TOSOH

EcoSEC GPC Systems

Experts in Chromatography

2018 Product Guide

EcoSEC® GPC Systems

- TOYOPEARL® Bulk Resin
- TSKgel® Bulk Resin
- Ca^{++}Pure-HA™ Resin
- TSKgel HPLC Columns

TOSOH BIOSCIENCE

A Tradition of GPC Excellence

Tosoh established itself as a world leader in the field of polymer analysis in 1971 with the introduction of TSKgel gel permeation chromatography (GPC) columns. The following year, Tosoh launched a dedicated instrument for GPC analysis. Since that first instrument there have been 7 generations of GPC systems with temperature control up to 50 °C, as well as 2 generations of high temperature GPC systems for analysis up to 220 °C. Today Tosoh continues a tradition of GPC excellence with a 3rd generation high temperature GPC system: the EcoSEC High Temperature GPC System.

1972: HLC-801 GPC System

- First GPC instrument from Tosoh
- "All-in-one" concept incorporated

1977: HLC-811 HT GPC System

- First Tosoh high temperature GPC System

1982: HLC-802A GPC System

- Dual Flow refractive index (RI) detector
- "Stable RI Baseline" implemented

1986: HLC-8020 GPC System

- Temperature controlled pump system
- "High Reproducibility" obtained

1993: HLC-8120 GPC System

- First semi-micro GPC columns from Tosoh
- "Semi-micro" concept incorporated

1998: HLC-8121 HT GPC System

- 2nd generation Tosoh high temperature GPC System

2008: EcoSEC GPC System

- 7th generation Tosoh GPC System
- Released in overseas market

2013: EcoSEC High Temperature GPC System

- 3rd generation Tosoh high temperature GPC System
- Released in overseas market

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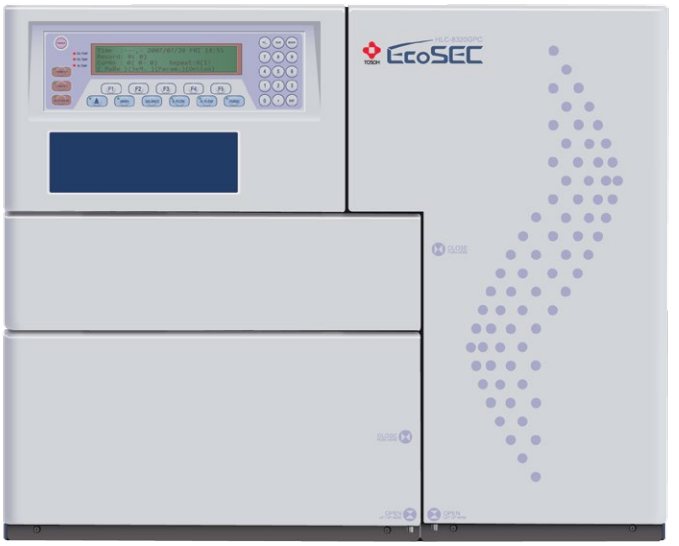


History of Performance: EcoSEC GPC System

An all-in-one system that delivers:

- **Superior Performance**
  - Unmatched baseline stability due to unique dual flow RI detector design
  - High degree of precision in retention time and molar mass determination due to advanced temperature controlled pumps and column oven
  - Exceptional reproducibility day to day, system to system, and site to site
- **Increased throughput**
  - Stable RI baseline with low baseline drift obtained within just 90 minutes of startup (in THF)
  - Unattended operation with built-in autosampler
- **Unparalleled versatility**
  - Column switching valve reduces time between column changes and rapidly establishes a stable baseline (within 15 minutes)
  - Easy to use, intuitive software specific to GPC analysis
  - Optional UV detector for measurement of UV-absorbing polymers
  - Compatible with external viscometry and light scattering detectors
- **Optional semi-micro columns**
  - 50% reduction in run times and solvent cost savings of 85% due to low dead volume design
  - TSKgel SuperMultiporeHZ columns are packed with particles synthesized with a range of pore sizes, resulting in no inflection points in the calibration curve. The lack of inflection points allows better accuracy and reproducibility when determining the molar mass distribution of polymers.

Call customer service: 866-527-3587,
technical service: 800-366-4875, option #4

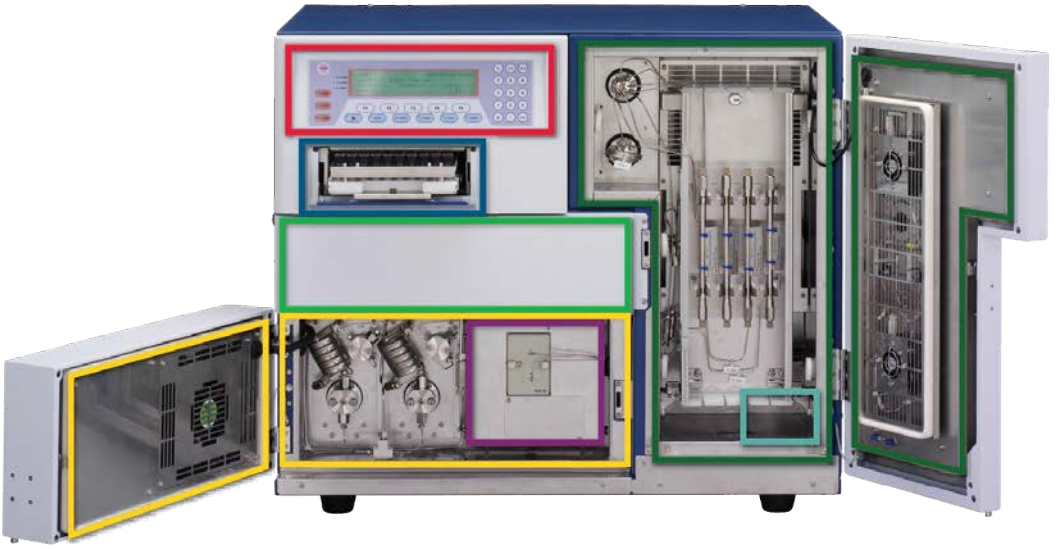
Meeting the Demands of High Temperature Analysis: EcoSEC High Temperature GPC Sysem

Only system on the market that offers the combination of:

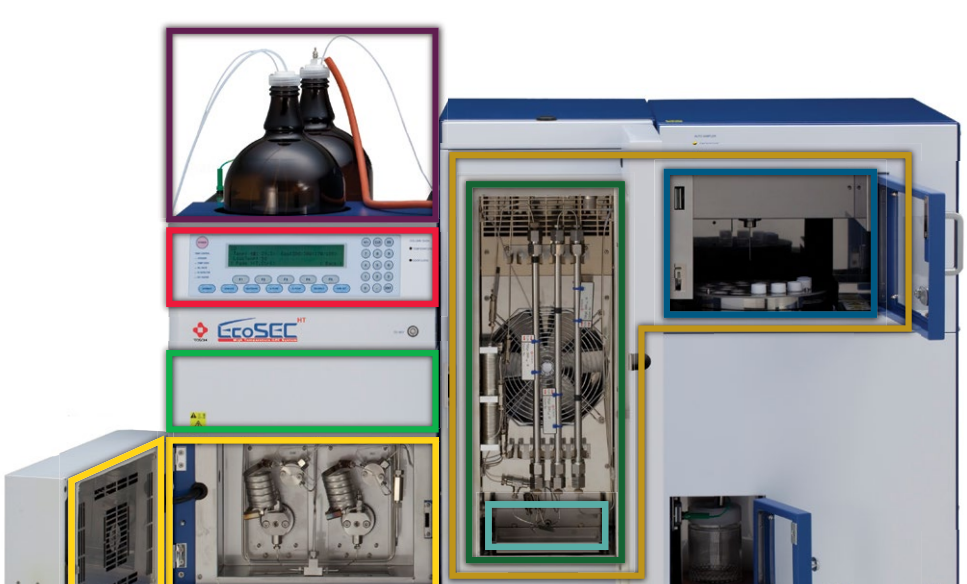
- **All-in-one system**
- **Dual flow pump RI**
- **Temperatures up to 220 °C**

Incorporates proven design and technology used in EcoSEC GPC System:

- dual flow RI detector design
- dual pump system
- spacious column oven
- intuitive software
- automatic sample injection



Component	Description	Benefit
All-in-one design	The EcoSEC GPC System is designed with low dead volume (<20 µL), temperature controlled pumps, and dual flow RI detection.	Improved resolution and molar mass distribution accuracy, excellent flow rate precision regardless of changes in laboratory temperature, and unmatched baseline stability.
Control panel	Allows the system to be controlled manually and at the discretion of the operator.	Saves time by controlling a series of operations without the use of the computer or software.
Autosampler	100 sample capacity, 1 to 1,500 µL per injection.	Automatic sample injection for unattended, around the clock operation.
Purge unit and degasser	20 and 40 mL solvent volume; variable degassing capacity (for semi-micro or 30 cm column).	Saves time with rapid solvent changes via purge valve eliminating solvent replacement and other time-consuming manual operations.
Temperature controlled pumps	Pump heads and solvent lines are maintained at a constant temperature.	Improves baseline stability by removing the effect of temperature fluctuations. This results in consistent and accurate flow rates and reproducible molar mass determinations.
Column oven	Engineered for precise (± 0.02 °C) column temperature; oven can accommodate up to 8, 30 cm length columns.	Constant column temperature ensures precise and reproducible molar mass determinations.
RI detector	Low dead volume flow cell, 2.5 µL. Solvent flows through a separate reference cell.	Enhanced baseline stability from dual flow cell RI detector.
UV detector (optional)	Low dead volume flow cell, 2 µL. Wavelength range from 195-350 nm.	Option for measuring UV-absorbing polymers.
Light Scattering detector (optional)	Various technologies available.	Absolute molar mass and polymer size determination.
Viscometry detector (optional)	Various designs available.	Universal calibration, Mark-Houwink plot, determination of intrinsic viscosity and polymer size.



Component	Description	Benefit
Solvent holder	Maintains a constant temperature of 40 °C.	Prevent possible solvent freezing.
Control panel	Allows the system to be controlled manually and at the discretion of the operator.	Saves time by controlling a series of operations without the use of the computer or software.
Temperature controlled pumps	Pump heads and solvent lines are maintained at a constant temperature.	Improves baseline stability by removing the effect of temperature fluctuations. This results in consistent and accurate flow rates and reproducible molar mass determinations.
Column oven	Maintains 40 - 220 °C. Can accommodate up to 8, 30 cm length columns.	Constant column temperature ensures precise and reproducible molar mass determinations.
Autosampler	24 sample capacity. Temperature controlled by aluminum block from 40 - 220 °C.	Precise injection volume. Variety of loop sizes. Door is locked under sampling operation for safety.
RI detector	Solvent flows through a separate reference cell. 10 µL volume flow cell.	Temperature controlled, stable baseline, quick response, low noise. Enhanced baseline stability from dual flow cell RI detector.
Purge unit and degasser	Variable degassing capacity. Temperature controlled degassing unit and auto purge function.	Saves time with rapid solvent changes via purge valve eliminating solvent replacement and other time-consuming manual operations.
Fully integrated temperature controlled system	High temperature homogeneously maintained in autosampler, column oven and RI detector, with no transfer line.	Reduces the risks of cold spots that cause sample precipitation in the system.


EcoSEC

EcoSEC GPC System

Engineered to deliver the following:

Superior Performance

- Baseline Stability
- Reproducibility
- Reliability

Unparalleled Versatility

- Ease of Use
- All-in-One Design

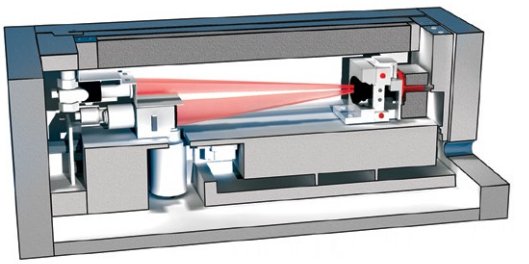
Increased Throughput

- Lower Operating Costs

Superior Performance

Unmatched Baseline Stability

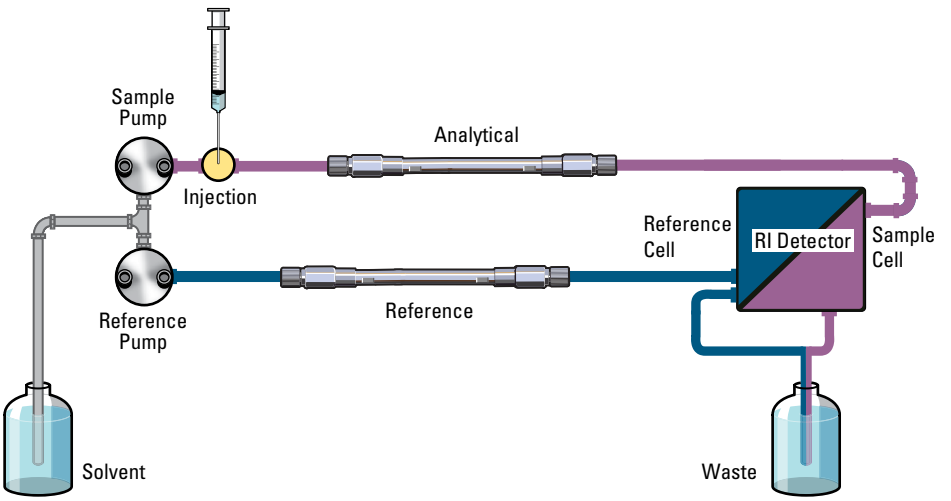
- Dual flow RI cell and pump design
- Continuous correction of RI baseline drift due to solvent instability
- Improved molar mass precision and accuracy
- Rapid baseline stability at startup



Dual Flow Pump Design

The EcoSEC GPC System has a unique dual flow design which includes the use of two pumps. Figure 1 demonstrates the flow paths of the sample and reference pumps. The sample pump flows solvent from the solvent reservoir through the following system components in sequence: autosampler, analytical column, sample side of RI detector cell, and waste container. The solvent flows via the reference pump from the solvent reservoir through a reference column, the reference side of the RI detector cell, and then the waste container.

Figure 1: Flow paths of sample and reference pumps in the EcoSEC GPC System



Dual Flow Refractive Index Detector

The refractive index detector in the EcoSEC GPC System is unlike any other refractive index detector on the market due to its unique dual flow design. The EcoSEC GPC System RI flow cell is constructed in such a way that there are two sides: (1) the reference side, containing a flowing stream of pure solvent; and (2) a sample side, containing a flowing stream of analyte in the same solvent as in the reference side (Figure 2).

The unique dual flow design of the EcoSEC GPC System results in superb RI baseline stability and reduced RI baseline drift. In a conventional RI detector, over time, the refractive index of the stagnant pure solvent in the reference side will slowly change and the two photodiodes will no longer produce equal signals, thus the contents of the reference and sample sides have different refractive indices and will produce a voltage difference similar to that of an analyte in solution. For example, the refractive index of THF slowly alters over time, due to the buildup of peroxide-related compounds, resulting in baseline drift (Figure 3). The dual flow design of the RI detector in the EcoSEC GPC System compensates for the changes in refractive index of the solvent over time by continuously flowing pure solvent through the reference side of the flow cell.

Another benefit of the dual flow cell is rapid attainment of baseline stability when the instrument is first started, as purging is not required. A stable baseline can be achieved by flowing only 50 mL of solvent through the instrument. Additionally, the reference side mobile phase can be sent to waste or recycled back to the solvent bottle.

Figure 2: Depiction of dual flow RI detector in the EcoSEC GPC System, showing the compensation of the changes in refractive index of the solvent over time

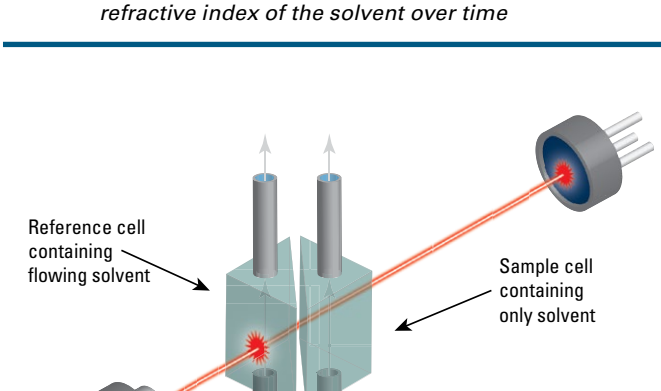
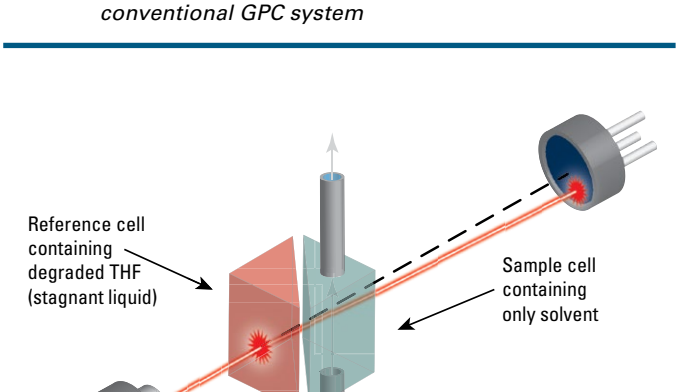


Figure 3: Depiction of RI detector flow cell showing the effects of THF degradation in the stagnant reference side of a conventional GPC system



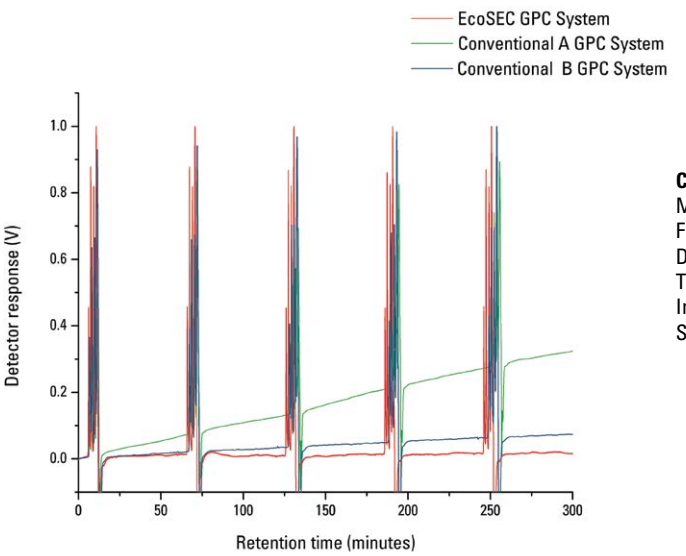
Comparison of Baseline Stability

The EcoSEC GPC System offers unmatched baseline stability because it is the only GPC system which uses a dual flow refractive index detector and temperature controlled pumps. Baseline stability is essential for the accurate calculation of polymer molar mass averages. For example, computer simulations predict a polymer with a polydispersity index (*PDI*) of 5 will have an 18% error for M_z if baseline instability leads to a 4% error in peak width determination. In addition, a 2% uncertainty in baseline height will result in a 20% error in M_z.[1]

A study was done to demonstrate the superb baseline stability of the EcoSEC GPC System compared to that of two conventional GPC systems using both 15 cm and 30 cm columns over a five hour time period. The figures below demonstrate that the EcoSEC GPC System maintains the efficiency of semi-micro columns and maintains a stable RI baseline when both conventional and semi-micro GPC columns are used.

As shown in Figures 4A and 4B, five consecutive injections of polystyrene standards with run times deliberately extended to one hour without auto zeroing the detectors between injections, resulted in an extremely stable baseline with low baseline drift on the EcoSEC GPC System and a significantly drifting baseline on the two conventional GPC systems. In comparison to the conventional GPC systems, the EcoSEC GPC System has both a lower baseline drift and a better signal to noise ratio.

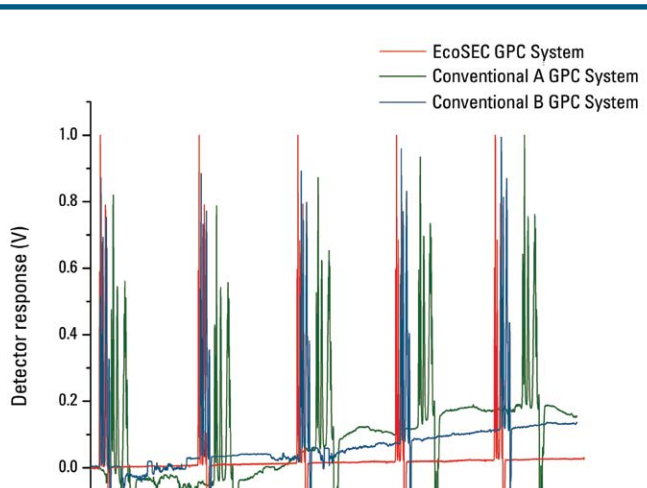
Figure 4A: Comparison of baseline drift of the dual flow refractive index detector of the EcoSEC GPC System and two conventional GPC systems using semi-micro columns



Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	polystyrene standards, PStQuick MP-M series

[1]Tcjir, W.J.; Rudin, A.; and Fyfe, C.A. Effects of data analysis on accuracy and precision of GPC results. *J. Polym. Sci. Polym. Phys. Ed.* **1982**, *20*, (8), 1443-1451.

Figure 4B: Comparison of baseline drift of the dual flow refractive index detector of the EcoSEC GPC System and two conventional GPC systems using conventional columns



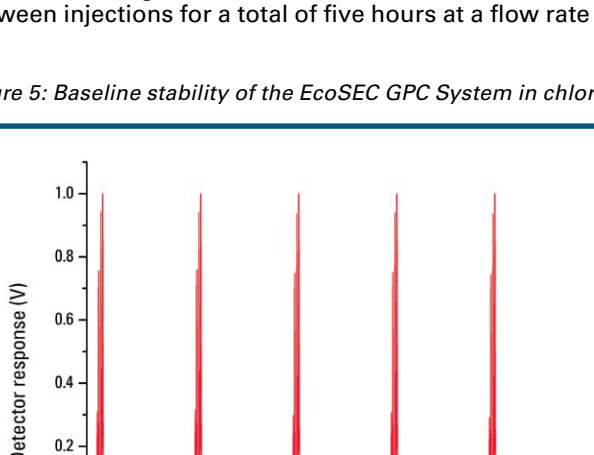
Column:	**TSKgel GMHXL-L, 5 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	polystyrene standards, PStQuick MP-M series

Baseline Stability in Various Solvents

The EcoSEC GPC System displays an extremely stable baseline with low baseline drift when analyzing polymers in neat, mixed, and complex solvent systems.

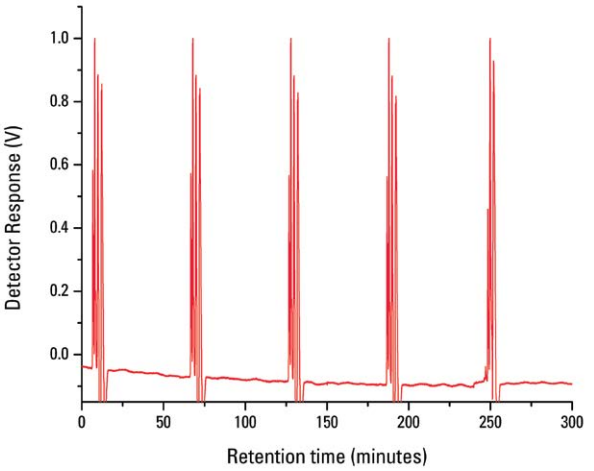
The following figures show five consecutive injections of polystyrene standards in chloroform (Figure 5), DMAc with 0.02 mol/L LiBr (Figure 6), and 95:5 Dichloromethane:HFIP with 5 mmol/L tetraethylammonium bromide (Figure 7) on semi-micro TSKgel GPC columns. The run times were deliberately extended to one hour without auto zeroing the detector between injections for a total of five hours at a flow rate of 0.35 mL/min.

Figure 5: Baseline stability of the EcoSEC GPC System in chloroform



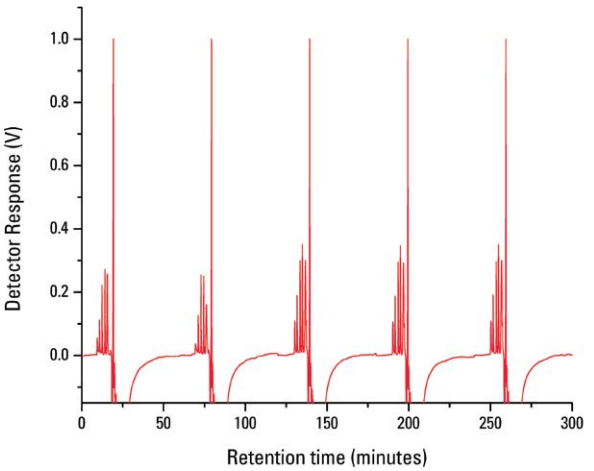
Column:	**TSKgel SuperHZM-M, 3 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	chloroform
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	polystyrene standards, PStQuick MP-M series

Figure 6: Baseline stability of the EcoSEC GPC System in DMAc with 0.02 mol/L LiBr



Column:	**TSKgel SuperHZM-M, 3 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	DMAc with 0.02 mol/L LiBr
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	polystyrene standards, PStQuick MP-M series

Figure 7: Baseline stability of the EcoSEC GPC System in 95:5 dichloromethane:HFIP with 5 mmol/L tetraethylammonium bromide



Column:	**TSKgel SuperHM-H, 3 µm, 6 mm ID × 15 cm × 2**
Mobile phase:	95:5 dichloromethane:HFIP with 5 mmol/L tetraethylammonium bromide
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	polystyrene standards, PStQuick B + PStQuick C

Comprehensive Temperature Control

Elution Time Precision

To assess the influence of environmental conditions within the laboratory on solvent flow, a study was done in which the EcoSEC GPC System and a conventional GPC system were placed in a chamber where the temperature was cycled between 23 °C and 26 °C. A series of 99 injections of polystyrene were made over a time period of ten hours. For each instrument the elution volume at peak maximum was measured; the resulting data is shown in Figures 8A and 8B below. The retention time drift of the EcoSEC GPC System was about 20% lower than that of the conventional GPC system.

