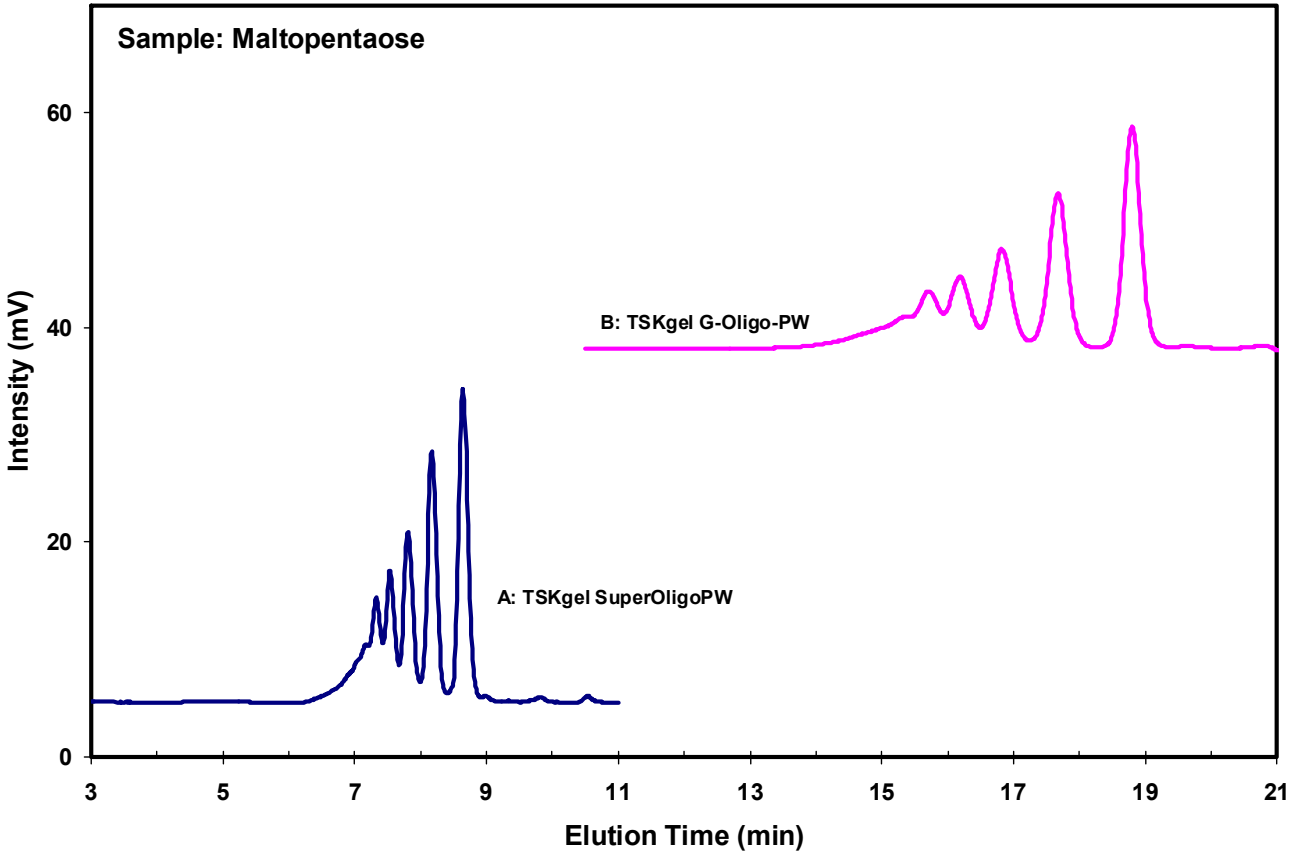
Figure 6: TSKgel SuperOligoPW column applications, cont.



Columns:	TSKgel SuperOligoPW, 6.0mm ID x 15cm x 2 TSKgel G-Oligo-PW, 7.8mm ID x 30cm x 2
Mobile phase:	H_2O
Flow rate:	A: 0.6mL/min B: 1.0mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	A: 20µL B: 100 µL

Applications using TSKgel SuperOligoPW column

Figure 6: TSKgel SuperOligoPW column applications, cont.



Columns:	TSKgel SuperOligoPW, 6.0mm ID x 15cm x 2 TSKgel G-Oligo-PW, 7.8mm ID x 30cm x 2
Mobile phase:	H_2O
Flow rate:	A: 0.6mL/min B: 1.0mL/min
Detection:	RI
Temperature:	25°C
Injection vol.:	A: 20µL B: 100 µL

Conclusions

- We developed a series of novel SEC semi-micro columns, TSK-GEL SuperMultiporePW, packed with spherical monodisperse polymethacrylate particles, each containing a wide range of pore sizes.
- TSK-GEL SuperMultiporePW columns exhibited high resolution and good linearity of the calibration curves across a wide range of molecular mass of PEO and PEG standards in aqueous eluent.
- Chromatograms obtained on TSK-GEL SuperMultiporePW columns did not show inflection points, thus allowing better accuracy and reproducibility when determining the molecular mass distribution of water soluble polymers.
- Various polymers and oligomers were analyzed on the TSK-GEL SuperMultiporePW and SuperOligoPW semi-micro columns. Compared with conventional SEC columns, the TSK-GEL columns produced superior results in half the run times and with 1/6th of the solvent consumption.