The results shown demonstrate that the engineering design concepts of the EcoSEC GPC System result in a high degree of reproducibility of retention time and molar mass determination.

Figure 8A: Mobile phase delivery reproducibility of the EcoSEC GPC System with ambient temperature changes

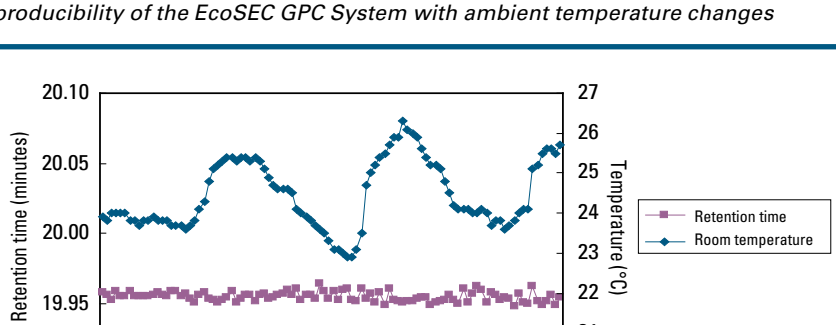
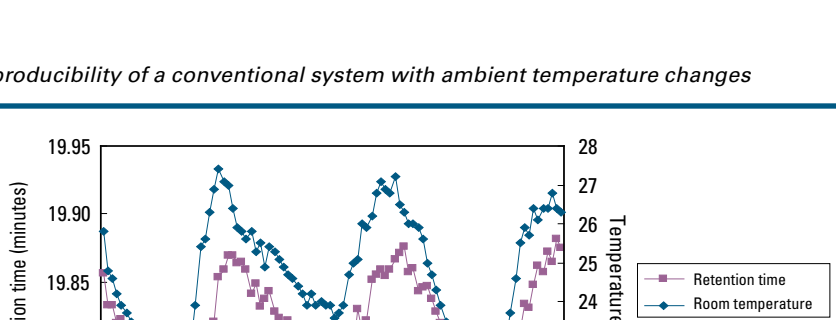


Figure 8B: Mobile phase delivery reproducibility of a conventional system with ambient temperature changes

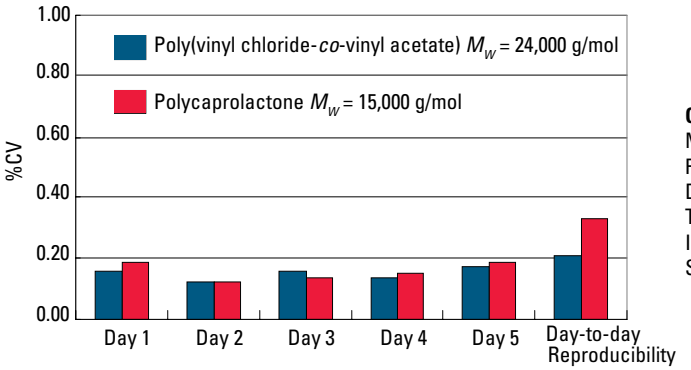


M_w Precision

Molar mass averages can be affected by changes in the environment and measuring conditions. Generally, these variations are the result of one or more factors including flow rate reproducibility, baseline drift and injection reproducibility. In addition to controlling column temperature, Tosoh engineers added temperature control for both pumps and inlet and outlet tubing on the EcoSEC GPC System to deliver top GPC analysis performance.

Figure 9 demonstrates the superiority of the EcoSEC GPC System for the determination of weight-average molar masses.

Figure 9: Reproducibility of M_w analysis

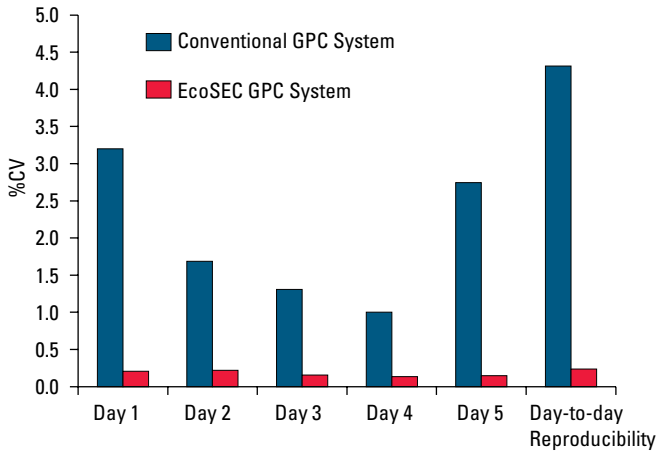


CV value less than 0.2% a day.
CV value less than 0.4% on different days.

Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Samples:	poly(vinyl chloride-*co*-vinyl acetate) and polycaprolactone

Figure 10 shows a comparison of M_w reproducibility for a sample injected 10 times a day for 5 days on the EcoSEC GPC System compared to a conventional GPC system. The reproducibility of the EcoSEC GPC System was superior by a factor of 3 to that of the conventional GPC system.

Figure 10: Comparing M_w reproducibility of the EcoSEC GPC System and a conventional GPC system



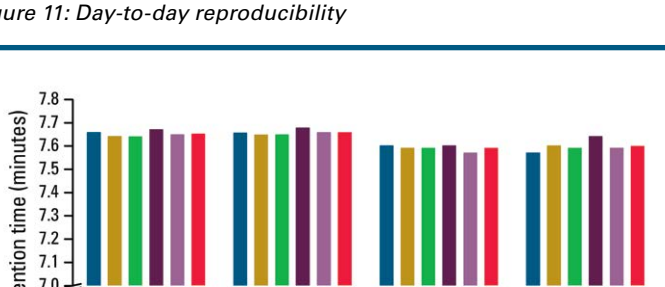
Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	10 µL
Samples:	poly(vinyl chloride-*co*-vinyl acetate)

System-to-System Reproducibility

Often measurements can be reproduced using the same equipment but results differ when an instrument from the same or another manufacturer is used. Among the system-specific factors which can influence the results of GPC analysis, fluctuations in elution time, in particular, can have a significant effect.

A study was performed using a polydisperse poly(vinyl chloride-*co*-vinyl acetate) sample run on four different EcoSEC GPC Systems by different operators to assess system reproducibility. The results are shown in Figure 11. The high precision of the EcoSEC GPC System results in minimal variation among instruments and from day-to-day.

Figure 11: Day-to-day reproducibility



Four EcoSEC GPC Systems, 4 operators,
4 column sets, 4 conditions, one location

Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	poly(vinyl chloride-*co*-vinyl acetate)

Site-to-Site Reproducibility

To test site reliability, a round-robin study was undertaken in which the same polydisperse poly(vinyl chloride-*co*-vinyl acetate) sample was run on EcoSEC GPC Systems located at four different sites. The results are displayed in Table 1.

Reproducibility from system-to-system and location-to-location is exceptional with the EcoSEC GPC System. Coefficients of variations for all molar mass averages were all well below 1%. Because of the high instrument-to-instrument reproducibility of the EcoSEC GPC System, methods developed at one location, *e.g.*, an R&D laboratory, can be reliably transferred to a second site, e.g., a QC lab at a manufacturing site, and so on.

Table 1: Site-to-site reproducibility

	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)
Site A	1.30×10^4	2.98×10^4	5.37×10^4
Site B	1.37×10^4	2.99×10^4	5.43×10^4
Site C	1.36×10^4	2.98×10^4	5.32×10^4
Site D	1.37×10^4	3.02×10^4	5.41×10^4
Average	1.37×10^4	2.99×10^4	5.38×10^4
Deviation	70	160	420
%CV	0.52	0.55	0.78

Four EcoSEC GPC Systems, 4 operators,
4 column sets, 4 conditions, 4 locations

Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	poly(vinyl chloride-*co*-vinyl acetate)

Average of values measured with each instrument (n = 10).

Column Switching Valve

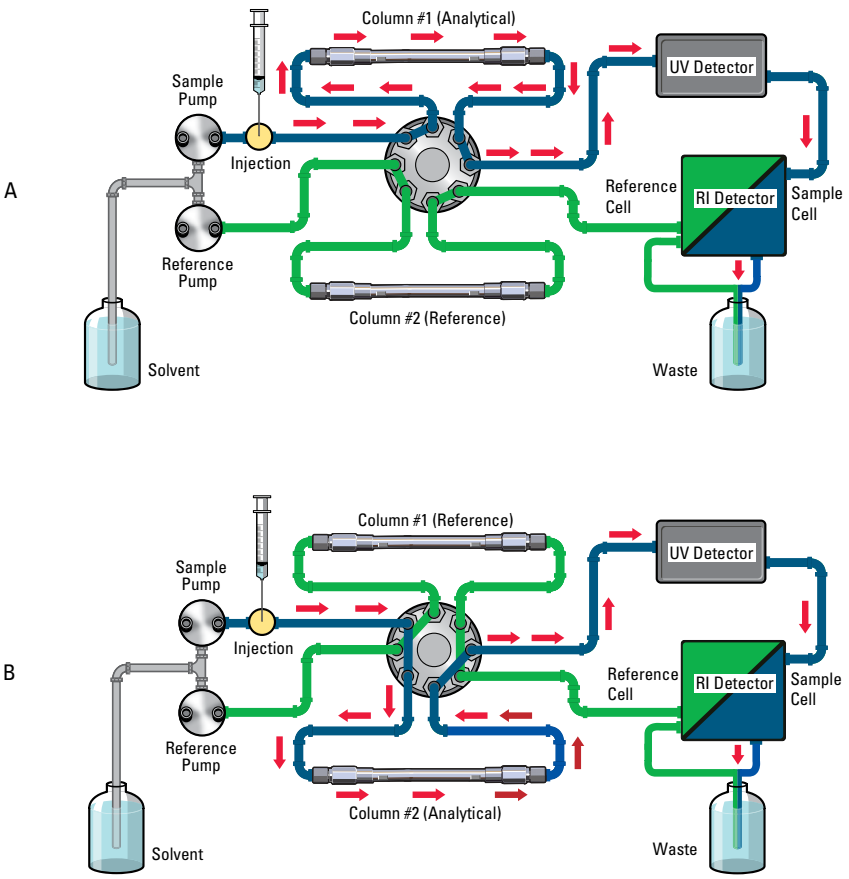
- Reduce column switching time
- Easily switch between low MM and high MM range columns
- Eliminate temperature related baseline drift following column change

Rapid Column Switching

The EcoSEC GPC System contains two pumps: a sample pump to deliver sample and solvent through the analytical column and the sample side of the RI detector flow cell and a reference pump to flow solvent (via a reference column) to the reference side of the RI detector flow cell. By installing an optional column switching valve and replacing the reference column with another analytical column, an analysis can be performed on column 1 while equilibrating column 2. After switching the valve, column 2 becomes the analytical column while column 1 will be in the flow path to the reference side of the RI detector flow cell (Figure 12).

Since the column switching valve changes column sets while the oven door remains closed and switches to an already equilibrated column set, a stable baseline is rapidly established.

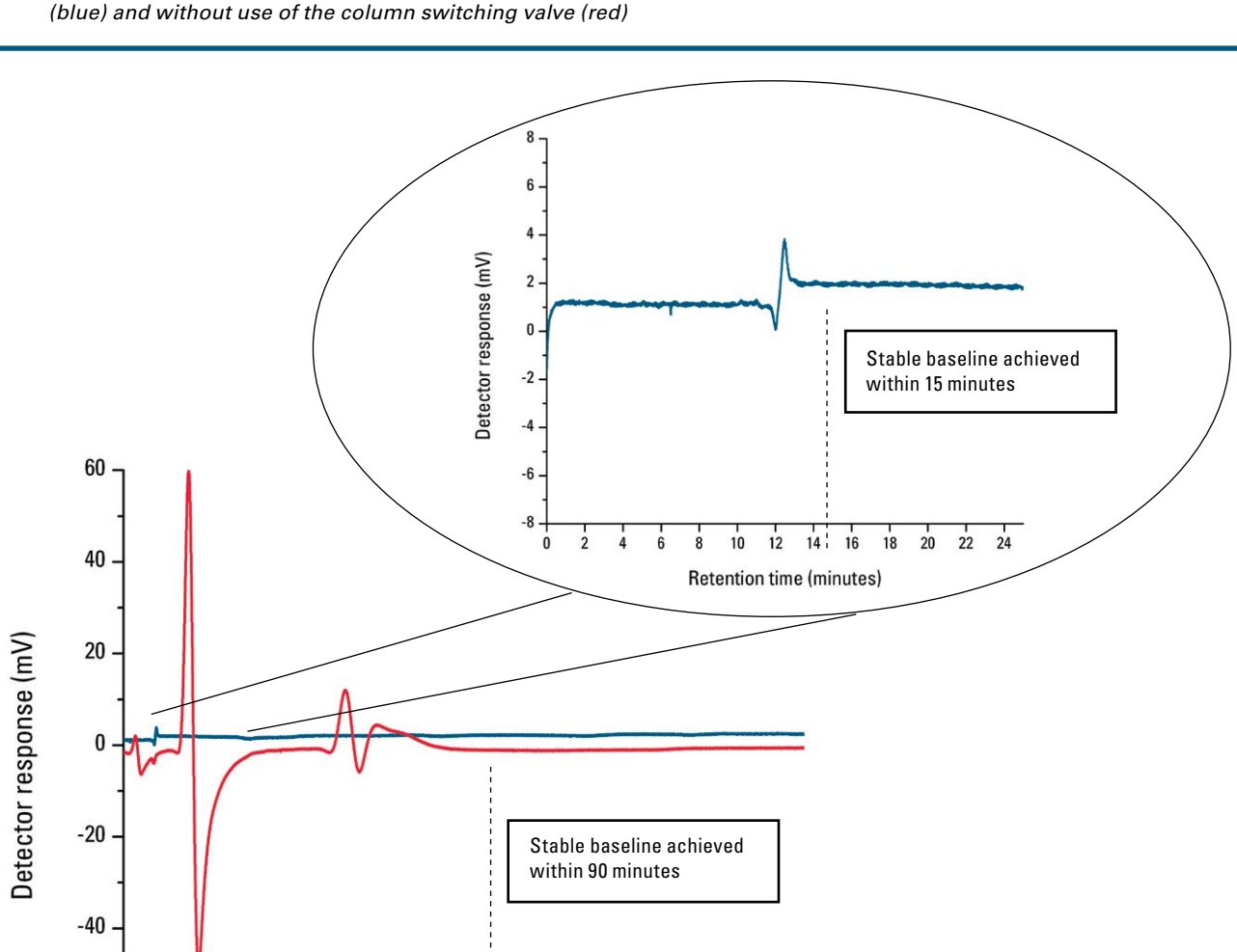
Figure 12: A: Flow path with column 1 as the analytical column B: Flow path with column 2 as the analytical column



Comparison of Time to Baseline Stability with and without the Column Switching Valve

On the EcoSEC GPC System the RI baseline is considered stabilized when the drift in signal is $1x10^{-7}$ RIU/h or less (based on THF at a flow rate of 1.0 mL/min). When a new set of columns is manually placed on the EcoSEC GPC System and the flow rate is started, the RI baseline stabilizes within 80 - 90 minutes. When a new column set is brought online using the column switching valve, the baseline stabilizes within 15 minutes. (Experimental conditions: THF, 35 °C, 0.35 mL/min, 20 min warm-up at 50% flow rate). Figure 13 clearly demonstrates the 65 - 75 minute savings in time required to reach a stable baseline when the columns are switched using the column switching valve compared to manually changing columns.

Figure 13: Overlay of refractive index detector signals during equilibration following a column change using the column switching valve (blue) and without use of the column switching valve (red)

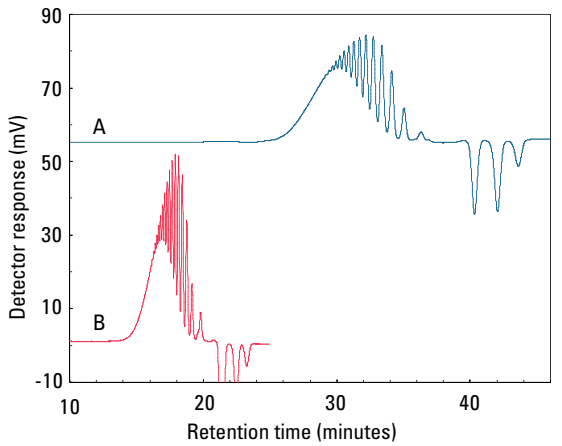


Increased Throughput and Lower Solvent Costs

Minimal extra-column band broadening is required to take full advantage of the highest efficiency GPC columns. The EcoSEC GPC System is engineered to minimize system dead volume. The semi-micro design allows the use of GPC columns with smaller ID (4.6 mm) and shorter lengths (15 cm) such as the TSKgel SuperMultiporeHZ columns. Together with a small stroke volume pump and a 2.5 µL RI flow cell, the EcoSEC GPC System allows accurate and precise molar mass measurements, particularly when benefiting from state-of-the-art column technology.

As shown in Figure 14, when run on the EcoSEC GPC System, the TSKgel SuperMultiporeHZ-N (4.6 mm ID × 15 cm) column achieves separation efficiency equivalent to that of a conventional high speed column (7.8 mm ID × 30 cm), but analysis time is reduced to half that of a conventional column and one-sixth the amount of solvent is consumed.

Figure 14: Comparing semi-micro and conventional GPC columns



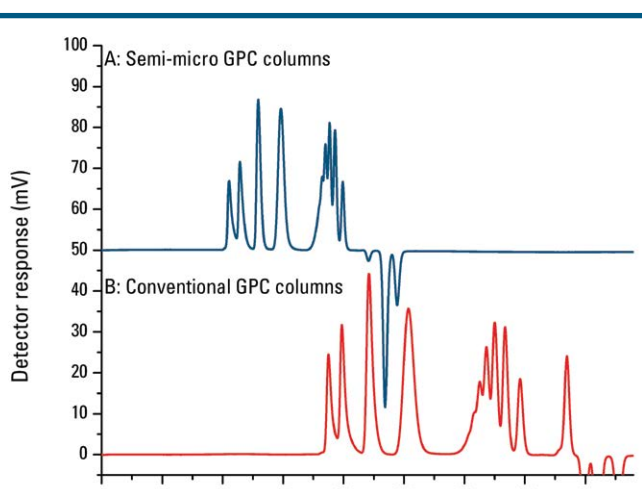
Columns:	A. Conventional columns, 7.8 mm ID × 30 cm × 4 **B. TSKgel SuperMultiporeHZ-N, 3 µm, 4.6 mm ID × 15 cm × 4**
Mobile phase:	THF
Flow rate:	A. 1.0 mL/min B. 0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	A. 50 µL B. 10 µL
Sample:	poly(teramethylene ether glycol)(PTMEG 650), 10 g/L

A comparison of chromatograms obtained from conventional and semi-micro TSKgel HXL and SuperHZ series columns are shown in Figures 15 and 16. TSKgel HXL and SuperHZ series columns have similar separation performance, solvent compatibility, stationary phase composition, and column efficiency. The differences between the two column series are particle size and column length.

A direct comparison between chromatograms obtained, under optimal operating conditions for each column length, for a mixture of polystyrene standards ranging in molar mass from 530 to 2.9 × 10^6 g/mol are shown in Figure 15. The resolution obtained via both column sets is virtually identical, the monomer, dimer, trimer, and tetramer of the lowest molar mass standard, 530 g/mol, can all be identified on both column lengths. Separation of the polystyrene standards using semi-micro GPC columns, Figure 15A, occurs in less than thirty minutes, approximately half the time required to obtain an identical separation using conventional GPC columns, Figure 15B.

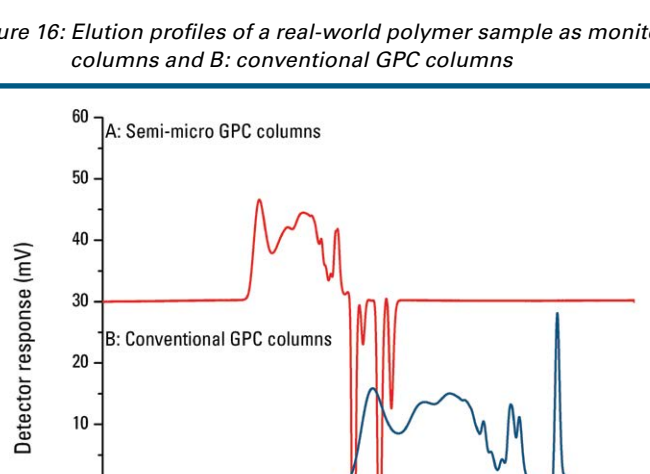
The GPC chromatogram of a real world polymer sample composed primarily of propylene glycol monomethyl ether acetate as obtained using the EcoSEC GPC System with semi-micro and conventional GPC columns was also compared. As can be seen in Figures 16A and 16B, a slight increase in resolution is observed towards the low molar mass, longer retention time region of the GPC chromatogram obtained using conventional GPC columns compared to semi-micro GPC columns. The combination of the low dead volume of the EcoSEC GPC System and the semi-micro GPC columns allowed for complete analysis in approximately 25 minutes, whereas analysis using conventional columns and the EcoSEC GPC System required an analysis times close to 45 minutes.

Figure 15: Elution profiles of polystyrene standards as monitored by RI on the EcoSEC GPC System with A: semi-micro GPC columns and B: conventional GPC columns



A Columns:	**TSKgel SuperHZ1000, SuperHZ2000, SuperHZ3000, SuperHZ4000, 3 µm, 4.6 mm ID × 15 cm**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	UV @ 248 nm, RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	30 µL
Samples:	polystyrene standards, PStQuick MP-M series
B Columns:	**TSKgel G1000HXL, G2000HXL, G3000HXL, G4000HXL, 5 µm, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 248 nm, RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	150 µL
Samples:	polystyrene standards, PStQuick MP-M series

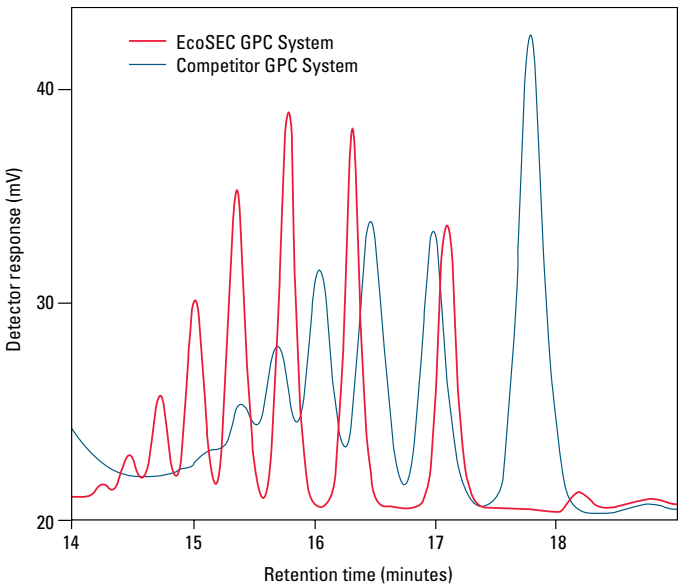
Figure 16: Elution profiles of a real-world polymer sample as monitored by RI on the EcoSEC GPC System with A: semi-micro GPC columns and B: conventional GPC columns



A Columns:	**TSKgel SuperHZ1000, SuperHZ2000, SuperHZ3000, SuperHZ4000, 3 µm, 4.6 mm ID × 15 cm**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	UV @ 248 nm, RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	30 µL
Sample:	real world polymer sample
B Columns:	**TSKgel G1000HXL, G2000HXL, G3000HXL, G4000HXL, 5 µm, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 248 nm, RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	150 µL
Sample:	real world polymer sample

Figure 17 shows an example of an oligomer (A-500) separation using four TSKgel SuperHZ2000 GPC columns in tandem on an EcoSEC GPC System and a conventional GPC system. A faster analysis and improved resolution is achieved with the EcoSEC GPC System as a result of the advanced engineering design of the system.

Figure 17: Comparison of resolution of a semi-micro column run on an EcoSEC GPC System and a conventional GPC system



Column:	**TSKgel SuperHZ2000, 3 µm, 4.6 mm ID × 15 cm × 4**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	styrene oligomer (A-500), 0.2 g/L

The combination of the EcoSEC GPC System and semi-micro columns provides significant solvent related cost savings while doubling sample throughput without compromising resolution. As shown in Table 2, the solvent related cost savings are extraordinary for samples requiring expensive solvents such as hexafluoroisopropanol.

Table 2: Annual solvent cost saving with semi-micro columns and the EcoSEC GPC System

Solvent	Competitive GPC System	EcoSEC GPC System	Savings
Chloroform ($17/L)	$1,830	$295	$1,535
DMF* ($25/L)	$2,600	$416	$2,184
NMP* ($30/L)	$3,082	$493	$2,589
THF* ($40/L)	$4,160	$666	$3,494
HFIP* ($1,000/L)	$96,493	$15,439	$81,054

* DMF: dimethylformamide; NMP: N-methylpyrrolidone; THF: tetrahyrofuran; HFIP: hexafluoroisopropanol

EcoSEC GPC System Specifications

Pump	Specification
Flow rate	0.010 to 2.000 mL/min in 0.001 mL/min steps
Accuracy	± 2%
Precision	± 0.2%
Maximum pressure	25 MPa or 3,500 psi
Safety features	Liquid supply stops if pressure rises above the upper limit or drops below the lower limit, Plunger drive count monitoring, Pan for liquid leakage
Stroke volume	7.51 µL
Auto-injector	
Injection volume	1 to 1,500 µL in 1 µL increments
Number of samples	100, 2 mL injection vials
Column Oven	
Temperature range	Ambient plus 10 °C to 60 °C
Capacity	7.8 mm ID × 30 cm × 8 columns
Accuracy	± 0.5 °C
Precision	± 0.2 °C
RI Detector	
Type	Bryce (dual flow type), Tungsten light source (1.00-1.80 RI range)
Optics	Deflection
Cell volume	2.5 µL
Cell pressure limit	0.5 MPa
Noise	2×10^{-9} RIU
Drift	1×10^{-7} RIU/h (THF, 1.0 mL/min)
Dynamic range	$\pm 2.5 \times 10^{-4}$ RIU
Temperature control	Off, 35 °C, 40 °C, 45 °C
Analog out	For connection to third party light scattering and viscometry detectors
Safety features	Leak sensor and thermal fuse for circuit block
Instrument	
Dimensions	680 (W) × 500 (D) × 550 (H) mm = 2.2′ x 1.6′ x 1.8′
Weight	95 kg = 210 lbs
Dead volume	<20 µL


EcoSEC

Unparalleled Versatility

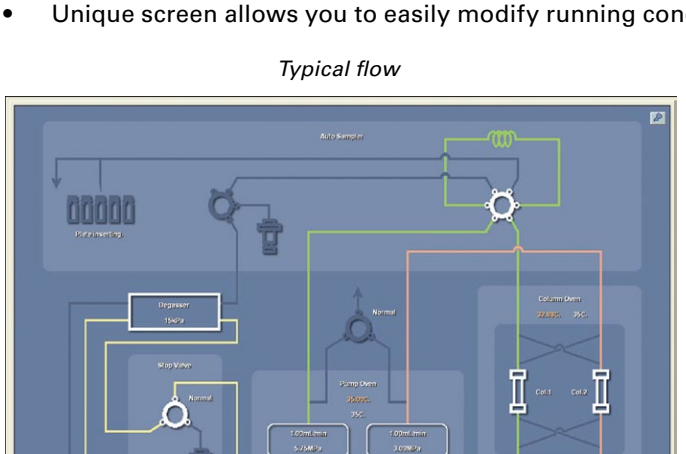
- GPC-specific EcoSEC GPC System software to simplify system control and data handling
- Controls up to 2 EcoSEC GPC Systems
- Excellent data handling and report generation
- Fully featured data handling system; analyze data from two detectors
- Start and stop system automatically
- One license for multiple locations

Features include:

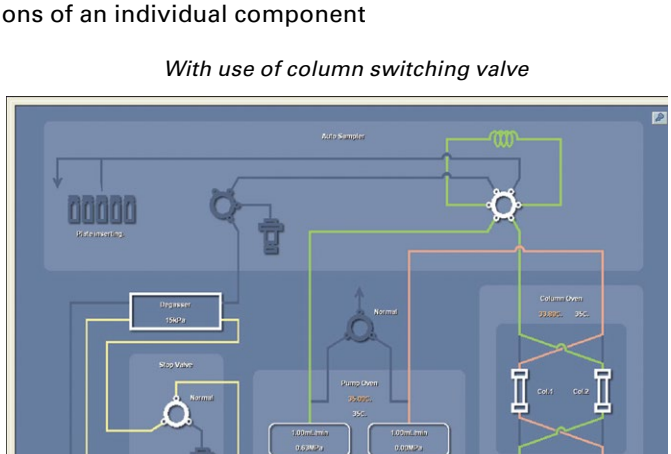
Flow Diagram

- Unique screen allows you to easily modify running conditions of an individual component

Typical flow

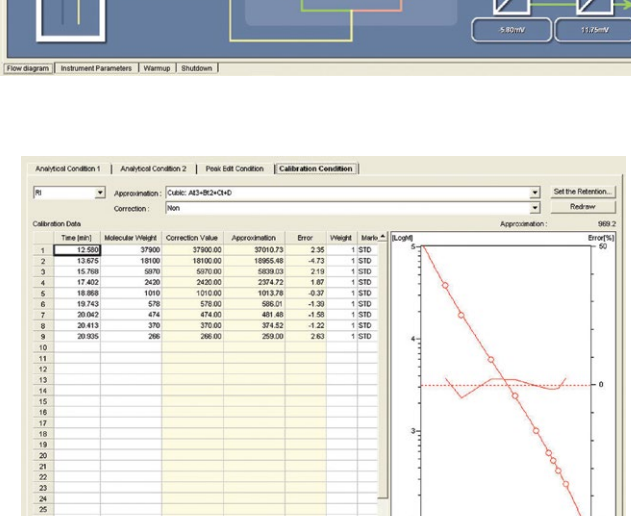


With use of column switching valve



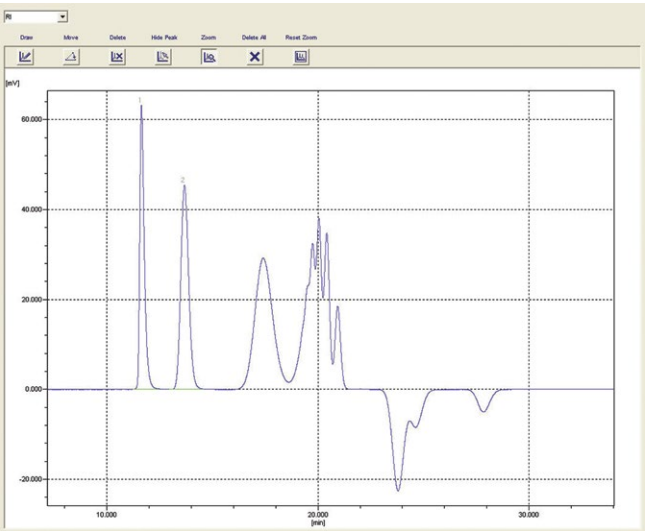
Method

- All parameters for data acquisition and peak integration, including baseline operations, are saved in the template method
- One click switching between calibration curves



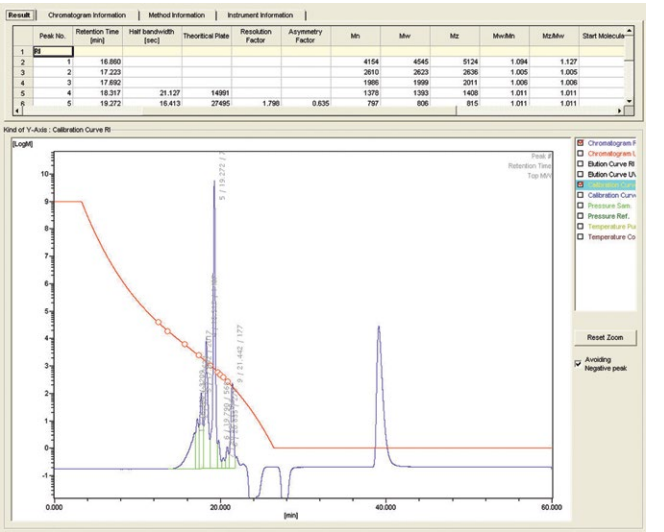
Peak Editing

- Full editing functionality including baseline setting and peak splitting using the mouse
- Automated peak editing



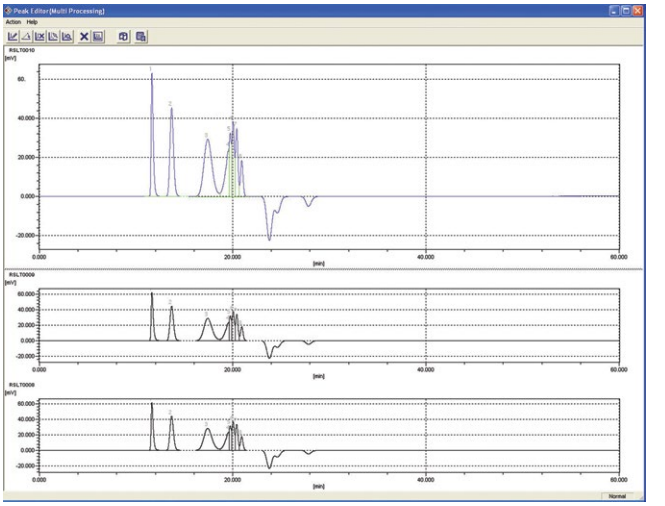
Data Management

- Allows viewing of chromatograms, elution curve, flow rate, pressure, and temperature



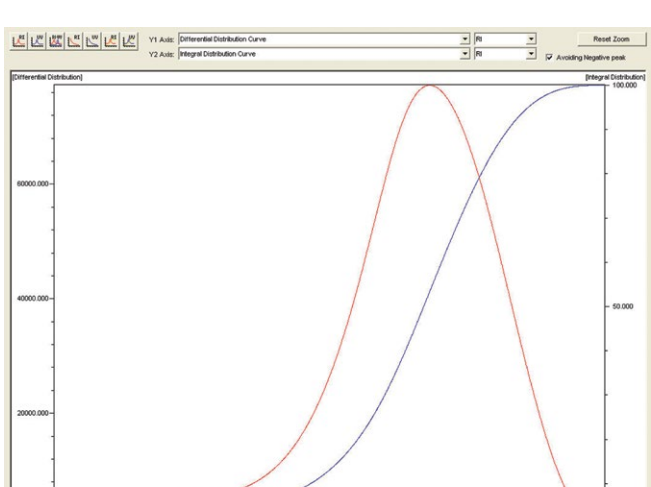
Multiprocessing Function

- Automatically applies exact set of peak detection and integration parameters to all chromatograms in a list
- Similar chromatograms are processed identically for enhanced reproducibility



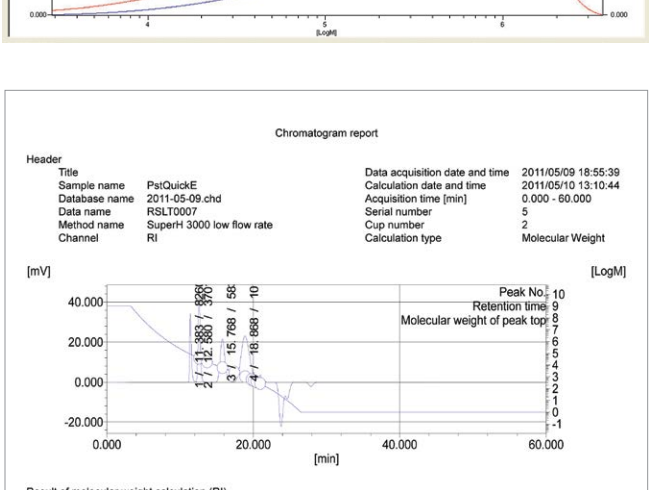
GPC Specific Quantitative Calculations

- M_n, M_w, and M_z molar mass averages
- Cumulative and differential molar mass plotting



Report Generation

- Large number of built in reports
- Customizable reports
- Easily export data into text or pdf files



Chromatogram report

Header

Title			
Sample name	PstQuickE	Data acquisition date and time	2011/05/09 18:55:39
Database name	2011-05-09.chd	Calculation date and time	2011/05/10 13:10:44
Data name	RSLT0007	Acquisition time [min]	0.000 - 60.000
Method name	SuperH 3000 low flow rate	Serial number	5
Channel	RI	Cup number	2
		Calculation type	Molecular Weight

[mV] [LogM]

40.000
20.000
0.000
-20.000
0.000
20.000
40.000
60.000
[min]
Peak No.
Retention time
Molecular weight of peak top
10
9
8
7
6
5
4
3
2
1
0
-1

Result of molecular weight calculation (RI)

Peak 1 Base Peak

	[min]	[mV]	[mol]		
Peak start	10.902	-0.101	117,191	Mn	78,936
Peak top	11.383	34.209	82,600	Mw	79,604
Peak end	12.147	0.040	49,010	Mz	80,212
				Mz+1	80,770
				Mv	79,604
Height [mV]			34.307	Mp	82,600
Area [mV*sec]			530.161	Mz/Mw	1.008
Area% [%]			12.045	Mw/Mn	1.008
[eta]			79603.68576	Mz+1/Mw	1.015

Software Specifications

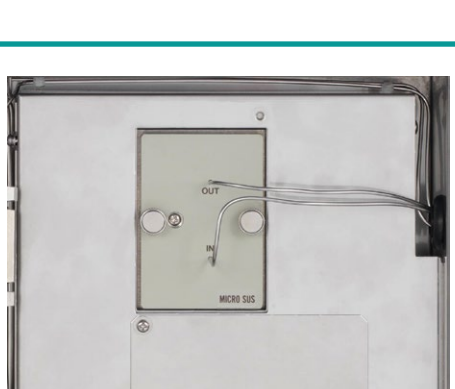
Feature	Description
Software	Provided on CD-ROM
Data acquisition	2-channel (RI,UV)/1-system USB connection
Acquisition time	0.0 to 999.9 minutes
Acquisition interval	50 ms or more (10 ms steps) Upper limit: 1000 ms
Acquisition rate	1 Hz to 20 Hz
Calibration curve approximation	• First-degree expression • 3rd-degree expression • 3rd-degree expression + hyperbola • 5th-degree expression • 7th-degree expression • 7th-degree expression (odd power) • 7th-degree expression (odd power) + hyperbola
Calibration curve correction	• Mark-Houwink • Q factor • Polymerization degree • USP
Quantitative calculation specific to GPC	• Molar mass averages (M_n, M_w, and M_z) • Polydispersity Index (*PDI*) • Cumulative/differential molar mass distributions • Concentration ratio
Special calculation function	• Internal standard correction function • Copolymer analysis • Molar mass fraction specific calculation • Calculation range specification • Lag time correction
Column test	• Theoretical plate number • Resolution • Symmetry factor • Half bandwidth
Calculation standard	• ASTM® • DIN® • USP • JIS • JP • ISO 16014 • Tosoh Standard
FDA 21 CFR Part 11	Software validation, authentication by user ID and password, log out, and audit trail
Warm up and shut down timers	• Daily • Weekly
RI and UV detector auto balance	Optional prior to injection

The standard EcoSEC GPC System consists of the following:

- EcoSEC GPC System instrument
- EcoSEC GPC Workstation Software
- Dual flow RI detector
- Optional 2-way column switching valve (see page 12)
- Optional UV detector

UV Detector

- Variable UV; 195 – 350 nm
- Semi-micro flow cell (2 µL)
- Factory installed option



The optional UV detector is variable from 195 to 350 nm and the detector flow path and electronics are optimized for the use of semi-micro columns. The volume of the flow cell is reduced to 2 µL and the shortest time constant is 0.5 seconds.

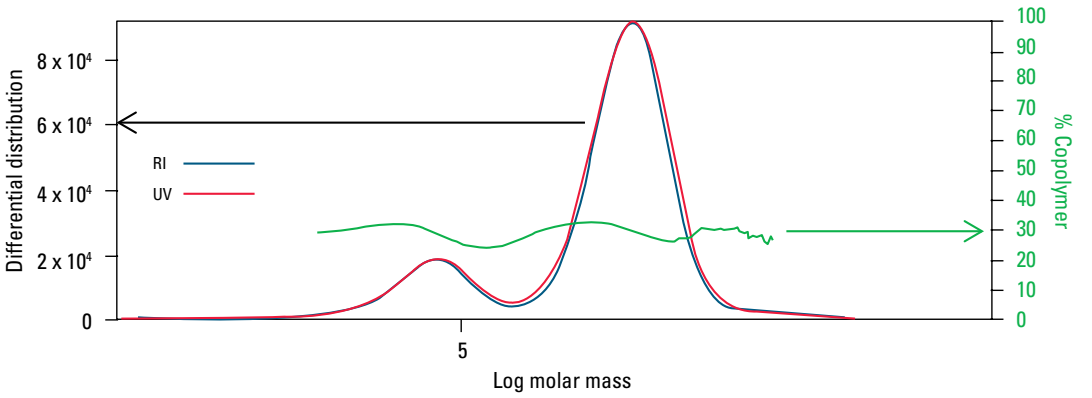
UV Detector Specifications

UV Detector	Specification
System	Dual beam, single flow cell
Light source	Deuterium lamp
Wavelength range	195 to 350 nm
Wavelength accuracy	± 2 nm
Bandwidth	8 nm
Range (FS)	0.5, 1, 2, 4 AU/1 V
Response	0.5, 1.0, 3.0 seconds
Drift	3 x 10^{-4} AU/h (254 nm, air in cell, response: 1.0 s)
Noise	2.5 x 10^{-5} AU (254 nm, air in cell, response: 1.0 s)
Flow cell volume	2 µL
Safety mechanism	Liquid leakage sensor; lighting time monitoring

Copolymer Analysis

The EcoSEC GPC System equipped with both RI and UV detectors can be used to determine the structural composition of an unknown copolymer, in which the copolymer contains one UV visible and one non-UV visible component. At least one copolymer of known composition must be available to create a copolymer calibration curve. The final result is a plot of the structural composition at each molar mass. This composition curve overlaid on the chromatogram, as seen in Figure 1, can be generated using the EcoSEC GPC Workstation Software. The software allows for the creation and use of separate UV and RI specific calibration curves while correcting for the inter detector delay volume.

Figure 1: Copolymer analysis of polystyrene-b-polybutene



Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI, UV @ 254 nm (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	10 µL
Samples:	PS-*b*-PB, 0.2 wt%

Enhanced EcoSEC GPC System Analysis

The addition of multiple detection methods to the EcoSEC GPC System allows for the characterization of a variety of polymer properties. A multi-detector GPC set up can be used to determine:

- Polystyrene relative molar mass averages based on RI or UV detection
- Copolymer compositional drift with RI and UV detection
- Universal calibration, intrinsic viscosity and viscometric radius with viscometry detection
- Absolute molar mass averages and radius of gyration with multi-angle light scattering (MALS) detection
- Hydrodynamic radius determination with quasi-elastic light scattering (QELS) detection

Summary of Detector Capabilities

Detector	Molar Mass Determination	Detects Most Polymers	Required For Copolymer Composition Analysis
RI	Relative	Yes	Yes
UV	Relative	No	Yes

Static Light Scattering Detectors

Detector	Measuring angle(s) (deg)	Molar Mass Range (g/mol)*	Radius of Gyration (R_G) range (nm) *
Low Angle Light Scattering (LALS)	7	$<10^3$ to $>10^7$	10 to 50 (only if combined with RALS)
Right Angle Light Scattering (RALS)	90	$<10^3$ to 10^5 (up to 10^6 if combined with viscometer)	N/A if used alone (calculated from Flory-Fox equation if combined with viscometer)
Multi Angle Light Scattering - 2 Angle	15, 90	$<10^3$ to 10^6	10 to 50
Multi Angle Light Scattering - 3 Angle	45, 90, 135	$<10^3$ to $>10^6$	10 to 50
Multi Angle Light Scattering - 7 Angle	35 to 145	$<10^3$ to $>10^6$	10 to 200
Multi Angle Light Scattering - 8 Angle	23 to 155 (solvent dependent)	$< 10^3$ to 10^7	10 to 200
Multi Angle Light Scattering - 9 Angle	28 to 156	$<10^3$ to 10^7	10 to 200
Multi Angle Light Scattering - 18 Angle	15 to 160 (solvent dependent)	$<10^3$ to $>10^7$	10 to 500
Multi Angle Light Scattering - 20 Angle	12 to 164	$<10^3$ to $>10^7$	10 to 500

**Sample dependent*

Viscometry Detectors

Detector	Split Ratio	Applicable to GPC/SEC	Obtainable Measurements
Single Capillary Viscometer	N/A	No	Relative viscosity
4-Capillary Differential Viscometer	50/50	Yes	• Intrinsic viscosity distribution • Molar mass distribution via universal calibration • Hydrodynamic radius • Mark-Houwink plots • Branching information • Conformation
4-Capillary Differential Viscometer	80/20	Yes	

Tosoh Bioscience can tailor a system to meet your application needs.

Does your analysis require additional detectors beyond RI and UV?
The EcoSEC GPC System provides easy and effortless connectivity when using multi-detector configurations. We offer external light scattering and viscometry detectors.

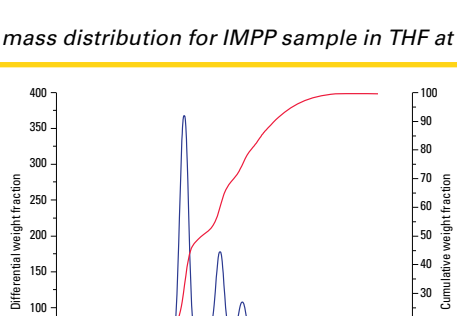
Contact us for a quote!

Molar Mass Measurements of an Isocyanate Modified Polyurethane Prepolymer in Less than 1 Hour with the EcoSEC GPC System

Isocyanates are both highly reactive and highly toxic low molar mass chemicals. One common technique used to take advantage of isocyanate reactivity while eliminating safety concerns is to synthesize polyurethane prepolymers for use in subsequent polymerizations. An EcoSEC GPC System encompassing a refractive index detector was used to perform size exclusion chromatography analysis on a isocyanate modified polyurethane prepolymer (IMPP) sample composed of 54% urethane prepolymer, 11.5% dimethyl sulfoxide (DMSO), and 34.5% 1,1,1,3,3 pentafluoropropane. The low dead volume of the EcoSEC GPC System combined with the use of semi-micro TSKgel GPC columns allowed for the successful determination in less than 30 minutes of the molar mass averages and polydispersity of the IMPP sample.

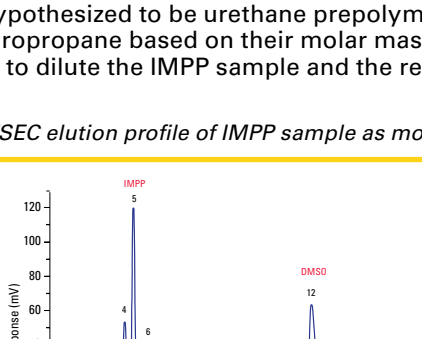
The polydispersity index, PDI = M_w/M_n, for the entire urethane prepolymer sample including 1,1,1,3,3 pentafluoropropane (peaks 1 through 9) was 2.26, while the nine individual components had PDI values ranging from 1.01 to 1.09. From the PDI values it can be concluded that collectively the sample is polydisperse with respect to molar mass but the nine visible components within the IMPP sample are virtually monodisperse with respect to molar mass. The molar mass distribution for the IMPP sample, as obtained at 0.3 mL/min, is shown in Figure 1.

Figure 1: Cumulative and differential molar mass distribution for IMPP sample in THF at 0.3 mL/min



The molar mass averages and polydispersity index of the IMPP sample was determined using a polystyrene relative calibration curve. Analysis of the IMPP was initially performed at a flow rate of 0.3 mL/min (the lowest recommended flow rate for the TSKgel SuperH3000 columns) and total analysis was achieved in 45 minutes. In order to increase the throughput of the EcoSEC GPC System the flow rate was increased to 0.6 mL/min (the highest recommended flow rate for the TSKgel SuperH3000 columns). The chromatogram of the IMPP displayed twelve distinctive peaks, as shown in Figure 2. Peaks 1 through 5 were determined to be the urethane prepolymer component of the IMPP and found to have a weight average molar mass ranging from 4,199 to 798 g/mol. The identity of peaks 6 through 9 were not confirmed but are hypothesized to be urethane prepolymer, unreactive species from the synthesis of the sample or 1,1,3,3 pentafluoropropane based on their molar mass range, M_w = 551-178 g/mol. Peaks 10 and 11 and peak 12 are due to the THF used to dilute the IMPP sample and the residual DMSO in the IMPP sample, respectively.

Figure 2. FSEC elution profile of IMPP sample as monitored by RI (blue) at 0.6 mL/min in THF at 35 °C



Column:	**TSKgel SuperH3000, 3 µm, 6.0 m ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.6 mL/min
Detection:	RI (EcoSEC GPC system)
Temperature:	35 °C
Injection vol.:	20 µL
Sample:	IMPP

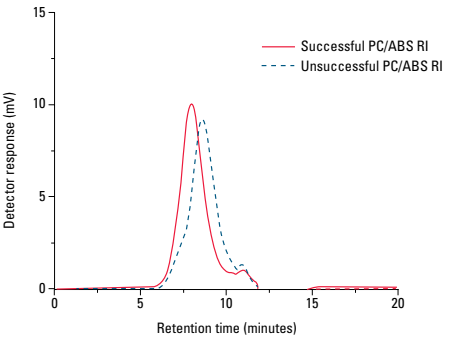
An Approach to Failure Analysis of PC/ABS Resins Used in Automobile Parts: Molar Mass Determination via Gel Permeation Chromatography

For polymeric materials the molar mass and molar mass distribution plays a vital role in the determination of mechanical, bulk, and solution properties. These properties govern polymer processing and the end-use performance of a given material[1,2]. The difference between a successful and unsuccessful polymer based material can be determined by observing the molar mass and molar mass distribution of the polymer(s) encompassing the end-use material. One polymeric material of particular interest to the automotive industry is an alloyed grade thermoplastic: polycarbonate acrylonitrile-butadiene-styrene (PC/ABS). An EcoSEC GPC System encompassing a dual flow refractive index (RI) detector was implemented to perform failure analysis on two PC/ABS automobile parts. The use of GPC for the failure analysis allowed for determination of the molar mass averages, molar mass distributions, and a comparison of successful and unsuccessful PC/ABS automobile parts.

The molar mass averages of two samples, successful and unsuccessful PC/ABS, were determined via GPC. The successful product was shown to perform up to standards while the unsuccessful product failed at some point during production or usage. The dual-detector GPC experiments provide two forms of comparison between the successful and unsuccessful PC/ABS automobile parts: GPC chromatograms and polystyrene relative molar mass averages and distributions.

The chromatograms of the successful and unsuccessful PC/ABS as monitored by the RI detector is shown in Figure 3. The successful PC/ABS sample elutes prior to the unsuccessful PC/ABS. The shorter retention time of the successful PC/ABS indicates that the successful PC/ABS sample is larger in polymeric size than the unsuccessful PC/ABS sample. Thus, the GPC chromatogram alone provides sufficient indication that the successful and unsuccessful PC/ABS samples are different from one another.

Figure 3. GPC elution profile of successful and unsuccessful PC/ABS automobile parts as monitored by RI



Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC system)
Temperature:	35 °C
Injection vol.:	10 µL
Sample:	PC/ABS at 1 g/L

The results of the experiments, in the form of polystyrene relative molar mass averages, are given in Table 1. The successful PC/ABS sample was determined to have a significantly higher number-, weight-, and z-average molar mass than the unsuccessful PC/ABS sample. The number-average molar mass, M_n, varies the greatest between the two samples, as M_n of the successful product is nearly twice that of the unsuccessful product. For PC/ABS, the molar mass averages directly influence the toughness and melt viscosity of the end-use material. Higher molar mass PC/ABS is tougher than their lower molar mass counterparts; thus, explaining one reason why the unsuccessful PC/ABS failed in the end-use material; the lower the molar mass, the weaker the end-use material.

Table 1. Molar mass averages and polydispersity index of successful and unsuccessful PC/ABS automobile parts

Sample (Detection Method)	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI_a
Successful PC/ABS (RI)	$1.100 \times 10^4 \pm 335^b$	$5.199 \times 10^4 \pm 752$	$1.339 \times 10^5 \pm 3{,}072$	4.73 ± 0.08
Unsuccessful (RI)	$6{,}064 \pm 35$	$3.036 \times 10^4 \pm 260$	$1.259 \times 10^5 \pm 1{,}465$	5.01 ± 0.02

[a] PDI = M_w/M_n ; [b] Standard deviations from six injections

The use of the EcoSEC GPC System for failure analysis of PC/ABS resins used in automobile parts allowed for immediate differentiation between the successful and unsuccessful PC/ABS samples based on the GPC elution profile. This differentiation was then confirmed through observed differences in the polystyrene relative molar mass averages of the successful and unsuccessful PC/ABS samples.

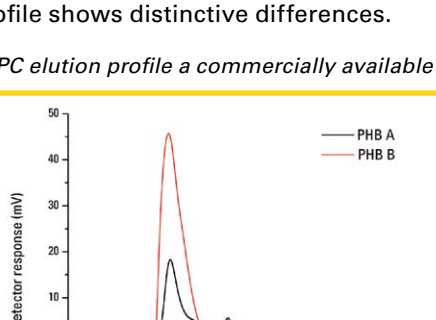
[1]Striegel, A.M.; Yau, W.W.; Kirkland, J.J.; Bly, D.D. Modern Size-Exclusion Liquid Chromatography 2nd ed; Wiley: New York, 2009.
[2]Mori, S.; Barth, H.G. Size Exclusion Chromatography; Springer: New York, 1999.

Characterization of a Plastic Alternative via Gel Permeation Chromatography: Polyhydroxybutyrate

During the past several decades there have been many promising developments of eco-friendly plastics. One promising biodegradable substitute for plastics that is not made from petroleum but from renewable resources is a biopolymer known as polyhydroxybutyrate or PHB. The use of PHB in commercial products is reliant on the development of low cost processes that produce biodegradable plastics with properties similar or superior to their petrochemical counterparts. Once a process for the production of PHB is developed, the physicochemical properties of the PHB must be characterized, as variations in properties such as the molar mass, will dictate how the biodegradable plastics performs compared to the petrochemical plastic. The chemical and thermal properties of PHB are typically analyzed using a collection of methods. The use of an EcoSEC GPC System encompassing a dual flow refractive index detector was implemented to determine the molar mass averages and molar mass distribution of two PHB polymers produced from different processes (commercially available and homemade).

The GPC chromatograms of the commercially available and the homemade PHB samples as monitored by the RI detector are shown in Figure 4. The commercially available PHB sample (PHB A) elutes prior to the homemade PHB sample (PHB B). The slightly shorter retention time of the PHB A sample indicates that the commercially available PHB is larger in polymeric size than the homemade PHB; as the elution order in GPC is that of an "inverse-sieving" technique, larger analytes sample a smaller pore volume than smaller analytes resulting in the larger analytes eluting from the column prior to the smaller analytes. In addition to variations in elution time amongst the two samples, the shape of the GPC elution profile shows distinctive differences.

Figure 4. GPC elution profile a commercially available PHB sample (PHB A) and a homemade PHB sample (PHB B) as monitored by RI



Column:	**TSKgel SuperHZM-M, 3 & 5 µm, 4.6 mm ID × 15 cm × 2**
Mobile phase:	Chloroform
Flow rate:	0.30 mL/min
Detection:	RI (EcoSEC GPC system)
Temperature:	35 °C
Injection vol.:	25 µL
Sample:	polyhydroxybutyrate (PHB) at 1g/L

The molar mass averages, M_n , M_w , and M_z, as determined via a polystyrene RI calibration curve are given in Table 2. The molar mass averages of the commercial available PHB (PHB A) and the homemade PHB (PHB B) are in agreement with the variations seen in the GPC elution profile, as the molar mass averages for PHB A are slightly less than those of PHB B. The polydispersity of the commercially available PHB, PHB A, is nearly double that of homemade PHB, PHB B, PDI=8.744 and PDI=4.863 for PHB A and PHB B, respectively (Table 2). The ability to determine variations in the molar mass averages and molar mass distributions of PHB is essential, as it can affect the thermoplasticity and biodegradability of the plastic.

Table 2. Molar mass averages and polydispersity index of a commercially available PHB sample (PHB A) and a homemade PHB sample (PHB B)

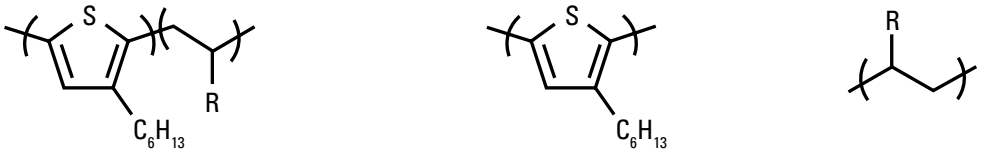
Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI_a
PHB A	$8.22 \times 10^4 \pm 0.49^b \times 10^4$	$7.17 \times 10^5 \pm 0.01 \times 10^5$	$1.44 \times 10^6 \pm 0.01 \times 10^6$	8.74 ± 0.38
PHB B	$2.15 \times 10^5 \pm 0.14 \times 10^5$	$1.04 \times 10^6 \pm 0.01 \times 10^6$	$2.00 \times 10^6 \pm 0.01 \times 10^6$	4.86 ± 0.30

[a] *PDI = M_w/M_n ;* [b] *Standard deviations from four injections*

Analysis of gradient copolymers using the EcoSEC GPC System

Gradient sequence copolymers are novel materials which have provoked interest due to their unique properties compared to their random, alternating and block equivalents. Unlike block copolymers which have an abrupt change in sequence, gradient sequence copolymers exhibit a gradual change in co-monomer composition from one type of monomer to another. An example of a gradient copolymer is poly(3-hexylthiophene-b-[1-hexane]), Figure 5, which is composed of poly(3-hexylthiophene) and poly(1-hexene).

Figure 5. Example of a gradient copolymer



poly(3-hexylthiophene-b-[1-hexene]) poly(3-hexylthiophene) poly(1-hexene)

The ability to characterize the molar mass averages and distributions of a π-conjugated gradient copolymer is critical for designing polymer blends as molar mass averages and distributions affect the phase separation of polymer blends. An EcoSEC GPC System housing a dual flow refractive index detector was used to perform gel permeation chromatography analysis on poly(3- hexylthiophene-b-[1-hexane]), poly(3-hexylthiophene) and poly(1-hexene). The GPC elution profiles and molar mass averages of the copolymer and homopolymer were obtained in less than fifteen minutes with the use of the EcoSEC GPC System and TSKgel semi-micro GPC columns, thus providing a fast and reliable method for the analysis of copolymers.

The GPC chromatograms of the copolymer, poly(3-hexylthiophene-b-[1-hexane]), and the two homopolymers, poly(3-hexylthiophene) and poly(1-hexene) are shown in Figures 6-8, respectively The copolymer, poly(3-hexylthiophene-b-[1-hexane]), displays a distinctive bimodal distribution while the two homopolymers have a mono-modal distribution. By comparing the retention times of the RI detector response for the three samples the later eluting species seen in Figure 6 has the same retention time as the homopolymer, poly(3-hexylthiophene), in Figure 7. The early eluting species seen in Figure 6 elutes later than that of the other homopolymer, poly(1-hexene) (Figure 8), an indication that the later elution species in Figure 6 is that of the copolymer. The copolymer elutes prior to the homopolymers is an indication that the copolymer is larger in polymeric size than the homopolymers.

Through the comparison of the GPC elution profiles and the molar mass averages of the copolymer, poly(3-hexylthiophene-b-[1-hexane]), and the two homopolymers, poly(3-hexylthiophene) and poly(1-hexene) it can be concluded that the copolymer sample, poly(3-hexylthiophene-b-[1-hexane]), contains copolymer and excess amounts of one of the homopolymers, poly(3-hexylthiophene).

Figure 6. GPC elution profile of the copolymer, poly(3-hexylthiophene-b-[1-hexane], as monitored by the RI (blue) and UV (red)

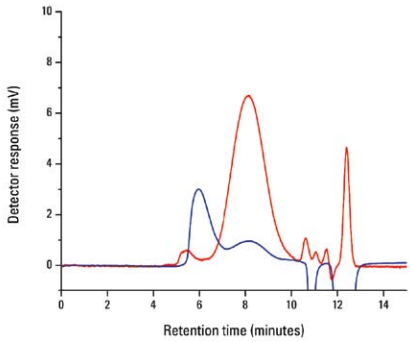


Figure 7. GPC elution profile of homopolymer, poly(3-hexylthiophene), as monitored by the RI (blue) and UV (red)

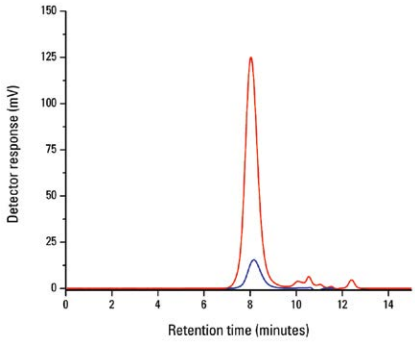
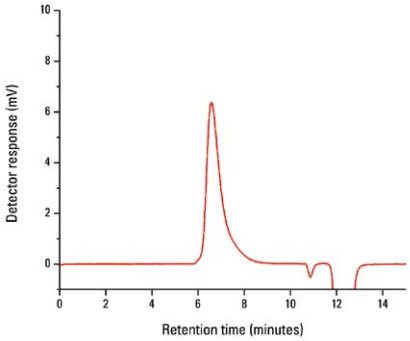


Figure 8. GPC elution profile of homopolymer, poly(1-hexene), as monitored by the RI



Columns:	**TSKgel SuperMultipore × 2 + TSKgel mixed bed x2**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC system)
	UV (EcoSEC GPC system @ 254 and 350 nm)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	poly(3-hexylthiophene-b-[1-hexane])

Renewable-Based Thermoplastic Polyurethanes

The demand for renewable or bio-based polymers continues to rise exponentially as manufacturers within the automotive, footwear, carpet, and furniture sectors seek to sell more sustainable products. One group of polymers gaining a great deal of interest is thermoplastic polyurethanes or TPUs. A TPU is an elastomer that resembles rubber in consistency and feel but by nature has outstanding abrasion resistance, great low temperature flexibility, resistance to oil, and a high threshold for support weight, in addition to being very bondable, durable, paintable, and impact resistant. The specific end-use properties, such as tensile strength, elongation, conductivity, chemical resistance, and toughness, depends on macromolecular properties such as molar mass, branching, degree of crosslinking, and polymeric size.

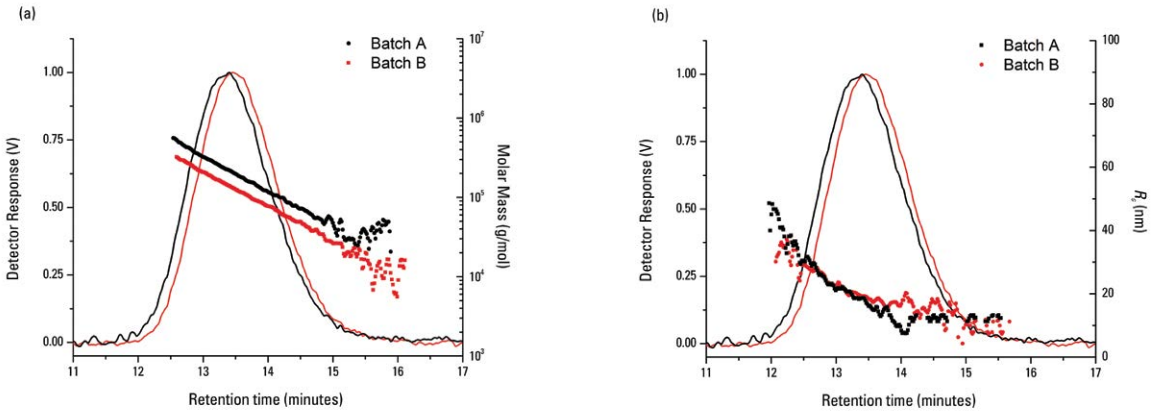
Two different batches of TPUs were characterized based on molar mass and polymeric size using the EcoSEC GPC System coupled to a multi-angle light scattering detector (MALS). The GPC elution profiles of the two samples are shown in Figures 9A and 9B. TPU Batch B elutes after TPU Batch A, indicating that TPU Batch B is slightly smaller in size compared to TPU Batch A.

The size comparison can be done quantitatively as the addition of a MALS detector to the EcoSEC GPC System permits for the determination of a polymeric sizing parameter, the root-mean-square radius or radius of gyration, R_G. Figure 9B shows the R_G distributions as plotted across the GPC elution profile: both curves overlay and the size of the TPUs decreases as a function of increasing retention time, as expected in a size exclusion mechanism. Although the average radius of gyration for both TPUs, A and B, were identical, R_G = 20 nm, the left end of the curves in Figure 9B shows that TPU Batch A does contain slightly more of large polymer species than TPU Batch B.

The molar mass distributions of the two different batches of TPUs were also plotted across the GPC elution profile, Figure 9A. The absolute weight average molar mass, M_w, is slightly higher for A than B, 1.64 × 10^5 and 1.42 × 10^5 g/mol, respectively. From Figure 9A it can be noticed that for any given retention time – and thus polymer size – TPU Batch A has a higher molar mass than TPU Batch B. This shows that the two TPUs have a different structure or conformation in the solvent.

In conclusion, the higher molar mass average of TPU Batch A is not only due to the presence of a small amount of larger species in the distribution, but also to a denser structure or conformation in solution, as compared to TPU Batch B.

Figure 9: Thermoplastic polyurethanes



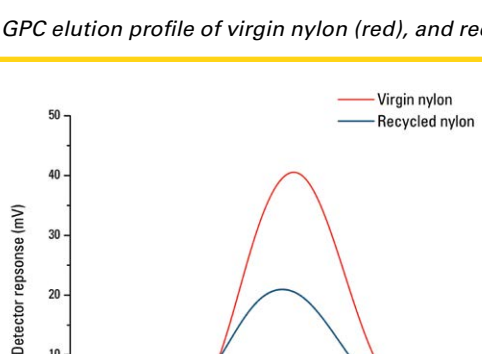
Column:	**TSKgel GMH$_{HR}$-H, mixed bed, 7.8 mm ID × 30 cm × 2**
Mobile phase:	DMF with 0.01% LiBr
Flow rate:	1.0 mL/min
Detectors:	RI (EcoSEC GPC System), MALS (Wyatt DAWN® 8+)
Temperature:	50 °C
Injection vol.:	100 µL
Sample:	thermoplastic polyurethanes

Environmentally Friendly Analysis of Nylon

Green initiatives are continuously approaching the polymer science discipline from all sides as companies are not only interested in greener products and additives but greener and more cost effective synthesis and characterization methods. One class of polymers that is of high interest is polyamides, more specifically nylons, as these plastics are common materials in everyday life which produce large quantities of environmental contaminates.[3] It is critical to be able to characterize virgin and recycled nylon as the recycling process of nylon can result in the reduction of physical-mechanical properties as well as changes in morphology resulting in different end-use properties. A greener and more cost effective method for the characterization of the molar mass averages and distributions of nylon in hexafluoroisopropanol (HFIP) was employed by using an EcoSEC GPC System and semi-micro GPC columns. The combination of the low dead volume of the EcoSEC GPC System and semi-micro GPC columns provides significant solvent related costs while doubling sample throughput without compromising resolution.

The GPC experiments provide two forms of comparison between the virgin and recycled nylon samples: GPC chromatograms and poly(methyl methacrylate) (PMMA) relative molar mass averages and molar mass distributions. The GPC elution profiles of the virgin and recycled nylon as monitored by the RI detector are shown in Figure 10. The virgin nylon elutes after the recycled nylon. The longer retention time of the virgin nylon indicates that the virgin material is slightly smaller in polymeric size compared to the recycled material: as elution order in GPC is that of an "inverse-sieving" technique, smaller analytes elute after the larger analytes.

Figure 10: GPC elution profile of virgin nylon (red), and recycled nylon (blue) as monitored by RI



Column:	**TSKgel SuperAWM-H, 9 µm, 6.0 mm ID × 15 cm × 2**
Mobile phase:	HFIP
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	40 °C
Injection vol.:	20 µL
Samples:	virgin and recycled nylon

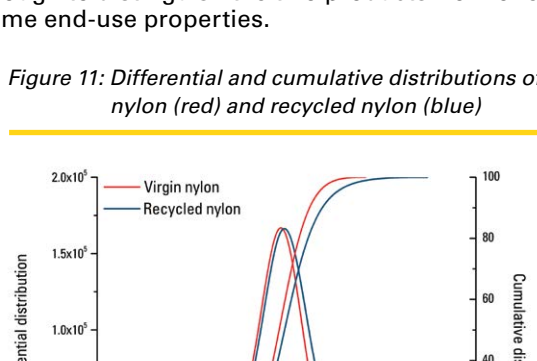
The molar mass averages and polydispersity index, *PDI*, as determined via a PMMA RI calibration curve are given in Table 3. A comparison of the molar mass averages and molar mass distribution, Figure 11, of the virgin nylon material with the recycled nylon material reveals an increase in the molar mass averages and breadth of the distribution curve of the recycled nylon compared to the molar mass averages of the virgin nylon. The molar mass averages and distributions of the virgin and recycled nylon samples obtained by GPC are different enough to distinguish the two products from one another but similar enough to both create successful products with the same end-use properties.

Table 3: Molar mass averages and polydispersity index of nylon samples via RI

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	*PDI*[a]
Virgin nylon	1.22×10^4 ± 46[b]	1.71×10^4 ± 75	2.29×10^4 ± 346	1.41 ± 0.01
Recycled nylon	1.33×10^4 ± 438	2.17×10^4 ± 210	3.93×10^4 ± 1,105	1.62 ± 0.05

[a] $PDI = M_w/M_n$; [b] Standard deviations from six injections

Figure 11: Differential and cumulative distributions of nylon (red) and recycled nylon (blue)



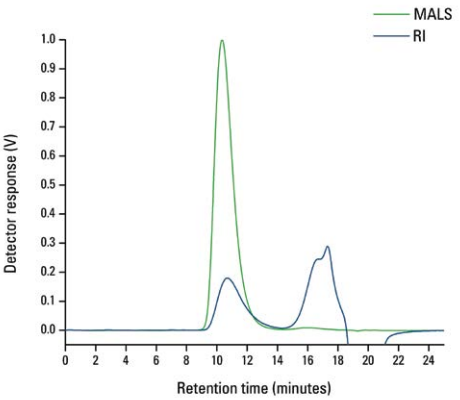
[3]Crespo, J.E; Parres, E.; Peydro, M.A.; Navarro, R. *Polym. Eng. Sci.*, **2013**, 53, 679-688.

Additives and Fillers in Commercial Polymers

Small quantities of additives and fillers are embedded in most commercial polymers in order to obtain certain desirable end-use properties. Typically additives and fillers are added to commercial polymers to improve compatibility of dissimilar elastomers, mixing, processing and surface tack, extrusion rates, appearance, and reinforcement. Commercial polymers can contain a wide variety of additives and fillers, some of which can easily be removed from the commercial polymer through filtering while others may require a separation method such as GPC. The ability to separate a commercial polymer from the various additives and fillers is necessary when analyzing the molar mass averages and distributions of a polymer as the additives and fillers can skew the molar mass averages and distributions.

An EcoSEC GPC System with a dual flow RI detector coupled to a multi-angle light scattering detector (MALS) was used to separate and identify the presence of an additive in a commercial rubber sample. Figure 12 shows the overlay of the GPC traces from the RI and MALS detectors. The RI detector shows two baseline resolved peaks while the MALS detector shows a single peak. The later eluting species, present only in the RI detector, are indicative of the additive, as materials polymeric in nature would be detectable by both the MALS and RI detectors. Additives are generally molecules low in molar mass and approaching the detection limit of the MALS detector (~1,000 g/mol) but present at a fairly high concentration, thus detectable by the concentration sensitive detector.

Figure 12: GPC elution profile of a rubber sample with additives as monitored by RI (blue) and MALS (green)



Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 3**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System), MALS (Wyatt DAWN 8+)
Temperature:	35 °C
Injection vol.:	30 µL
Sample:	rubber

The baseline separation of the rubber from the additive allows for the determination of the polystyrene relative molar mass averages of both species and the absolute molar mass averages of the rubber, Table 4. The polystyrene relative and absolute molar mass averages obtained for the rubber are not expected to match, as the polystyrene relative values are dependent on the chemistry and architecture of the sample and standards. The dual detector GPC set-up allows for the identification of the presence of an additive and determination of the molar mass averages of both the rubber and additive within the commercial polymer sample.

Table 4: Molar mass averages and polydispersity index of a rubber sample and additive via RI and MALS

Sample (Detection Method)	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI[a]
Rubber (RI)	$1.33 \times 10^5 \pm 0.02^b \times 10^5$	$3.10 \times 10^5 \pm 0.02 \times 10^5$	$4.80 \times 10^5 \pm 0.03 \times 10^5$	2.33 ± 0.01
Additive (RI)	455 ± 6	$1.06 \times 10^3 \pm 0.01 \times 10^3$	$2.42 \times 10^3 \pm 0.04 \times 10^3$	2.33 ± 0.02
Rubber (MALS)	$3.98 \times 10^5 \pm 0.39 \times 10^5$	$7.34 \times 10^5 \pm 0.21 \times 10^5$	$1.08 \times 10^6 \pm 0.21 \times 10^5$	1.849 ± 0.126

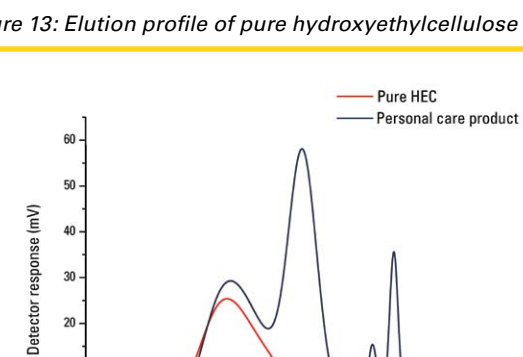
[a] $PDI = M_w/M_n$; [b] Standard deviations from four injections

Polymers in Personal Care Products

Cosmetic and personal care companies are interested in the ability to characterize one of the most highly used non-ionic, water soluble polymers in their formulations, hydroxyethylcellulose (HEC). HEC is derived from cellulose and used in products such as shampoos, body washes, shower gels, and eye drops as it has the ability to thicken solutions and reduce the amount of suds or foam they form. The characterization of pure HEC and HEC within a personal care product was performed utilizing the EcoSEC GPC System with an internal dual flow RI detector and semi-micro columns for polymer analysis in an aqueous mobile phase.

The chromatograms of the pure HEC and the HEC within a personal care product, as monitored by the RI detector, are shown in Figure 13. The elution profile of the pure HEC displays the presence of one species while the personal care product displays a distinctive bimodal distribution in the location of the pure HEC as well as two additional components in the low molar mass region of the chromatogram. The bimodal distribution in the HEC region of the chromatogram for the personal care product could be a result of either two completely different polymer species in the product or the presence of two distinctive size (molar mass) distributions of HEC in the product with the lower molar mass portion of the HEC being present at a higher concentration than the high molar mass portion. The two later eluting species in the chromatogram for the personal care product are two additional components of the product that are significantly smaller in size than the main polymeric components of the product.

Figure 13: Elution profile of pure hydroxyethylcellulose and hydroxyethylcellulose in a personal care product



Column:	**TSKgel SuperMultiporePW-H, 8 µm, 4.6 mm ID × 15 cm × 3**
Mobile phase:	H_2O with 0.1 mol/L $NaNO_3$ and 0.02% NaN_3
Flow rate:	0.50 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	35 °C
Injection vol.:	25 µL
Sample:	hydroxyethylcellulose

The polyethylene oxide and polyethylene glycol RI relative molar mass averages of the pure HEC and the HEC within a personal care product are given in Table 5. The molar mass averages for the HEC within the personal care product were shown to vary from that of the pure HEC when the molar mass averages of both components in the HEC region of the chromatogram for the personal care product were determined collectively and separately. The molar mass distribution of the pure HEC and the HEC region of the personal care product indicate a polydisperse polymer as *PDI*=9.82 and *PDI*=12.64 (collectively) or *PDI*= 2.27 and 1.59 (separately), respectively.

Table 5: Molar mass averages and polydispersity index of pure hydroxyethylcellulose and hydroxyethylcellulose in a personal care product

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	*PDI*[a]
Pure HEC	$1.50 \times 10^5 \pm 0.04$[b] $\times 10^5$	$1.47 \times 10^6 \pm 0.01 \times 10^6$	$5.93 \times 10^6 \pm 0.01 \times 10^6$	9.82 ± 0.20
HEC in a personal care product (collectively)	$4.67 \times 10^4 \pm 0.01 \times 10^4$	$5.89 \times 10^5 \pm 0.02 \times 10^5$	$2.78 \times 10^6 \pm 0.06 \times 10^6$	12.61 ± 0.03
HEC in a personal care product (separately)	$5.21 \times 10^5 \pm 0.06 \times 10^5$ $2.69 \times 10^4 \pm 0.07 \times 10^4$	$1.12 \times 10^6 \pm 0.04 \times 10^6$ $4.32 \times 10^4 \pm 0.09 \times 10^4$	$2.47 \times 10^5 \pm 0.16 \times 10^5$ $6.38 \times 10^4 \pm 0.01 \times 10^4$	2.29 ± 0.01 1.61 ± 0.23

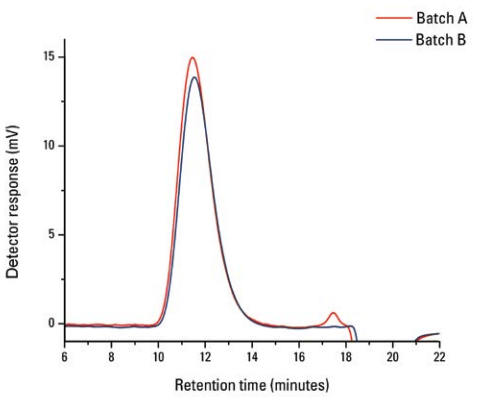
[a] $PDI = M_w/M_n$; [b] Standard deviations from four injections

Utilities of GPC in Industry

One of the primary focuses of the polymer and plastics industries is the ability to differentiate polymers in a sustainable and time effective manner. Currently GPC methods are being used to distinguish polymers based on molar mass or hydrodynamic volume (size) in solution, as GPC is a fast, reliable, and robust method for polymer characterization. Most companies involved in the manufacturing and development of end-use products that involve polymers rely heavily on GPC. Throughout the polymer and plastics industries, the EcoSEC GPC System is used to detect differences from batch-to-batch or lot-to-lot of a given polymer, to monitor reaction processes, to determine variations in molar mass averages obtained through different synthesis routes, and to distinguish between polymers with the same chemical compositions but different end-use properties, to name a few.

Some of the utilities of the EcoSEC GPC System in the polymer and plastics industries are shown in Figures 14-16. Figure 14 compares the GPC elution profiles of two different batches of a PMMA based molding resin that can be used in automotive, home appliances, and electronics. Batch A extends further in the larger polymeric size, shorter retention time direction of the GPC elution profile than Batch B, an indication that the two batches differ in polymeric size. The slight variation in the GPC elution profile results in an approximately 10% difference in the poly(methyl methacrylate) molar mass averages between the two batches, Table 6. The difference in molar mass averages between Batch A and Batch B may or may not affect the end-use properties of a given polymer as the polydispersity index, *PDI*, remains essentially constant amongst the two batches.

Figure 14: GPC elution profiles of two different batches of a PMMA based molding resin



Columns:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 2 + TSKgel SuperHZ2500, 3 µm, 4.6 mm ID × 15 cm × 1**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	35 °C
Injection vol.:	20 µL
Sample:	PMMA based molding resin

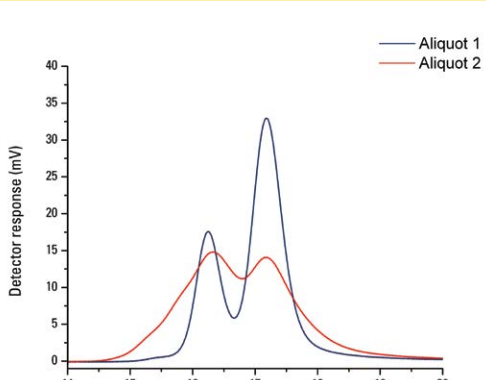
Table 6: Molar mass averages and polydispersity index of two different batches of a PMMA based molding resin

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	*PDI*[a]
Batch A	$6.59 \times 10^4 \pm 0.15^{b} \times 10^4$	$1.38 \times 10^5 \pm 0.02 \times 10^5$	$2.24 \times 10^5 \pm 0.03 \times 10^5$	2.11 ± 0.02
Batch B	$5.90 \times 10^4 \pm 0.10 \times 10^4$	$1.24 \times 10^5 \pm 0.01 \times 10^5$	$2.02 \times 10^5 \pm 0.03 \times 10^5$	2.11 ± 0.03

[a] $PDI = M_w/M_n$; [b] Standard deviations from four injections

An example of using the EcoSEC GPC System to monitor a reaction process is shown in Figure 15 by overlaying aliquots of a reaction collected thirty minutes apart. Each aliquot produces a different GPC elution profile which can be used to determine if the reaction process taking place is correct through a comparison process with known GPC elution profiles for various stages of the reaction. In general for this sample as the reaction process progresses the two individual components, indicated by the distinctive bimodal GPC elution profile of aliquot 1, blend to become one component in the final product, indicated by the decrease in the bimodality of aliquot 2.

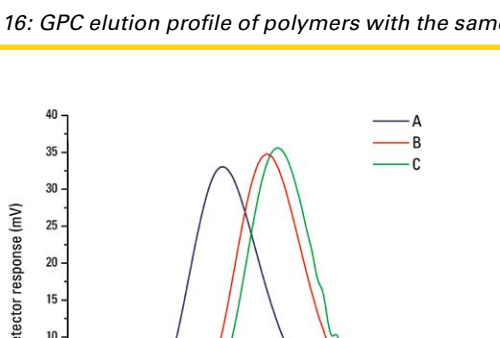
Figure 15: GPC elution profile of aliquots of a reaction collected thirty minutes apart



Column:	**TSKgel GMHXL, 9 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	35 °C
Injection vol.:	100 µL
Sample:	synthetic rubber

The use of the EcoSEC GPC System to distinguish between polymers obtained through different synthesis routes with the same chemical composition but different end-use properties is shown in Figure 16. The GPC elution profile for three polyimide samples shows a variation in retention time, thus also in the molar mass averages, Table 7. While these three polyimide samples are composed of the same chemical composition, the samples are shown to have different end-use properties due to differences in their molar mass averages and molar mass distributions.

Figure 16: GPC elution profile of polymers with the same chemical composition but different end-use properties



Column:	**TSKgel GMHXL, 9 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	DMF with 0.02 mol/L LiBr
Flow rate:	1.0 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	35 °C
Injection vol.:	100 µL
Sample:	polyimides

Table 7: Molar mass averages and polydispersity index of polymers with the same chemical composition but different end-use properties

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI[a]
A	$3.98 \times 10^4 \pm 0.01^b \times 10^4$	$6.47 \times 10^4 \pm 0.01 \times 10^4$	$8.98 \times 10^4 \pm 0.01 \times 10^4$	1.62 ± 0.02
B	$1.86 \times 10^4 \pm 0.01 \times 10^4$	$2.87 \times 10^4 \pm 0.01 \times 10^4$	$3.95 \times 10^4 \pm 0.01 \times 10^4$	1.54 ± 0.01
C	$1.53 \times 10^4 \pm 0.01 \times 10^4$	$2.34 \times 10^4 \pm 0.01 \times 10^4$	$3.20 \times 10^4 \pm 0.01 \times 10^4$	1.52 ± 0.01

[a] $PDI = M_w/M_n$; [b] Standard deviations from four injections

Polymer-Based Therapeutics

Polymer–based drug and gene delivery systems began to emerge from the laboratory benches about 30 years ago as a promising therapeutic strategy for treatment of devastating human diseases. Polymeric materials are useful for solving drug delivery problems as they are relatively large compared to low molar mass drugs, and when combined with these drugs they can augment the drug's performance and change their bioavailability.[4] The use of synthetic polymers in therapeutics is continuously growing, thus increasing the need for a method to characterize the molar mass averages and molar mass distributions of these polymers as variations in molar mass averages and molar mass distributions can affect aspects of the therapeutic such as in vitro binding activity and biodegradation.[5] The molar mass averages and molar mass distributions of a polymer being used in therapeutics is critical for designing an effective polymer-based therapeutic and is most commonly characterized using GPC.

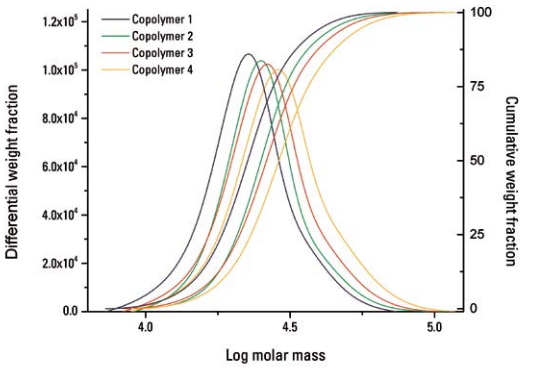
The EcoSEC GPC System was used to determine the molar mass averages and distributions of four block copolymers intended to be used in polymer-based drug or gene delivery systems. The polystyrene relative molar mass averages, M_n, M_w, and M_z, are given in Table 8. The variation of the molar mass averages for the four block copolymers may be great enough to affect the role the polymer plays in the polymer-based therapeutic within the body. For example, the molar mass of the polymer can influence the biodegradation of synthetic polymer in the body, thus resulting in the production of lower molar mass polymer that has different biological effects. In addition to the molar mass averages, the molar mass distribution can also influence various properties of therapeutics. The molar mass distributions of the four block copolymers are compared in Figure 17.

Table 8: Molar mass averages and polydispersity index of four block copolymers for use in a polymer-based therapeutic

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI[a]
Copolymer 1	2.09×10^4 $\pm 0.01^b \times 10^4$	2.38×10^4 $\pm 0.01 \times 10^4$	2.70×10^4 $\pm 0.01 \times 10^4$	1.13 ± 0.01
Copolymer 2	2.38×10^4 $\pm 0.01 \times 10^4$	2.64×10^4 $\pm 0.01 \times 10^4$	2.93×10^4 $\pm 0.01 \times 10^4$	1.11 ± 0.01
Copolymer 3	2.48×10^4 $\pm 0.01 \times 10^4$	2.81×10^4 $\pm 0.01 \times 10^4$	3.22×10^4 $\pm 0.01 \times 10^4$	1.14 ± 0.01
Copolymer 4	2.74×10^4 $\pm 0.01 \times 10^4$	3.10×10^4 $\pm 0.01 \times 10^4$	3.55×10^4 $\pm 0.01 \times 10^4$	1.14 ± 0.01

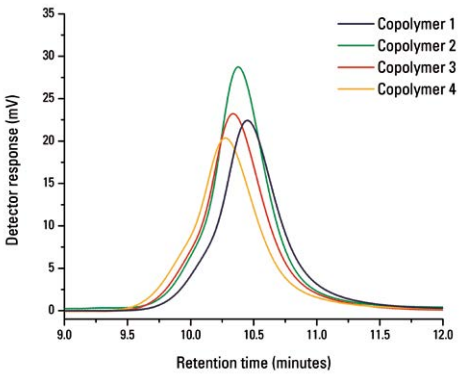
[a] $PDI = M_w/M_n$; [b] Standard deviations from four injections

Figure 17: Overlay of cumulative and differential molar mass distribution of four block copolymers for use in a polymer-based therapeutic



Information regarding the differences between the four block copolymers for use in a polymer-based therapeutic can be seen by comparing their GPC elution profiles, Figure 18. The shift in GPC retention time amongst the four block copolymers indicates a variation in polymeric size between the block copolymers, as elution order in GPC is that of an "inversing-sieving" technique, large analytes sample a smaller pore volume than smaller analytes resulting in the larger analytes eluting from the column prior the smaller analytes. Variations in polymeric size within a polymer-based therapeutic can dramatically affect its behavior within a biological system.

Figure 18: GPC elution profile of four block copolymers for use in a polymer-based therapeutic



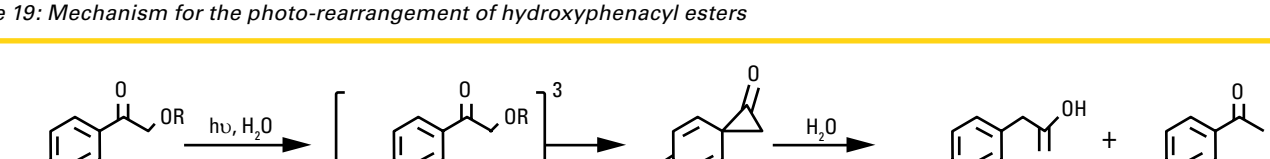
Columns:	**TSKgel SuperHZ4000, 3 µm, 4.6 mm ID × 15 cm + TSKgel SuperHZ3000, 3 µm, 4.6 mm ID × 15 cm + TSKgel SuperHZ2000, 3 µm, 4.6 mm ID × 15 cm**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	35 °C
Injection vol.:	10 µL
Sample:	block copolymer

[4]Kabanov, A.V.; Okano, T. Challenges in Polymer Therapeutics. In *Polymer Drugs in the Clinical Stage: Advantages and Prospects*, Volume 519; Maeda, H.; Kabanov, A.V.; Kataoka, K., Okano, T. eds.; Academic Press: New York, 2003; pp 1-20.

Photodegradable Polymer Degradation Analysis

Due to the need for polymers that are both photodegradable and biodegradable, Dr. Abraham Joy and his colleagues at the University of Akron have developed polycarbonate materials based on the alkoxyphenacyl photoactive moiety.[6] This new class of polymers is mechanically robust, biodegradable, and stable to high temperatures in the absence of light with potential applications in controlled drug release devices, ocular implants, and dermal patches. Upon radiation, the photoactive moiety undergoes a Favorski type of rearrangement, resulting in two major products, the phenylacetic acid derivative and the reduced acetophenone (Figure 19).[7]

Figure 19: Mechanism for the photo-rearrangement of hydroxyphenacyl esters



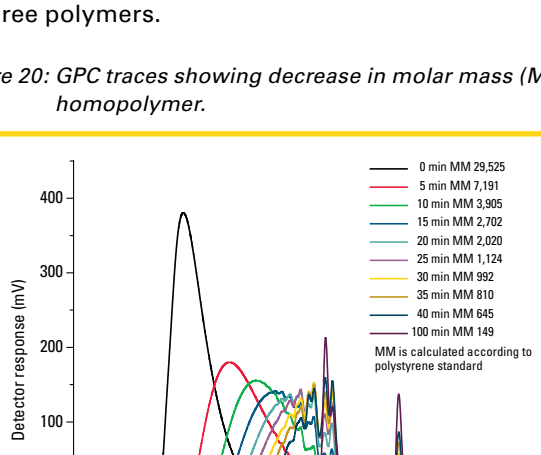
The EcoSEC GPC System was used to determine the polystyrene relative molar mass averages, M_n and M_w, and the polydispersity index, *PDI*, of an alkoxyphenacyl-based polycarbonate homopolymer, 5% PEG copolymer, and 10% PEG copolymer, all given in Table 10. The *PDI*s of the 5% and 10% PEG copolymer are smaller than the *PDI* of the homopolymer because the PEG copolymer samples were fractioned twice and the homopolymer was fractioned only once.

Table 10: Molar mass distributions and polydispersity index for homopolymer and copolymers

Composition	M_n (g/mol)	M_w (g/mol)	*PDI*
Homopolymer	1.29×10^4	2.95×10^4	2.3
5% PEG	2.27×10^4	2.63×10^4	1.2
10% PEG	8,810	1.04×10^4	1.2

Photodegradation of the homopolymer and copolymers was investigated by irradiation of the polymers in chloroform in a Rayonet reactor at 300 nm. Figure 20 shows GPC traces indicating time-dependent degradation with a 75% reduction in average molar mass within 5 minutes of irradiation. Subsequent analysis (data not shown) shows similar degradation for all three polymers.

Figure 20: GPC traces showing decrease in molar mass (M_w) with increasing radiation time for the alkoxyphenacyl-based polycarbonate homopolymer.



Columns:	**TSKgel SuperH3000, 3 µm, 6.0 mm ID x 15 cm x 2 + TSKgel SuperH4000, 3 µm, 6.0 mm ID x 15 cm x 1**
Mobile phase:	chloroform
Flow rate:	0.38 mL/min
Detection:	UV (EcoSEC GPC System @ 278 nm)
Temperature:	40 °C
Injection vol.:	10 µL
Sample:	alkoxyphenacyl polycarbonate homopolymer, 1 - 2 mg/mL

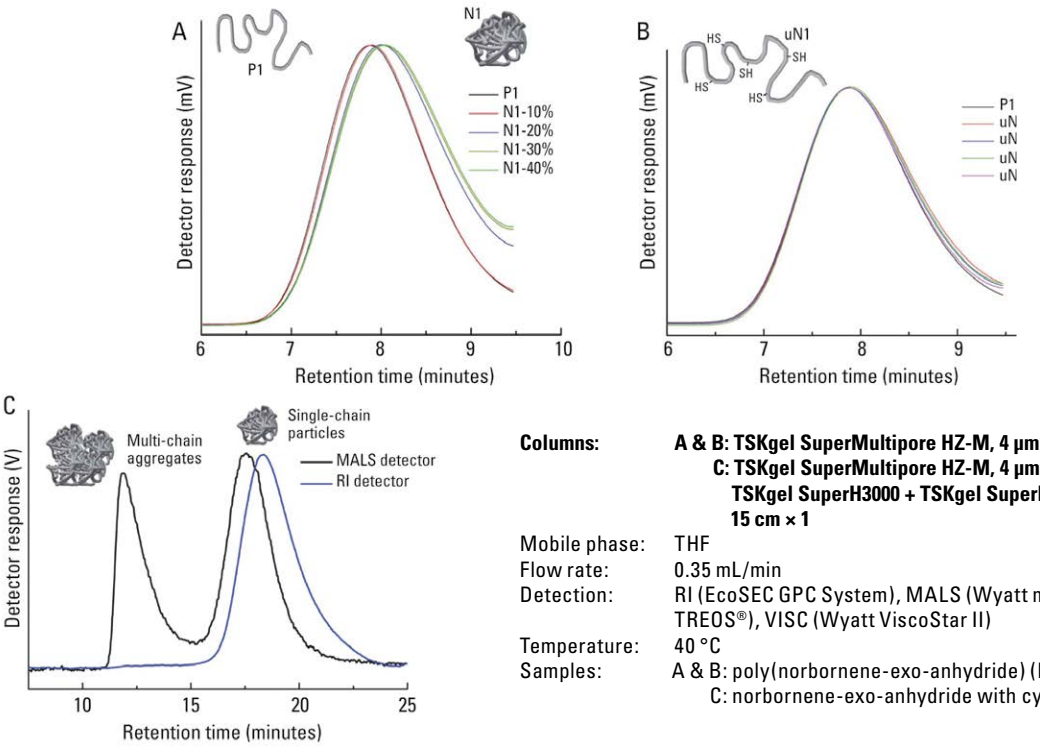
[6]Sun, S.; Chamsaz, E. A.; Joy, A. *Macro Lett.*, **2012**, 1 (10), 1184–1188.
[7]Givens, R. S.; Heger, D.; Hellrung, B.; Kamdzhilov, Y.; Mac, M.; Conrad, P. G.; Cope, E.; Lee, J. I.; Mata-Segreda, J. F.; Schowen, R. L.; Wirz, J. J. *Am. Chem. Soc.* **2008**, 130, 3307-3309.

Single-chain Polymer Nanoparticles

Dr. Erik Berda's research group at the University of New Hampshire is working on the fabrication and characterization of single-chain polymer nanoparticles (SCNPs) that can reversibly undergo a coil to particle transition via formation and cleavage of intramolecular disulfide cross-links.[8] In their initial studies Dr. Berda's group synthesized poly(norbornene-exo-anhydride) (P1), via ROMP using third generation Grubbs catalyst as an initiator and controlled the degree of collapse that occurs during nanoparticle (N1) formation by varying the amount of difunctional cross-linker added. The coil to particle transition was then characterized using the EcoSEC GPC System with dual flow RI via polystyrene relative molar mass averages. Figure 21A shows a series of GPC traces for P1 and its corresponding N1 after various extents of intramolecular cross-linking. As expected, an increase in GPC retention time is observed as the intramolecular cross-linking reaction progresses. This is due to a decrease in hydrodynamic volume that occurs as the coil collapses. Once the folding of the chains into SCNP was confirmed via the GPC retention times, dithiotheritol was introduced to unfold the N1 back to their original conformation. The transition from particle to coil was also confirmed via decreased GPC retention time, signifying an increase in hydrodynamic volume, Figure 21B.

To complement their initial studies Dr. Berda's group synthesized a second polymer, norbornene-exo-anhydride with cyclooctadiene (COD) (P2), to characterize via triple-detector GPC. For the characterization of P2, the EcoSEC GPC System with dual flow RI was coupled to multi-angle light scattering (MALS) and differential viscometry (VISC). The effectiveness of the triple-detector GPC system was highlighted by determining the difference between single-chain and multi-chain behavior. Figure 21C shows an overlay of the MALS and RI traces when the intra-molecular cross-linking reaction was extended with a slight excess of the cross-linker to encourage intermolecular coupling. The RI detector shows a single peak that can be attributed to single-chain particles, while the MALS detector shows two peaks of nearly equal intensity. The later eluting MALS peak corresponds to the single-chain particles while the early eluting peak is that of multi-chain aggregates, which are present at a negligible concentration as indicated by the RI detector. For this particular sample analysis, single-detector GPC would not have revealed the presence of the larger aggregates.

Figure 21: Single-chain polymer nanoparticles



Columns: **A & B: TSKgel SuperMultipore HZ-M, 4 µm, 4.6 mm ID × 15 cm × 2**
C: TSKgel SuperMultipore HZ-M, 4 µm, 4.6 mm ID × 15 cm × 2 + TSKgel SuperH3000 + TSKgel SuperH4000, 3 µm, 4.6 mm ID × 15 cm × 1
Mobile phase: THF
Flow rate: 0.35 mL/min
Detection: RI (EcoSEC GPC System), MALS (Wyatt miniDAWN™ TREOS®), VISC (Wyatt ViscoStar II)
Temperature: 40 °C
Samples: A & B: poly(norbornene-exo-anhydride) (P1)
C: norbornene-exo-anhydride with cyclooctadiene

[8]Tuten, B.T.; Chao, D.; Lyon C.K.; Berda, E.B. *Polym. Chem.* **2012**, 3, 3068-3071.

HFIP Reproducibility

Dr. Li Jia and co-workers at the University of Akron are investigating different synthetic routes for the formation of polypeptoids with alternating block structures. Highly reproducible data is needed to obtain subtle molar mass distribution trends from the various synthetic routes. The EcoSEC GPC System and a set of TSKgel mixed bed columns were used successfully to obtain high quality molar mass distribution (MMD) data of a series of Dr. Jia's block poly-ß-alkylalanoids with hexafluoroisopropanol (HFIP) as the mobile phase in under 15 minutes.

As shown in Table 11, percent standard deviations are more than 10x lower than values previously reported for polyamides in HFIP.[9] Percent relative standard deviation of the polydispersity index (*PDI*) ranged from 0.1 to 0.5%, permitting one to report *PDI*s within three significant figures. The high precision of the EcoSEC GPC System allows for the detailed study of polymerization reactions.

Table 11: Averaged values from three consecutive injections and the percent relative standard deviations

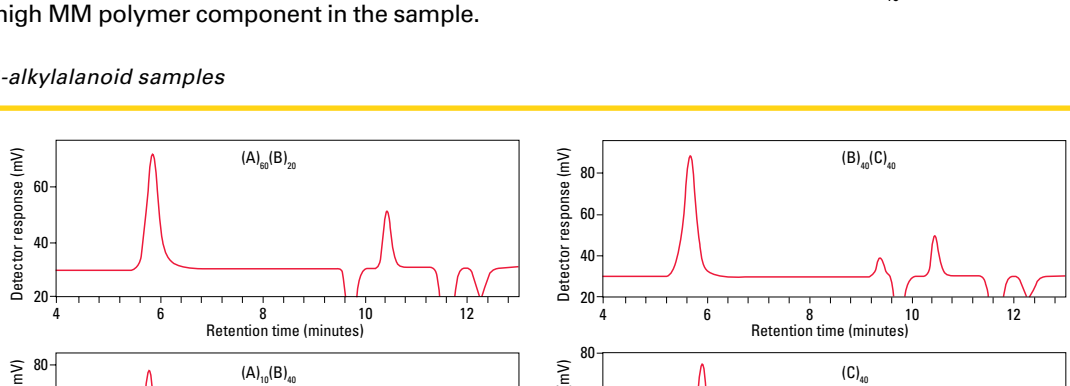
Sample[a]	M_n[b] (g/mol)		M_w[b] (g/mol)		*PDI*[b]	
		Rel std dev		Rel std dev		Rel std dev
$(A)_{10}(B)_{40}$	$2.65 \times 10^4 \pm 10$	0.04%	$3.03 \times 10^4 \pm 30$	0.11%	1.14 ± 0.01	0.09%
$(A)_{60}(B)_{20}$	$3.33 \times 10^4 \pm 170$	0.52%	$4.07 \times 10^4 \pm 28$	0.07%	1.22 ± 0.01	0.50%
$(A)_{40}(B)_{40}$	$4.87 \times 10^4 \pm 220$	0.45%	$6.09 \times 10^4 \pm 160$	0.26%	1.25 ± 0.01	0.10%
$(C)_{40}$	$3.01 \times 10^4 \pm 50$	0.18%	$3.64 \times 10^4 \pm 140$	0.37%	1.21 ± 0.01	0.39%

[a] Block lengths were determined by Dr. Jia from independent measurements. Chemical composition of blocks A, B and C will be published by L. Jia.

[b] Molar mass data were obtained from a PMMA calibration curve. Molar mass averages given in the table are averages of three sequential injections per sample. Based on block lengths, MMD are significantly overestimated.

Sample chromatograms from 4 selected poly-ß-alkylalanoid samples run on an EcoSEC GPC System using two TSKgel GMHHR-M, 5 µm, 4.6 mm ID × 15 cm columns are shown in Figure 22. Sample profiles display very little tailing and no baseline drift, allowing for highly precise data not available with conventional systems. All samples, with the exception of $(C)_{40}$, contain almost symmetrical, narrow polymer profiles eluting around 6 minutes. The shoulder seen in $(C)_{40}$ is indicative of another population of a high MM polymer component in the sample.

Figure 22: Poly-ß-alkylalanoid samples



Column: **TSKgel GMHHR-M, 5 µm, 4.6 mm ID × 15 cm × 2 packed in HFIP**
Mobile phase: HFIP containing 5 mmol/L sodium trifluoroacetate
Flow rate: 0.35 mL/min
Detection: RI (EcoSEC GPC System)
Temperature: 40 °C
Injection vol.: 10 µL
Samples: selection of poly-ß-alkylalanoid samples

[9]Robert, E. C.; Bruessau, R.; Dubois, J.; Jacques, B.; Meijerink, N.; Nguyen, T. Q.; Niehaus, D. E.; Tobisch, W. A. *Pure Appl. Chem.* **2004**, 76, 2009–2025.

Analysis of Styrene and Isoprene Block Copolymers

Dr. Jimmy Mays' group from the Department of Chemistry at the University of Tennessee, Knoxville, is synthesizing and characterizing the bulk morphology of fluorinated and sulfonated block copolymers. Well-defined block copolymers of sulfonated polystyrene-*b*-fluorinated polyisoprene (*s*PS-*b*-*f*PI), Figure 23, were synthesized by anionic polymerization followed by fluorination and sulfonation.[10] The EcoSEC GPC System, equipped with TSKgel SuperMultiporeHZ columns, was then used to determine the number-average molar mass, M_n, and the polydispersity index, *PDI*, of *s*PS-*b*-*f*PI, as well as that of the precursor polymer (PS-*b*-PI), Table 12. As seen in Figure 24, complete analysis of *s*PS-*b*-*f*PI was obtained in less than 10 minutes with excellent resolution using the EcoSEC GPC System.

Figure 23: Structure of sulfonated polystyrene-b-fluorinated polyisoprene (sPS-b-fPI)

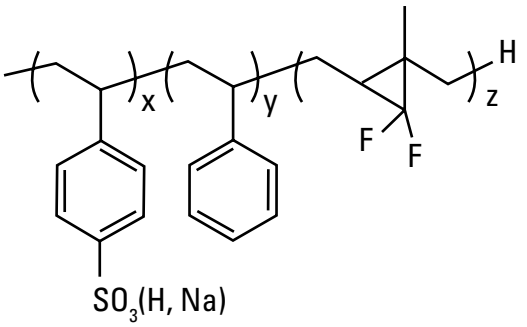
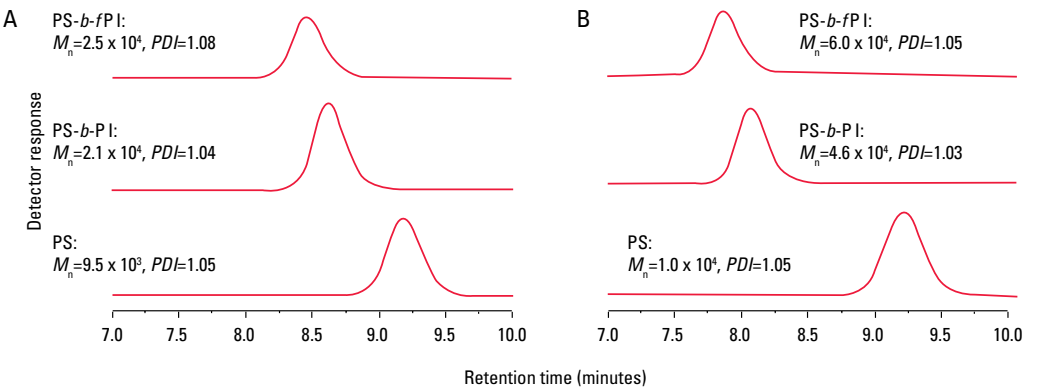


Table 12: Number-average molar mass, M_n, and the polydispersity index (PDI) of sPS-b-fPI and the precursor polymer (PS-b-PI)

	PS-*b*-PI		*s*PS-*b*-fPI	
Series[a]	M_n (g/mol)	*PDI*	M_n (g/mol)	*PDI*
1	2.1 x 10^4	1.04	2.5 x 10^4	1.08
2	4.6 x 10^4	1.03	6.0 x 10^4	1.05

[a] series 1 in acid form; series 2 in Na form

Figure 24: Sulfonated polystyrene-b-fluorinated polyisoprene precursor samples



Column:	**TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID x 15 cm**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI (EcoSEC GPC System)
Temperature:	35 °C
Injection vol:	20 µL
Samples:	A. series 1, table 12 B. series 2, table 12

[10]Wang, X.; Hong, K.; Baskaran, D.; Goswami, M.; Sumpter, B.; Mays, J. *Soft Matter*, **2011**, 7, 7960.

EcoSEC High Temperature GPC System

Engineered to deliver the following:

Superior Performance

- Baseline Stability
- Reproducibility
- Reliability

Unparalleled Versatility

- Ease of Use
- All-in-One Design

Thermal Stability

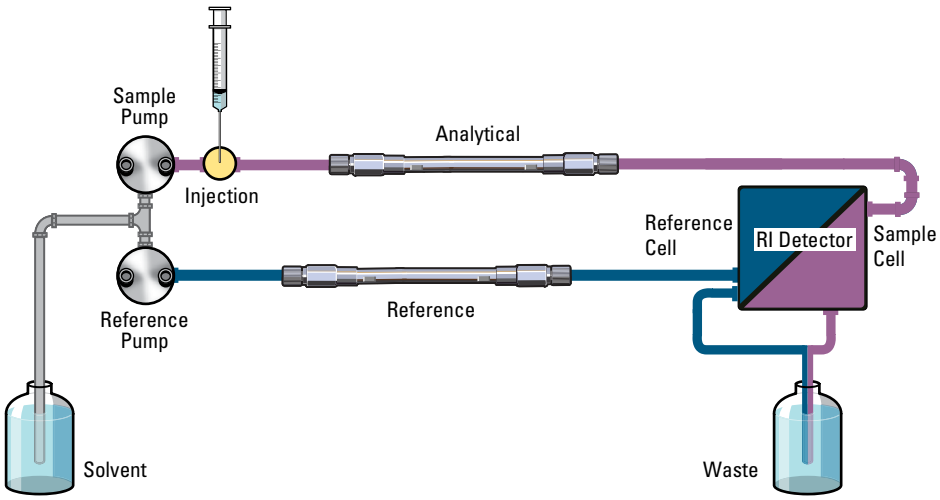
- Heated Solvent Holder
- Complete Thermal Precision

Superior Performance

Baseline Stability

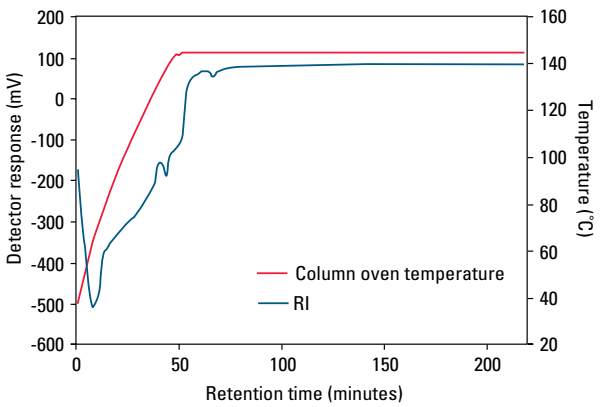
Incorporated into the design of our two pump delivery system is 40+ years experience in engineering. The EcoSEC High Temperature GPC System has a unique dual flow design which includes the use of two pumps. Figure 1 demonstrates the flow paths of the sample and reference pumps. The sample pump flows solvent from the solvent reservoir through the following system components in sequence: autosampler, analytical column, sample side of RI detector cell, and waste container. The solvent flows via the reference pump from the solvent reservoir through a reference column, the reference side of the RI detector cell, and then the waste container. The entire flow system is temperature controlled to eliminate the effects of fluctuations in ambient temperature.

Figure 1: Flow paths of sample and reference pumps in the EcoSEC High Temperature GPC System



On the EcoSEC High Temperature GPC System the RI baseline is considered stabilized when the drift in the signal is 3.0×10^{-7} RIU/h or less. When a new set of columns is manually placed on the EcoSEC High Temperature GPC System and the flow rate and temperature controls are started, the RI baseline stabilizes within 3 hours. Figure 2 demonstrates the equilibration time from start-up of the EcoSEC High Temperature GPC System in orthodichlorobenzene (ODCB).

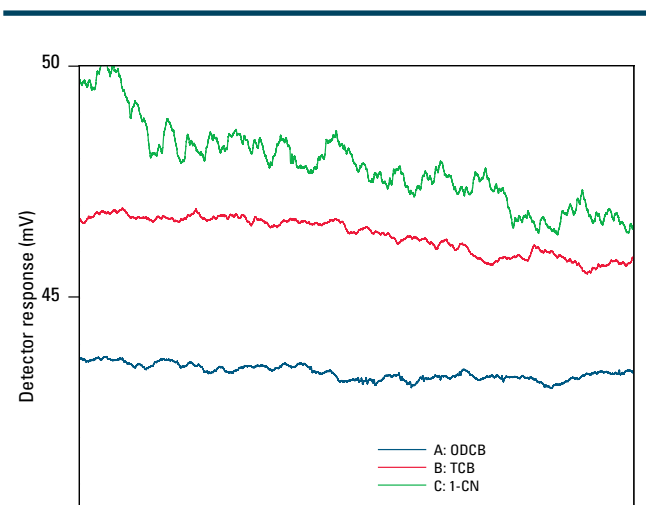
Figure 2: Refractive index detector signal during equilibration of the EcoSEC High Temperature GPC System



Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	ODCB with 0.05% BHT
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	145 °C

Advanced engineering, along with complete temperature control and a dual flow RI detector, means rock steady baselines in even the most challenging solvents and temperatures. The RI baselines as obtained for three commonly used high temperature GPC solvents: Trichlorobenzene (TCB) at 145 °C, orthodichlorobenzene (ODCB) at 145 °C and 1-chloronaphthalene (1-CN) at 210 °C are shown in Figure 3. The RI baseline drift for all three solvents is less than 1 mV/h.

Figure 3: Baseline drift of the dual flow refractive index detector of the EcoSEC High Temperature GPC System for TCB, ODCB, and 1-CN



Column: **TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase: A: ODBC
B: TCB
C: 1-CN
Flow rate: 1.0 mL/min
Detector: RI (EcoSEC High Temperature GPC System)
Temperature: A and B: 145 °C
C: 210 °C

The unmatched baseline stability of the dual flow RI detector in the EcoSEC High Temperature GPC System is also shown in Table 1 through the drift, fluctuation, and noise obtained when ODCB at 145 °C, TCB at 145 °C, 1-CN at 210 °C, and THF at 40 °C are used as the mobile phase.

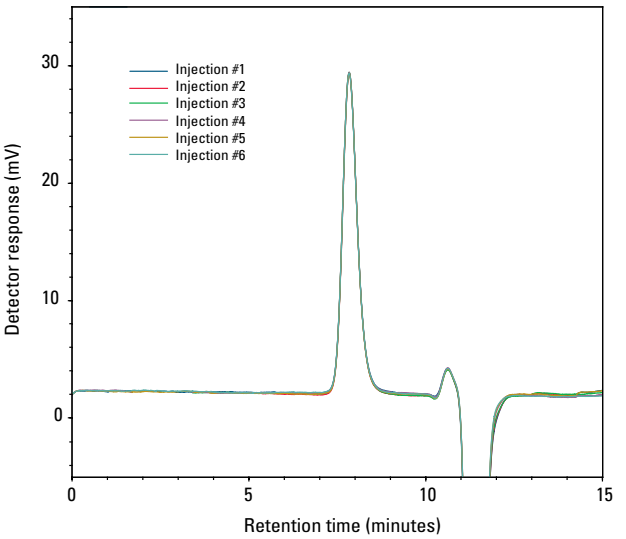
Table 1: Baseline drift, fluctuation and noise of the EcoSEC High Temperature GPC System in ODCB, TCB, 1-CN, and THF

Solvent (temperature)	Drift (mV/h)	Fluctuation (mV)	Noise (mV)
ODCB (145 °C)	-0.41	0.54	0.044
TCB (145 °C)	-1.30	0.69	0.046
1-CN (210 °C)	-0.91	1.61	0.098
THF (40 °C)	-0.35	0.23	0.022

Reproducibility

The dual flow design of the RI detector and the temperature controlled pumps of the EcoSEC High Temperature GPC System deliver precise flow rates at all temperatures, even when changes in environmental conditions occur, thus producing reproducible results sample after sample, day after day. The intraday and day-to-day reproducibility of the EcoSEC High Temperature GPC System are shown in Figure 4.

Figure 4: GPC elution profile of intraday reproducibility of the EcoSEC High Temperature GPC System



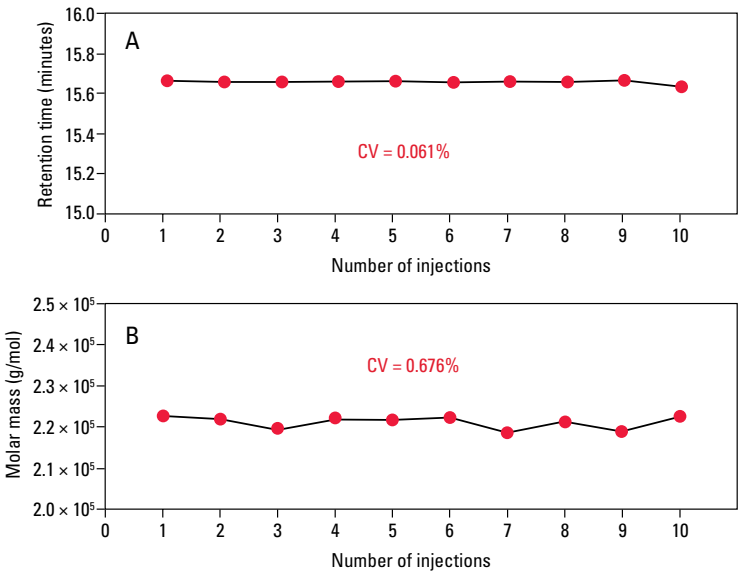
Reproducibility (intraday, n=6)
R.T.: CV 0.017%
Area: CV 0.42%

Reproducibility (day to day, n=5)
R.T.: CV 0.047%
Area: CV 0.71%

Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	ODCB with 0.05% BHT
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	145 °C
Injection vol.:	300 µL
Sample:	polystyrene (F-20), 0.02%

The engineering design concepts of the EcoSEC High Temperature GPC System result in a high degree of reproducibility of retention times (Figure 5A) and molar mass determinations (Figure 5B). The coefficients of variation for retention time and weight-average molar mass, M_w, are well below 1% for successive injections.

Figure 5A and 5B: A: Intraday retention time reproducibility, B: Intraday weight-average molar mass reproducibility



Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	ODCB with 0.05% BHT
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	145 °C
Injection vol.:	300 µL
Sample:	polypropylene

EcoSEC High Temperature GPC System Workstation Software

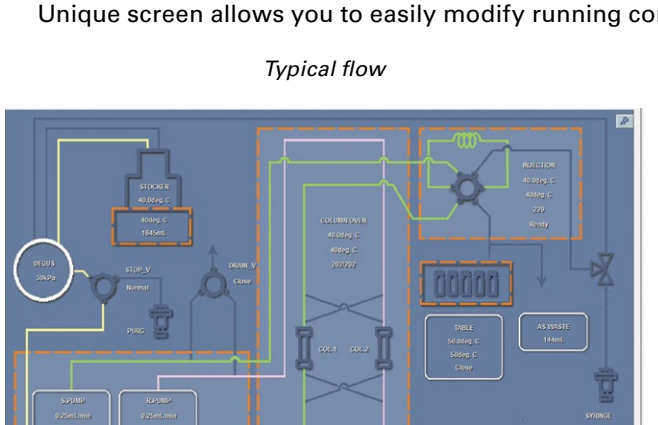
- GPC-specific EcoSEC High Temperature GPC System software to simplify system control and data handling
- Controls up to 2 EcoSEC High Temperature GPC Systems
- Excellent data handling and report generation
- Fully featured data handling system; analyze data from two detectors
- Start and stop system automatically
- One license for multiple locations

Features include:

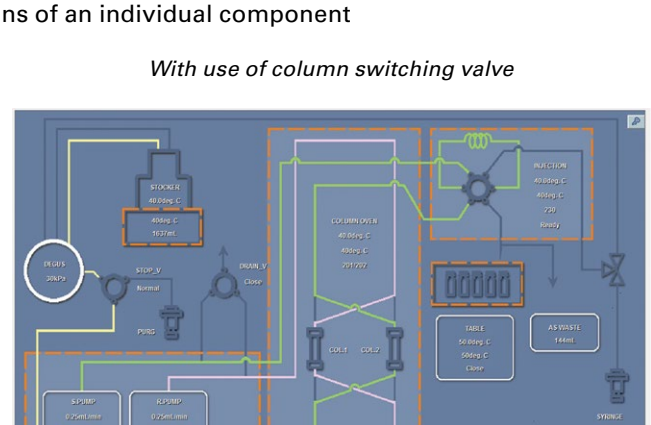
Flow Diagram

- Unique screen allows you to easily modify running conditions of an individual component

Typical flow

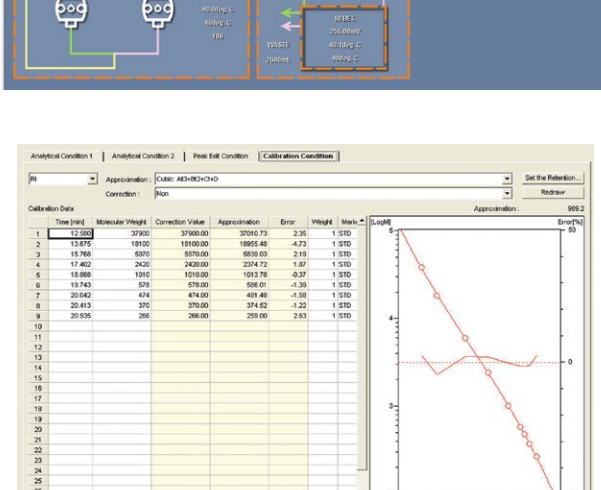


With use of column switching valve



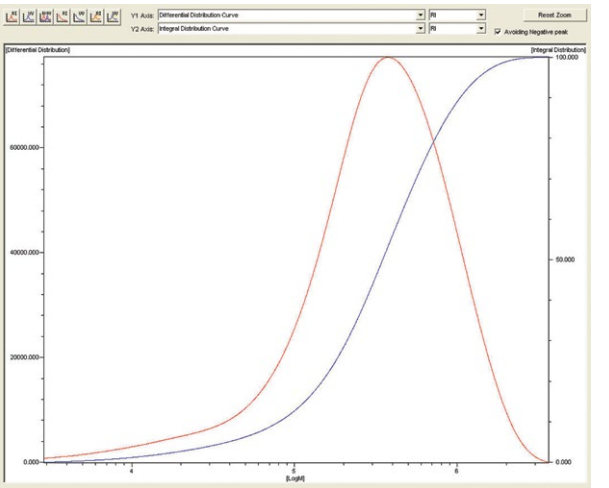
Method

- All parameters for data acquisition and peak integration, including baseline operations, are saved in the template method
- One click switching between calibration curves



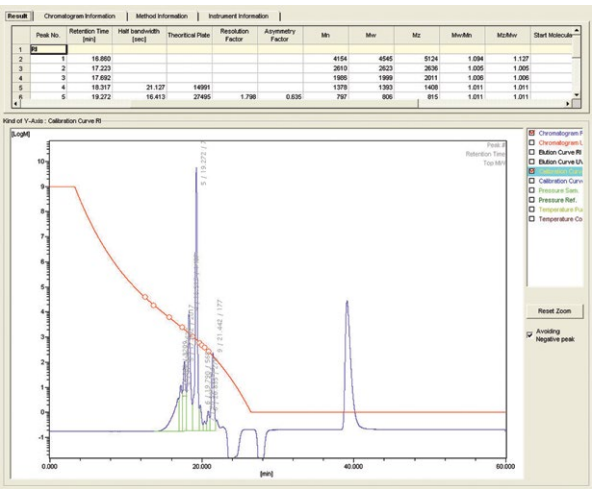
GPC Specific Quantitative Calculations

- M_n, M_w, and M_z molar mass averages
- Cumulative and differential molar mass plotting
- Polydispersity index (*PDI*) values



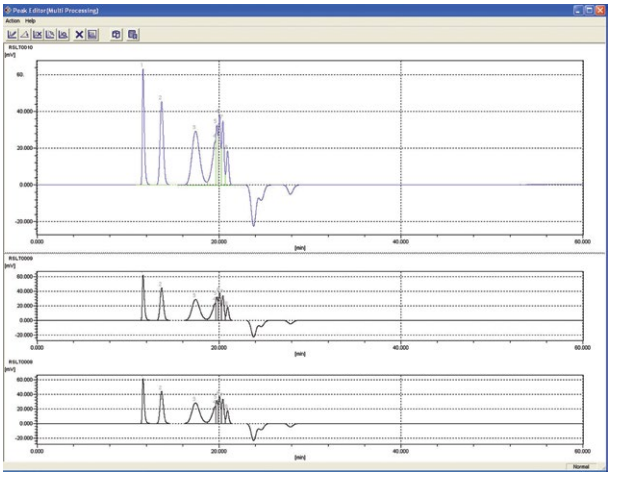
Data Management and Report Generation

- Allows viewing of chromatograms, elution, curve, flow rate, pressure, and temperature.
- Large number of built in reports
- Fully customizable reports
- Easily export data into text or pdf files.



Peak Editing and Multiprocessing Function

- Full editing functionality including baseline setting and peak splitting using the mouse
- Automatic peak editing
- Automatic application of peak detection and integration parameters to multiple chromatograms of the same sample using the multiprocessing function; resulting in identical processing for similar chromatograms for enhanced reproducibility.



Please see software specifications on page 26.

Enhanced EcoSEC GPC System Analysis: External Detectors and Accessories

The addition of multiple detection methods to the EcoSEC GPC System allows for the characterization of a variety of polymer properties. A multi-detector GPC set up can be used to determine:

- Polystyrene relative molar mass averages based on RI
- Branching, universal calibration, intrinsic viscosity, and hydrodynamic radius with viscometry detection
- Absolute molar mass averages and radius of gyration with multi-angle light scattering (MALS) detection

Sample Prep System

- Sample shaker 10 - 100 RPM
- 24 vial capacity
- Aluminum heated block
- 40 - 220 °C

Column Switching Valve

- Easily change between 2 column sets
- Equipped above column oven
- Manual switching
- Position is recognized by software

Tosoh Bioscience can tailor a system to meet your application needs.

Does your analysis require additional detectors beyond RI and UV?
The EcoSEC GPC System provides easy and effortless connectivity when using multi-detector configurations. We offer external light scattering and viscometry detectors.

Contact us for a quote!


TOSOH
TSKgel
TSKgel G2000HHR HT2
TOSOH
TSKgel
TSKgel G2000HHR HT2

Polypropylene: Random Copolymer

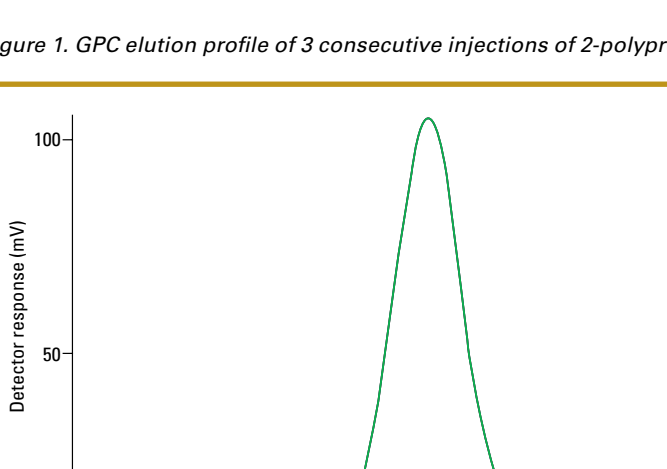
The polypropylene market is one of the largest most versatile polymer markets today, with over 50 million tons produced annually and sold into a wide variety of household and industrial applications. In the home, polypropylene can be found in everything from audio speakers to carpets and automotive components. Industrially, polypropylene is essential in living hinges, RF capacitors, medical devices, and contact lens molding.

The variety of products in which polypropylene is present require versatility in mechanical, thermal and chemical properties. For this reason, depending upon the application, three major categories of polypropylene exist: homopolymer, block copolymer and random copolymer. While homopolymer is the general purpose grade of polypropylene, block copolymers that usually containing 5-15% ethylene exhibit enhanced impact resistance. Random copolymers containing 1-7% ethylene are more malleable and crystal clear. For these reasons, random copolymers are often used in medical applications and contact lens production.

The molar mass averages and polydispersity of two polypropylene random copolymer samples via refractive index (RI) detection using the EcoSEC High Temperature GPC System and TSKgel columns were determined. The number, weight and z-average molar mass values (M_n, M_w, and M_z) and polydispersity index, *PDI*, were calculated for polypropylene equivalents via EcoSEC Workstation software by applying Mark-Houwink constants. The obtained values are given in Tables 1 and 2.

The enhanced thermal, flow rate, and dual flow RI detector stability of the EcoSEC High Temperature GPC System in combination with the excellent resolving power of the TSKgel GMH$_{HR}$-H (20) HT2 high temperature GPC columns produce reliable and highly reproducible data for two polypropylene random copolymer samples analyzed in triplicate (Figures 1 and 2). Very low variation in sample retention and superb baseline stability are observed when overlaying three consecutive RI injections of each sample.

Figure 1. GPC elution profile of 3 consecutive injections of 2-polypropylene random copolymer sample #1 as monitored by RI

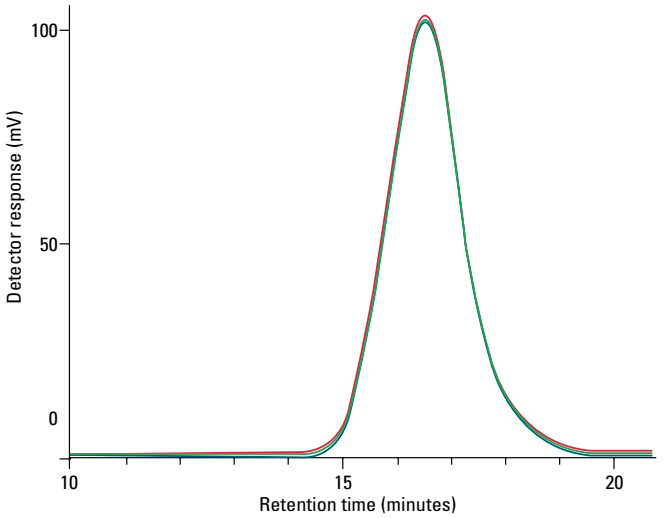


Column:	**TSKgel GMH$_{HR}$-H (20) HT2, 20 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	trichlorobenzene [butylated hydroxyl toluene (BHT) added, 200 ppm]
Flow rate:	1 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Sample:	polypropylene random copolymer

Table 1. Molar mass averages and polydispersity index of 2-polypropylene random copolymer sample #1 via RI

Injection Number	Retention Time (min)	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI (M_w / M_n)
1	16.532	54,380	145,630	286,074	2.678
2	16.527	54,153	145,548	289,290	2.688
3	16.548	54,027	145,195	286,331	2.687
Average	16.537	54,187	145,458	287,232	2.684
Standard Deviation	0.011	179	231	1787	0.005
CV%	0.066	0.330	0.159	0.620	0.200

Figure 2. GPC elution profile of 3 consecutive injections of 2-polypropylene random copolymer sample #2 as monitored by RI.



Column: **TSKgel GMH$_{HR}$-H (20) HT2, 20 µm, 7.8 mm ID × 30 cm x 2**
Mobile phase: trichlorobenzene [butylated hydroxyl toluene (BHT) added, 200ppm]
Flow rate: 1 mL/min
Detector: RI (EcoSEC High Temperature GPC System)
Sample: polypropylene random copolymer

Table 2. Molar mass averages and polydispersity index of 2-polypropylene random copolymer sample #2 via RI

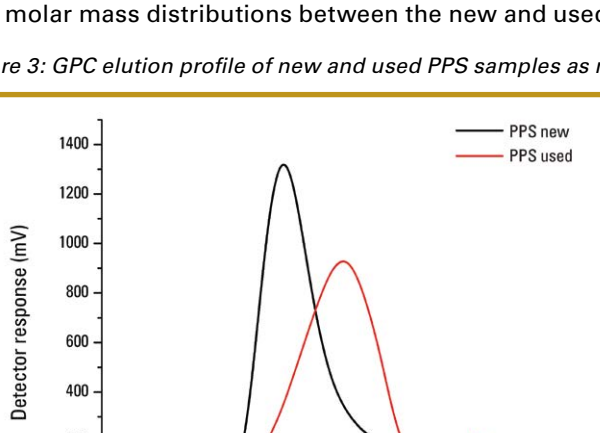
Injection Number	Retention Time (min)	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI (M_w / M_n)
1	16.532	52,396	145,040	292,193	2.768
2	16.532	52,519	145,298	292,904	2.767
3	16.533	54,427	145,729	291,369	2.677
Average	16.532	53,114	145,356	292,155	2.737
Standard Deviation	0.001	1139	348	768	0.052
CV%	0.004	2.14	0.24	0.26	1.90

Polyphenylene Sulfide

Polyphenylene Sulfide (PPS) has attracted a considerable amount of interest in the polymer industry due to its high tensile strength, good dimensional stability, flame resistance, and excellent stability in organic liquids. PPS is virtually insoluble in most organic solvents at ambient temperatures and thus can only be characterized in the solid state or by using elevated temperatures. The limited solubility of PPS makes it very difficult to determine macromolecular properties, such as molar mass and molar mass distribution, that play a vital role in the determination of mechanical, bulk and solution properties of the processing and end-use properties of a given material. Traditionally, PPS has been characterized by infrared spectrometry and thermal analysis methods. One method which can also be used to characterize PPS is high temperature GPC as PPS is soluble in 1-chloronaphthanlene (1-CN) at extremely elevated temperatures (> 200 °C). 1-CN is a difficult solvent to use for analytical experiments as the solvent ambers over time and can cause havoc for detection methods such as RI. GPC analysis of PPS in 1-CN for the determination of molar mass averages and molar mass distributions is possible using the EcoSEC High Temperature GPC System due to the unique dual flow refractive index detector.

A new and a used PPS sample were compared for failure investigation through their GPC elution profiles, Figure 3, and their polystyrene relative molar mass averages, Table 3. As seen in Figure 3, the new PPS sample eluted prior to the used PPS sample. The shorter retention time of the new PPS sample indicated that the new PPS sample was larger in polymeric size than the used PPS sample, as the elution order in GPC is that of an "inverse-sieving" technique, larger analytes sample a smaller pore volume than smaller analytes resulting in the larger analytes eluting from the GPC column prior the smaller analytes. As seen in Table 3, the new PPS sample was determined to have a higher number-, weight-, and *z*-average molar mass and greater polydispersity index, *PDI*, than the used PPS sample. The approximately 20 to 50% decrease in the molar mass averages and 25% increase in *PDI* observed between the new PPS and the used PPS is potentially enough evidence to determine that after a predetermined amount of time the end-use product(s) made with this PPS sample will begin to fail or will no longer be able to perform up to standards. The use of GPC/RI for the failure investigation of PPS allows for immediate differentiation between the new and used PPS samples based on the GPC/RI elution profile, which was then confirmed through differences in the polystyrene relative molar mass averages and molar mass distributions between the new and used PPS samples.

Figure 3: GPC elution profile of new and used PPS samples as monitored by RI



Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	1-CN
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	220 °C
Injection vol.:	300 µL
Sample:	polyphenylene sulfide

Table 3: Molar mass averages and polydispersity index of new and used PPS samples via GPC/RI

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	*PDI*[a]
PPS new	5,790	3.91×10^4	7.19×10^4	6.74
PPS used	3,176	1.62×10^4	5.54×10^4	5.10

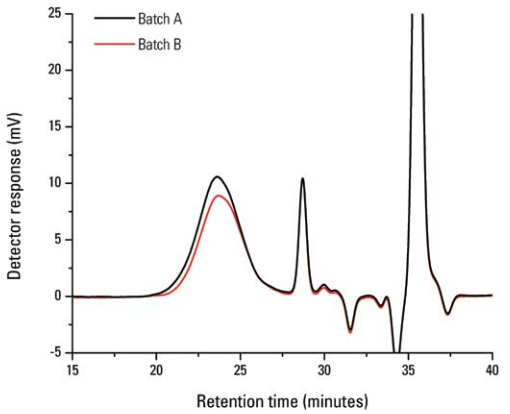
[a] $PDI = M_w/M_n$

Polyethylene

One of the most common plastics and commercially available polymers on the market is polyethylene. Polyethylene in general describes a huge family of resins obtained by the polymerization of ethylene gas. Polyethylene is available in a range of flexibilities and properties depending on the production process. Properties of polyethylene such as toughness, hardness, and clarity can be regulated by altering the molar mass averages, comonomer type, and comonomer content. Most polyethylene resins for commercial products are fabricated by controlling the molar mass average, molar mass distribution and branching characteristics. The molar mass averages and molar mass distributions of polyethylene can be determined using the EcoSEC High Temperature GPC System.

High temperature GPC experiments provide two forms of comparison between the two difference batches of polyethylene samples: GPC chromatograms and polystyrene relative molar mass averages and molar mass distributions. Figure 4 shows the GPC elution profiles as monitored by the RI detector in the EcoSEC High Temperature GPC system for the difference batches of polyethylene. Batch A extends further in the larger polymeric size, shorter retention time direction of the GPC elution profile than Batch B, an indication that the two batches differ slightly in polymeric size, as elution order in GPC is that of an "inverse-sieving" technique, as smaller analytes elute after larger analytes.

Figure 4: GPC elution profile of two batches of polyethylene as monitored by RI



Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 3**
Mobile phase:	ODCB with 0.05% BHT
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	135 °C
Injection vol.:	300 µL
Sample:	polyethylene

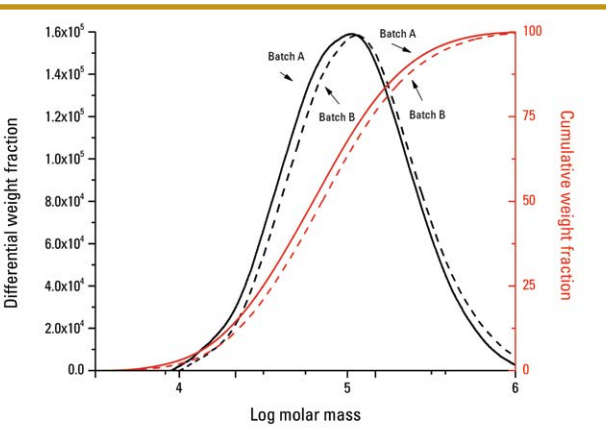
The molar mass averages and polydispersity index, *PDI*, as determined by the polystyrene RI calibration curve are given in Table 4. A comparison of the molar mass averages and molar mass distribution, Figure 5, of the two different batches of polyethylene reveals an approximately 10 to 15% difference in the polystyrene molar mass averages and distributions between the two batches. The molar mass averages and distributions of the two different batches of polyethylene obtained by high temperature GPC are different enough to distinguish the two batches from one another but may be similar enough to both create a successful commercial plastic with the same end-use properties.

Table 4: Molar mass averages and polydispersity index of two batches of polyethylene via GPC/RI

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI[a]
Batch A	4.48×10^4 ± 364[b]	1.18×10^5 ± 790	2.95×10^5 ±1,821	2.64 ± 0.06
Batch B	3.66×10^4 ± 135	1.03×10^5 ± 124	2.64×10^5 ± 2,806	2.80 ± 0.01

[a] $PDI = M_w/M_n$

Figure 5: Overlay of cumulative and differential molar mass distribution of two batches of polyethylene



Polythiophene

Conducting polymers, such as polythiophenes, have been widely investigated over the past several decades due to their potential industrial applications based on their conductivity and organic light-emitting capability. To date polythiophenes have been used in the development of electronics, energy storage batteries, photochromic devices and nonlinear optical devices. The heavy focus on synthesis of conducting polymers facilitates the need for characterization methods. Among the methods employed for the characterization of the intermediates and final conducting polymers are FT-IR, NMR, GPC, and microscopy. Some conducting polymers have limited solubility thus require the use of high temperature GPC for determination of the molar mass averages and molar mass distributions. Similar to other polymers, the molar mass averages and molar mass distributions of conducting polymers play a role in determining the end-use properties of the applications for which the polymer is used.

The molar mass averages and molar mass distributions of two conducting polymers similar to polythiophene were determined using the EcoSEC High Temperature GPC System. The polystyrene relative molar mass averages, M_n, M_w, and M_z, are given in Table 5. The variation between the molar mass averages of the two conducting polymers may be enough to change the conductivity of the polymers, thus their end-use applications. In addition to the molar mass averages, the molar mass distribution can also influence various properties of conducting polymers. The molar mass distributions of the two conducting polymers are compared in Figure 6. The molar mass distribution of polymer A is significantly larger than that of polymer B.

Information regarding the difference between the two conducting polymers can be seen by comparing their GPC elution profiles, Figure 7. The shift in GPC retention time amongst the two conducting polymers indicates a variation in polymeric size between the two conducting polymers, as elution order in GPC is that of an "inverse-sieving" technique, large analytes sample a smaller pore volume than smaller analytes resulting in larger analytes eluting from the GPC column prior to the smaller analytes. Based on the GPC elution profile, polymer A is significantly larger in polymeric size than polymer B.

Table 5: Molar mass averages and polydispersity index of two conducting polymer sammples via GPC/RI

Sample	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)
Polymer A	$2.58 \times 10^4 \pm 0.01 \times 10^4$[a]	$6.51 \times 10^4 \pm 0.02 \times 10^4$	$1.34 \times 10^5 \pm 0.03 \times 10^5$
Polymer B	$9.39 \times 10^3 \pm 0.01$[a] $\times 10^3$	$1.26 \times 10^4 \pm 0.04 \times 10^4$	$1.60 \times 10^4 \pm 0.01 \times 10^4$

[a] Standard deviation from two injections

Figure 6: Overlay of cumulative and differential molar mass distribution of two conducting polymer samples

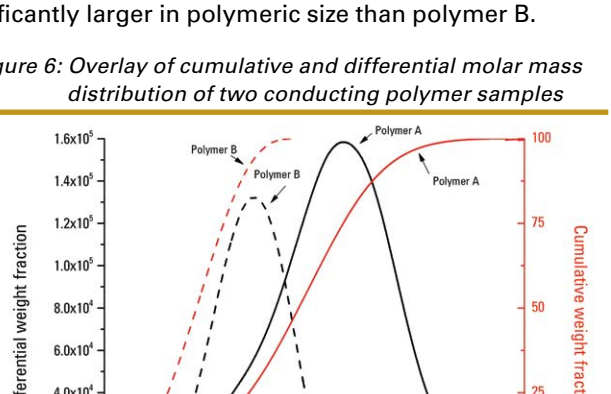
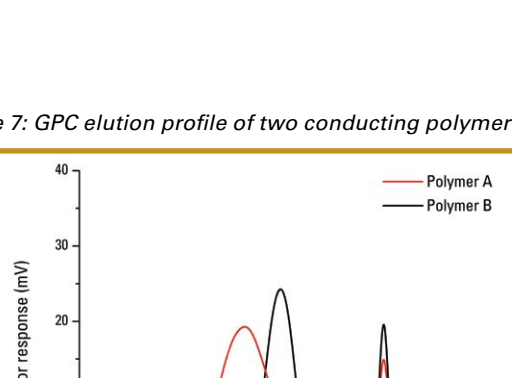


Figure 7: GPC elution profile of two conducting polymer samples as monitored by RI



Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	TCB
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	135 °C
Injection vol.:	300 µL
Sample:	polythiophene

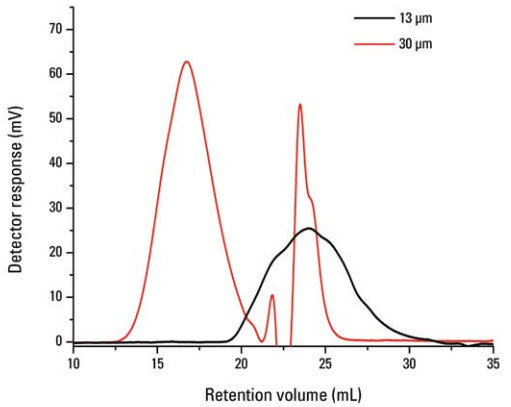
High Molar Mass Polymers

High temperature GPC is a common and important technique used for the characterization of polyolefins. GPC analysis of polyolefins can be difficult as those containing over 10% ethylene and polypropylene monomers have limited solubility due to their characteristically high strength and toughness that results from their high crystallinity. In addition to the limited solubility of most polyolefins, high molar mass polyolefins, such as ultra-high molar mass polyethylene (UHMMPE) present their own subset of issues when being analyzed by GPC. High molar mass polyethylenes are extremely long polymer chains with a molar mass greater than 2×10^6 g/mol. Polymers greater than a million in molar mass have been shown to experience on-column flow induced degradation when analyzed by GPC. To decrease the amount of degradation that occurs when UHMMPE samples are analyzed by high temperature GPC and thus obtain the most accurate molar mass averages and molar mass distributions, GPC columns packed with larger size particles with large pores are ideal.

An EcoSEC High Temperature GPC System with a dual flow refractive index detector was used in conjunction with 13 µm and 30 µm TSKgel high temperature GPC columns to determine the molar mass averages and distributions of a UHMMPE. Figure 8 show the GPC elution profiles obtained on both column sets. The shape of the GPC elution profile varies between the two column sets. The elution profile obtained using the 13 µm high temperature GPC column has a shoulder in the high molar mass region (the molar mass region most likely affected by on-column flow induced degradation) while the elution profile obtained using the 30 µm high temperature GPC column does not.

The polystyrene RI relative molar mass averages of the UHMMPE obtained using two different high temperature GPC column sets are given in Table 6. The molar mass averages obtained using the 13 µm high temperature GPC columns are significantly smaller than those obtained using the 30 µm high temperature GPC columns. The sample degradation is more prevalent in the high molar mass region of the sample as the *z*-average molar mass is two orders of magnitude greater when analysis is performed on the 30 µm high temperature GPC columns. The molar mass distribution of the UHMMPE obtained by both high temperature GPC column sets indicate an extremely polydisperse polymer. The use of 30 µm high temperature GPC columns provides a better representation of the polystyrene relative molar mass averages as the larger size particles and pores decrease the amount of degradation experienced by UHMMPE.

Figure 8: GPC elution profile of UHMMPE samples as monitored by RI with 13 µm and 30 µm TSKgel high temperature GPC columns



Column:	**TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, 7.8 mm ID × 30 cm × 2 + TSKgel G2000H$_{HR}$(20) HT2, 20 µm, 7.8 mm ID × 30 cm TSKgel GMH$_{HR}$-H (30) HT2, 30 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	TCB
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	135 °C
Injection vol.:	300 µL
Sample:	polyethylene

Table 6: Molar mass averages and polydispersity index of UHMMPE samples via RI with 13 µm and 30 µm TSKgel high temperature GPC columns

Column (particle size)	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	PDI[a]
13 µm	2.23×10^4	5.76×10^5	4.41×10^6	25.75
30 µm	9.21×10^4	7.74×10^6	2.55×10^8	84.07

[a] $PDI = M_w/M_n$

TSKgel GPC Columns

A wide variety of HPLC columns are available for the analysis of polymers in aqueous, organic and polar organic solvents.

High temperature columns for applications up to 140 °C and ultra-high temperature oclumns for applications up to 220 °C for the analysis of organic-soluble polymers are offered.

TSKgel GPC Columns

Tosoh introduced its first line of GPC columns in 1971. Ever since, Tosoh scientists have made important contributions to advances in polymer analysis by developing state-of-the-art GPC columns for the most demanding applications.

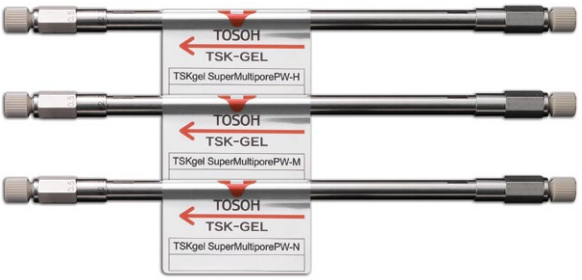
TSKgel GPC Columns for EcoSEC GPC System

Semi-micro columns are the TSKgel columns of choice for use with the EcoSEC GPC System.
They are referred to as such since their dimensions are smaller than conventional columns in terms of internal diameter as well as in length: 4.6 mm or 6 mm ID x 15 cm vs. 7.8 mm ID x 30 cm.

GPC columns for polymers soluble in organic solvents

Semi-micro columns (4.6 or 6.0 mm ID × 15 cm)

- TSKgel SuperMultiporeHZ columns
- TSKgel SuperHZ columns for ultra-low adsorption
- TSKgel SuperH columns for low adsorption



Conventional columns (7.8 mm ID × 30 cm)

- TSKgel HXL columns for ultra-low adsorption
- TSKgel HHR columns for low adsorption
- TSKgel HHR HT and HT2 columns for high temperature analysis

GPC columns for polymers soluble in polar organic solvents

Semi-micro columns (6.0 mm ID × 15 cm)

- TSKgel SuperAW columns

Conventional columns (7.8 mm ID × 30 cm)

- TSKgel Alpha columns

GPC columns for polymers soluble in aqueous solvents

Semi-micro columns (6.0 mm ID × 15 cm)

- TSKgel SuperMultiporePW columns

Conventional columns (7.5 or 7.8 mm ID × 30 or 60 cm)

- TSKgel PW columns
- TSKgel PWXL columns for higher efficiency
- TSKgel PWXL-CP columns for analysis of cationic polymers

TSKgel GPC Columns for EcoSEC High Temperature GPC System

Conventional columns (7.8 mm ID × 30 cm)

- TSKgel HHR HT and HT2 columns for high temperature analysis

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 HXL, HHR, 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 HXL 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 HXL columns are conventional GPC columns of 7.8 mm ID × 30 cm. The column line consists of eight columns with different pore sizes, TSKgel G1000HXL through TSKgel G7000HXL, and three columns with an extended linear range of the calibration curve, TSKgel GMHXL, TSKgel GMHXL-L and TSKgel MultiporeHXL-M. The 5 µm particles in the TSKgel MultiporeHXL-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 HXL 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 G1000HXL - G3000HXL, and 80 °C for the remaining columns in the TSKgel HXL column line.

- The TSKgel HHR column line consists of eight conventional GPC columns of 7.8 mm ID × 30 cm with different pore sizes, TSKgel G1000HHR through TSKgel G7000HHR, 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 GMHHR-L, GMHHR-N, GMHHR-M, to GMHHR-H. The main characteristic of these TSKgel HHR columns is a broad solvent range.

  In addition, nine TSKgel HHR mixed bed columns are available for high temperature analysis. The maximum operating temperature of the TSKgel HHR HT columns is 140 °C and the maximum operating temperature of the TSKgel HHR HT2 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 HHR 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 1. 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 2. Suggested flow rates for solvent exchange in TSKgel SuperH and HHR columns are outlined in Table 3. Table 4 lists the recommended solvents by application for TSKgel H series columns.

Table 1: Comparison of TSKgel H series columns

TSKgel series	SuperMultiporeHZ	SuperHZ	SuperH	HXL	HHR
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 2: Solvent compatibility for TSKgel H series columns

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

[1] In case of TSKgel SuperHZ and HXL, keep flow rate as mentioned below during solvent change. Solvent can be changed one way/one time only.
TSKgel HXL: 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 HHR, see Table 3 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 HXL, HHR, 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 G1000HXL 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.

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

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

Table 3: Recommended flow rates (mL/min) for TSKgel SuperH and HHR columns

Solvent	TSKgel SuperH 6.0 mm ID × 15 cm	TSKgel HHR 7.8 mm ID × 30 cm
n-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, *o*-dichlorobenzene	0.1	0.2
quinoline, hexafluoroisopropanol, 1-chloronaphthalene	0.05	0.1

Table 4: 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
o-Dichlorobenzene (ODCB)	polyethylene, polypropylene
chloroform	polycarboxylic ether, acrylic resin, epoxy resin, polystyrene
m-Cresol/Chloroform	nylon, polyester, polyamide, poly (ethylene terephthalate)
toluene	polybutadiene, polysiloxane

TSKgel HXL Size Exclusion Columns

TSKgel HXL 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 HXL column lines consists of eight columns with different pore sizes, TSKgel G1000HXL through TSKgel G7000HXL, and three columns with an extended linear range of the calibration curve, TSKgel GMHXL, TSKgel GMHXL-L and TSKgel MultiporeHXL-M.

The TSKgel HXL column line consists of the following columns:

- TSKgel G1000HXL
- TSKgel G2000HXL
- TSKgel G2500HXL
- TSKgel G3000HXL
- TSKgel G4000HXL
- TSKgel G5000HXL
- TSKgel G6000HXL
- TSKgel G7000HXL
- TSKgel GMHXL mixed bed
- TSKgel GMHXL-L mixed bed
- TSKgel MultiporeHXL-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.

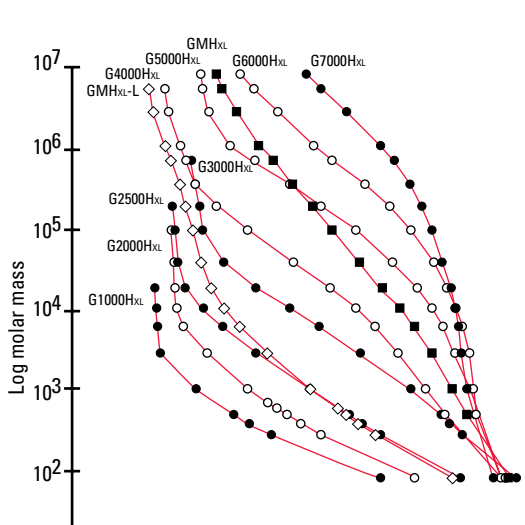
Attributes and Applications:

Product attributes of all of the TSKgel HXL columns are shown in Table 5. 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 HXL columns are shipped in THF. 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 1-2 show the calibration curves for the TSKgel HXL columns.

Table 5: Product attributes

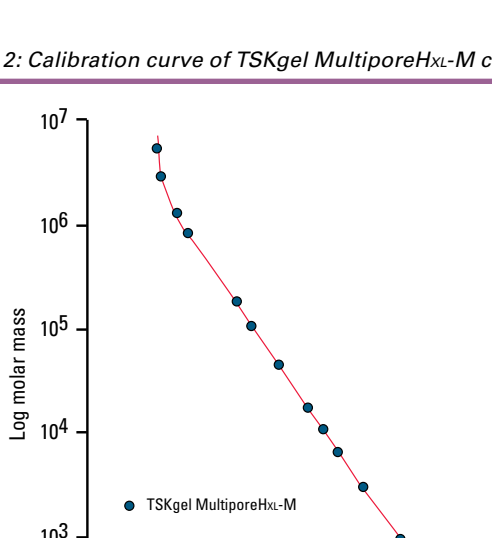
TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
G1000HXL	5 µm	1.5 nm	1,000 Da	60 °C
G2000HXL	5 µm	2 nm	1.0×10^4 Da	60 °C
G2500HXL	5 µm	3 nm	2.0×10^4 Da	60 °C
G3000HXL	5 µm	7.5 nm	6.0×10^4 Da	60 °C
G4000HXL	5 µm	20 nm	4.0×10^5 Da	80 °C
G5000HXL	9 µm	65 nm	4.0×10^6 Da	80 °C
G6000HXL	9 µm	>65 nm	4.0×10^7 Da	80 °C
G7000HXL	9 µm	>65 nm	4.0×10^8 Da	80 °C
GMHXL	9 µm	mixed pore sizes	4.0×10^8 Da	80 °C
GMHXL-L	5 µm	mixed pore sizes	4.0×10^6 Da	80 °C
MultiporeHXL-M	5 µm	broad distribution of pore size in each particle	2.0×10^6 Da	60 °C

Figure 1: Calibration curves of TSKgel HXL columns



Columns:	**TSKgel HXL 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 2: Calibration curve of TSKgel MultiporeHXL-M column

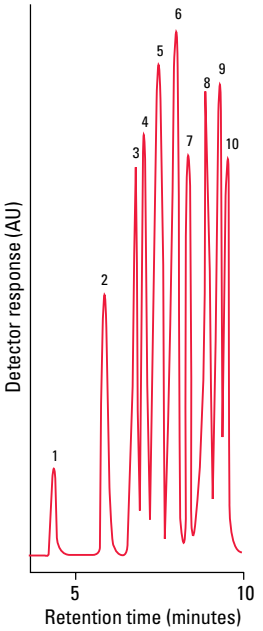


Columns:	**TSKgel MultiporeHXL-M, 5 µm, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 254 nm
Temperature:	40 °C
Sample:	polystyrene standards

Phthalate Esters

Figure 3 demonstrates the high efficiency separation on a TSKgel G1000HXL 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 3: High resolution of phthalate esters

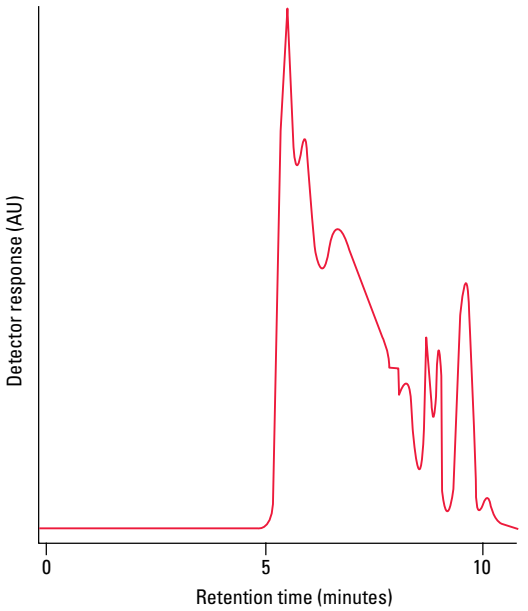


Column:	**TSKgel G1000HXL, 5 µm, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 254 nm
Samples:	1. polystyrene (1.0×10^4 Da) 2. dioctylphthalate (391 Da) 3. dibutylphthalate (278 Da) 4. diprophylphthalate (250 Da) 5. diethylphthalate (222 Da) 6. dimethylphthalate (194 Da) 7. n-propylbenzene (120 Da) 8. ethylbenzene (116 Da) 9. toluene (92 Da) 10. benzene (78 Da)

Phenol Resin

The TSKgel GMHXL-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 4. Other applications for the TSKgel GMHXL-L column include analyses of paint materials, bond and adhesive components and synthetic polymer additives.

Figure 4: Separation of phenol resin



Column:	**TSKgel GMHXL-L, 5 µm, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 254 nm
Sample:	phenol resin

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 G2000H_{HR} (20) HT
- 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 GMH_{HR}-H HT mixed bed
- TSKgel GMH_{HR}-H (S) HT mixed bed
- TSKgel GMH_{HR}-H HT2 mixed bed
- TSKgel GMH_{HR}-H (S) HT2 mixed bed
- TSKgel G2000H_{HR} (20) HT2

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), 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.

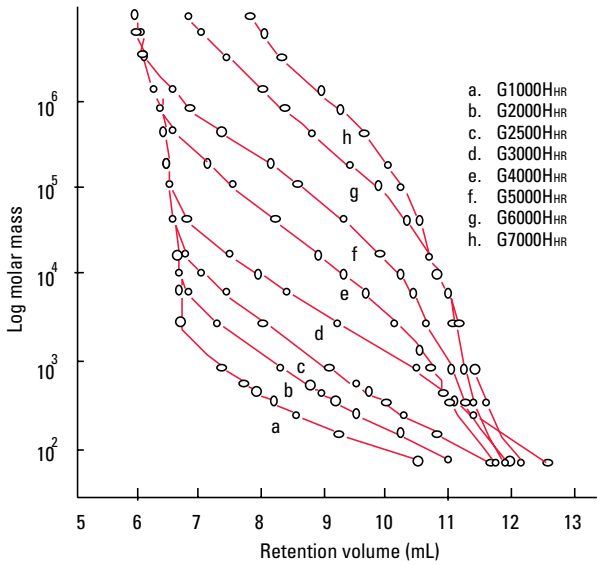
Attributes and Applications:

The product attributes for all of the TSKgel H_{HR} columns is shown in Table 6. 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. 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 5-7 show the calibration curves for the TSKgel H_{HR} columns.

Table 6: Product attributes

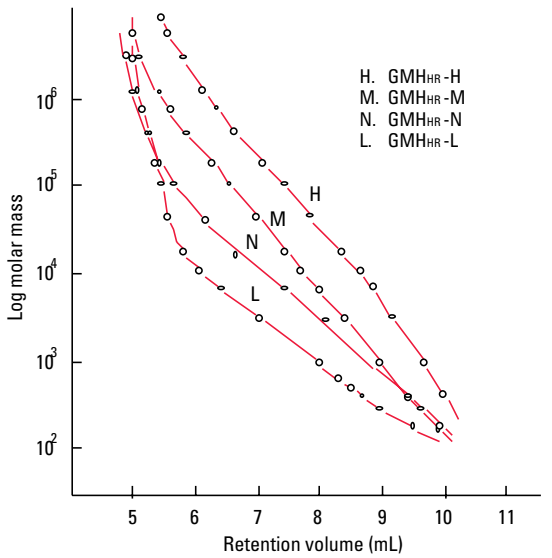
TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
G1000H_{HR}	5 µm	1.5 nm	1,000 Da	140 °C
G2000H_{HR}	5 µm	2 nm	1.0×10^4 Da	140 °C
G2500H_{HR}	5 µm	3 nm	2.0×10^4 Da	140 °C
G3000H_{HR}	5 µm	7.5 nm	6.0×10^4 Da	140 °C
G4000H_{HR}	5 µm	20 nm	4.0×10^5 Da	140 °C
G5000H_{HR}	5 µm	65 nm	4.0×10^6 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}-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
GMH_{HR}-H HT	5 µm	mixed pore sizes	4.0×10^8 Da	140 °C
GMH_{HR}-H (20) HT	20 µm	mixed pore sizes	4.0×10^8 Da	140 °C
GMH_{HR}-H (30) HT	30 µm	mixed pore sizes	4.0×10^8 Da	140 °C

Figure 5: Calibration curves of TSKgel H_{HR} columns



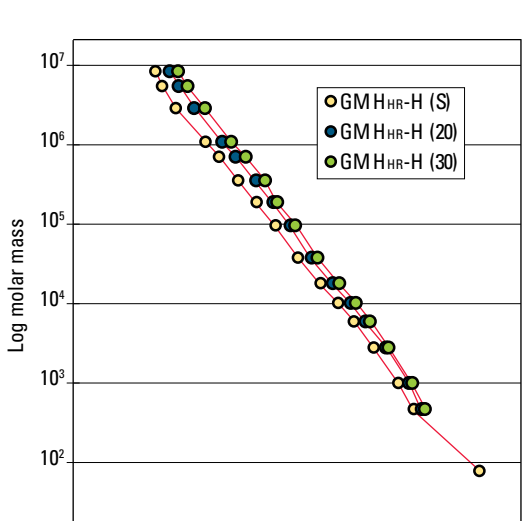
Columns:	**TSKgel H_{HR} columns, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 254 nm
Temperature:	25 °C
Samples:	polystyrene standards

Figure 6: 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 7: Calibration curves of TSKgel H_{HR}-H columns

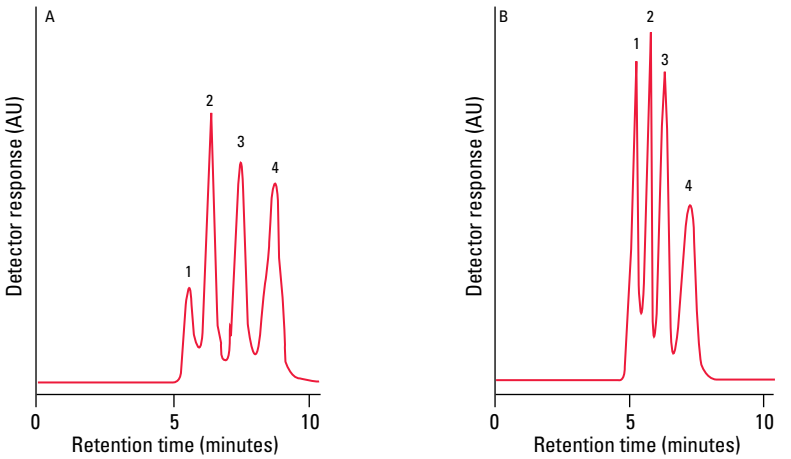


Columns:	**TSKgel GMH$_{HR}$-H (S), 13 µm, 7.8 mm ID × 30 cm** **TSKgel GMH$_{HR}$-H (20), 20 µm, 7.8 mm ID × 30 cm** **TSKgel GMH$_{HR}$-H (30), 30 µm, 7.8 mm ID × 30 cm**
Mobile phase:	THF
Flow rate:	1.0 mL/min
Detection:	UV @ 254 nm
Temperature:	25 °C
Sample:	polystyrene standards

Polymethyl methacrylate

The effect of different pore size distributions in the mixed beds of TSKgel GMH$_{HR}$-H and TSKgel GMH$_{HR}$-M is illustrated in Figure 8. The TSKgel GMH$_{HR}$-M produces sharper polymethyl methacrylate peaks in the 8.0 × 10^5 to 1.0 × 10^4 Da range.

Figure 8: Comparison of standard polymethylmethacrylate mixture



Columns:	**A. TSKgel GMH$_{HR}$-H, 5 µm, 7.8 mm ID × 30 cm**
	B. TSKgel GMH$_{HR}$-M, 5 µm, 7.8 mm ID × 30 cm
Mobile phase:	5 mmol/L sodium trifluoroacetate in HFIP
Flow rate:	1.0 mL/min
Detection:	UV @ 220 nm
Temperature:	40 °C
Sample:	standard polymethylmethacrylate
	1. 8.2 × 10^5 Da
	2. 6.7 × 10^4 Da
	3. 1.02 x 10^4 Da
	4. 1,950 Da

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 HHR 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 HHR 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.

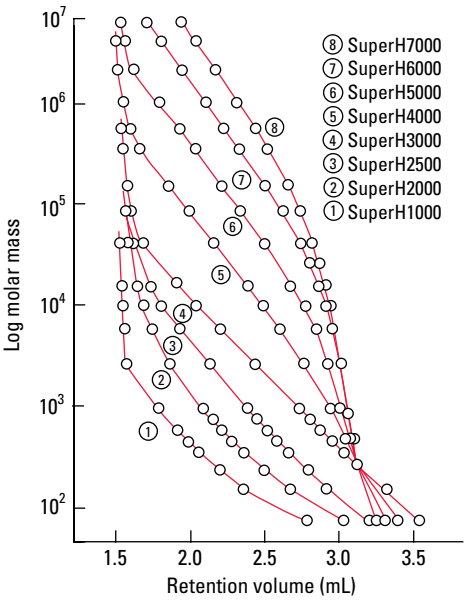
Attributes and Applications:

Table 7 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 9-10 show the calibration curves for the TSKgel SuperH columns.

Table 7: Product attributes

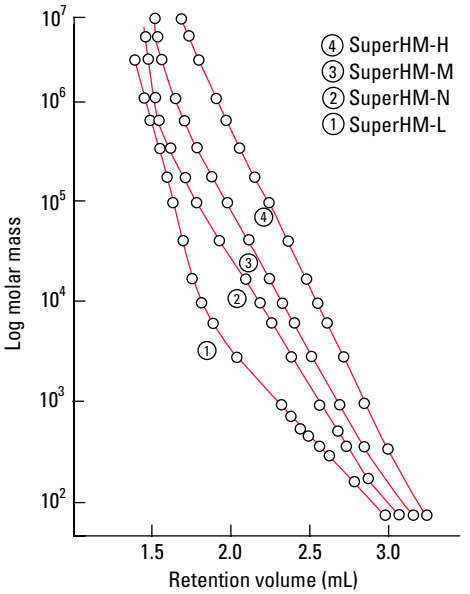
TSKgel column	Particle size (mean)	Pore size (mean)	Exclusion limit
SuperH1000	3 µm	1.5 nm	1,000 Da
SuperH2000	3 µm	2 nm	1.0×10^4 Da
SuperH2500	3 µm	3 nm	2.0×10^4 Da
SuperH3000	3 µm	7.5 nm	6.0×10^4 Da
SuperH4000	3 µm	20 nm	4.0×10^5 Da
SuperH5000	3 µm	65 nm	4.0×10^6 Da
SuperH6000	5 µm	>65 nm	4.0×10^7 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 9: Calibration curves for TSKgel SuperH columns



Columns:	**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 10: Calibration curves for TSKgel SuperH mixed bed columns



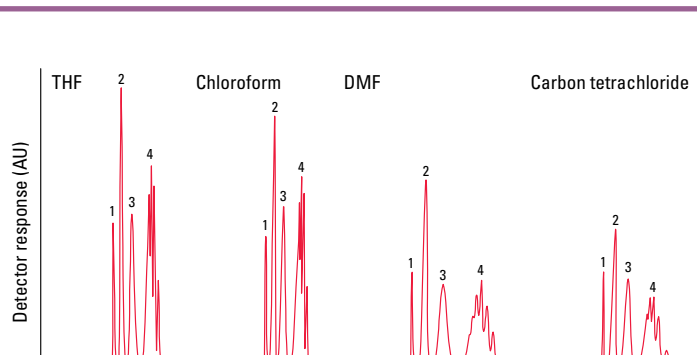
Columns:	**TSKgel SuperH columns, 6.0 mm ID × 15 cm**
Mobile phase:	THF
Flow rate:	0.6 mL/min
Detection:	UV @ 254 nm
Temperature:	25 ˚C
Sample:	polystyrene standards

Polystyrene Mixtures

Figure 11 compares chromatograms of standard polystyrene mixtures separated using a TSKgel SuperH2500 column with various organic solvents (THF, $CHCl_3$, DMF, and CCl_4) and Figure 12 compares chromatograms of standard polystyrene mixtures separated using a TSKgel SuperHM-H column with various organic solvents.

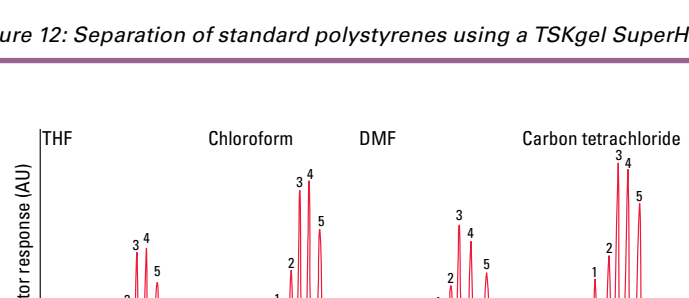
Due to the interaction between the packing material and standard polystyrene when using DMF as the mobile phase, the elution volume of standard polystyrenes is greater than it is with "good" solvents such as THF and $CHCl_3$. This effect is particularly noticeable with TSKgel SuperH2500, a column for 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 11: 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 (1.9×10^5 Da) 2) polystyrene (9.1×10^4 Da) 3) polystyrene (2,800 Da) 4) polystyrene A-500

Figure 12: Separation of standard polystyrenes using a TSKgel SuperHM-H column



Column:	**TSKgel SuperHM-H, 3 µm, 6 mm ID x 15 cm**
Mobile phase:	THF, chloroform, DMF, carbon tetrachloride
Flow rate:	0.6 mL/min
Temperature:	25 °C
Detection:	UV/VIS @ 254 nm
Samples:	1. polystyrene (2.89×10^6 Da) 2. polystyrene (4.22×10^5 Da) 3. polystyrene (1.07×10^5 Da) 4. polystyrene (1.67×10^4 Da) 5. polystyrene (2,800 Da)

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 SuperHZM-M to SuperHZM-N to SuperHZM-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 SuperHZM-H mixed bed
- TSKgel SuperHZM-M mixed bed
- TSKgel SuperHZM-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 SuperHZM-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 8 shows the product attributes of TSKgel SuperHZ columns, while Table 9 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 13-14.

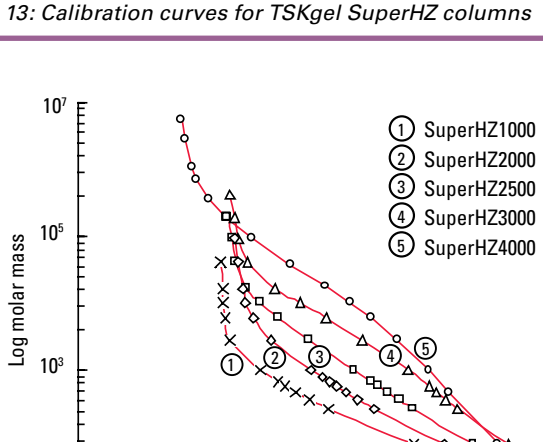
Table 8: Product attributes

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
SuperHZM-H	10 µm	mixed pore sizes	4.0×10^8 Da	80 °C
SuperHZM-M	3 µm	mixed pore sizes	4.0×10^6 Da	80 °C
SuperHZM-N	3 µm	mixed pore sizes	7.0×10^5 Da	80 °C

Table 9: Features and benefits of TSKgel SuperHZ columns

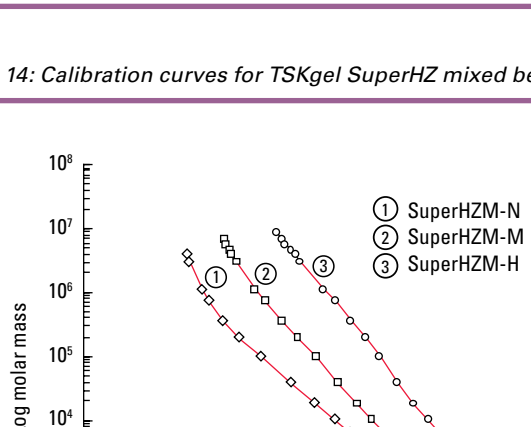
Feature	Benefit
Ultra-fine particles used in packing material	• Short measurement time is achieved. • Resolution equivalent to conventional columns (30 cm) can be obtained in ½ measurement time • Resolution does not deteriorate even under a high flow rate.
Semi-micro columns (4.6 mm ID and 6.0 mm ID)	• 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	• Shear degradation in polymers with high molar mass can be prevented
Adoption of low-adsorption packing materials	• Applicable to wide range of samples

Figure 13: Calibration curves for TSKgel SuperHZ columns



Columns: **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 14: Calibration curves for TSKgel SuperHZ mixed bed columns

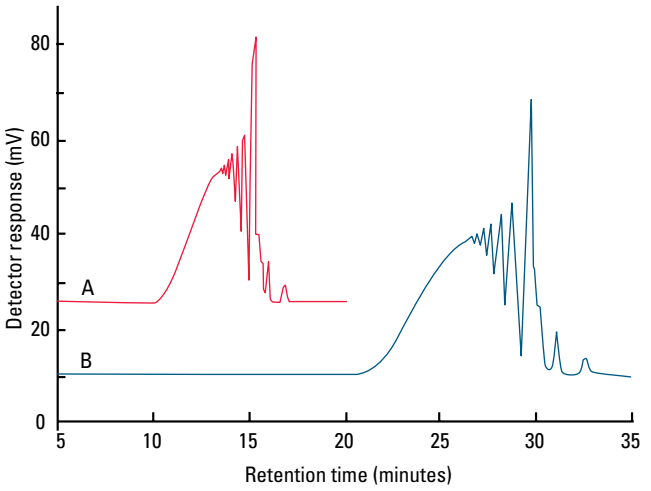


Columns: **TSKgel SuperHZ columns, 4.6 mm ID × 15 cm**
Mobile phase: THF
Flow rate: 0.35 mL/min
Temperature: 25 °C
Injection vol.: 2 µL
Samples: polystyrene standards

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 15.

Figure 15: Comparison of analysis on TSKgel SuperHZ and TSKgel HXL columns

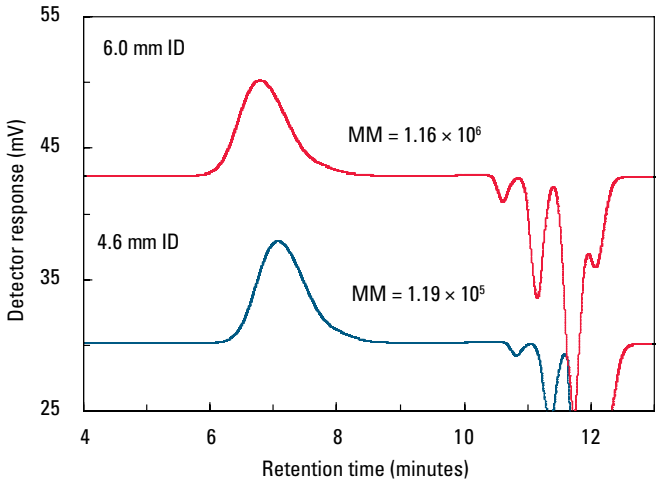


Columns:	**A. TSKgel SuperHZ columns (4000, 3000, 2500), 4.6 mm ID × 15 cm × 3** **B. TSKgel HXL columns (4000, 3000, 2500), 7.8 mm ID × 30 cm × 3**
Mobile phase:	THF
Flow rate:	A. 0.35 mL/min B. 1.0 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	A. 5 µL B. 30 µL
Sample:	phenolic resin

Polyisobutylene

The chromatogram in Figure 16 shows the analysis of polyisobutylene using two TSKgel SuperHZM-M columns in series.

Figure 16: Analysis of polyisobutylene



Column:	**TSKgel SuperHZM-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)

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 × 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

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 17, 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 17: Graphical representations illustrate the multi-pore particle synthesis technology

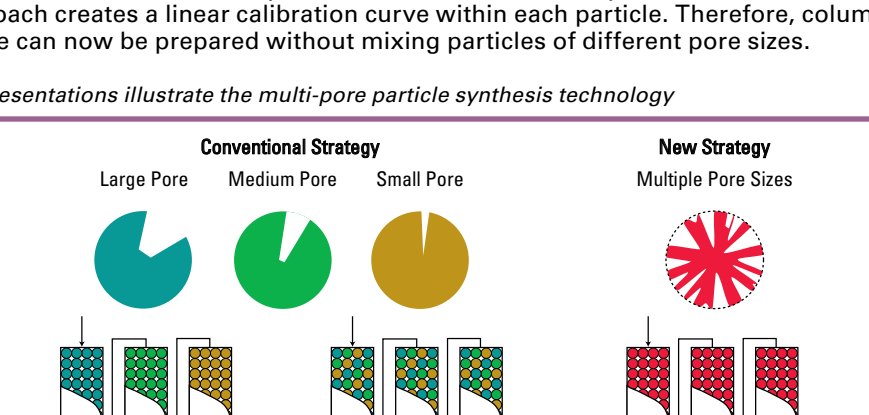
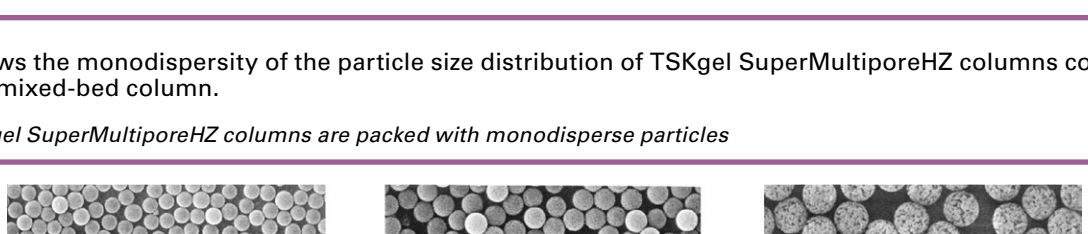


Figure 18 shows the monodispersity of the particle size distribution of TSKgel SuperMultiporeHZ columns compared to a conventional mixed-bed column.

Figure 18: TSKgel SuperMultiporeHZ columns are packed with monodisperse particles



Attributes and Applications:

Product attributes for the TSKgel SuperMultiporeHZ columns are listed in Table 10. Table 11 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 19 shows the calibration curves for the TSKgel SuperMultiporeHZ columns.

Table 10: Product attributes

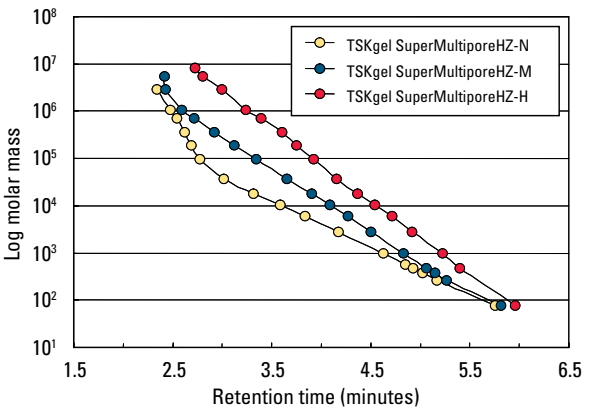
TSKgel column	SuperMultipore HZ-N	SuperMultipore HZ-M	SuperMultipore HZ-H
Base material	PS-DVB	PS-DVB	PS-DVB
Particle size	3 µm*	4 µm*	6 µm*
Pore size	8 nm	14 nm	>14 nm
Exclusion limit (PST/THF)	1.2×10^5 Da	2.0×10^6 Da	4.0×10^7 Da
Separation range	300 ~ 5.0×10^4 Da	500 ~ 1.0×10^6 Da	1,000 ~ 1.0×10^7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

* Particle size distribution is monodisperse.

Table 11: Features and benefits

Feature	Benefit
Multi-pore packing material (wide range of pores contained in single particle)	• 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)	• 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	• Reduced solvent consumption • 1/6th the consumption of conventional (30 cm) columns
Low adsorption packing material	• Can be used for a wide variety of samples

Figure 19: Calibration curves for TSKgel SuperMultiporeHZ columns

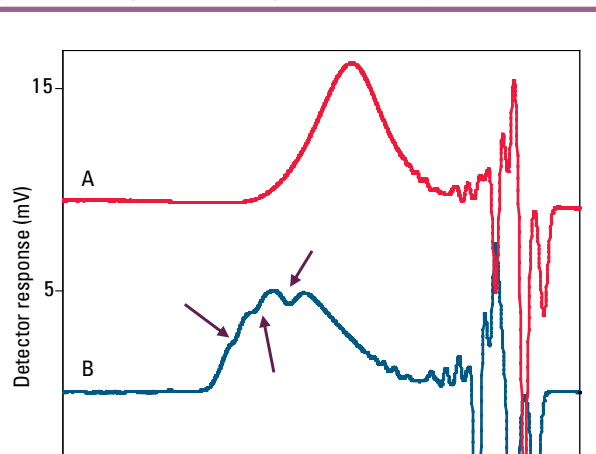


Columns: **TSKgel SuperMultiporeHZ-N, 3 µm, 4.6 mm ID × 15 cm**
TSKgel SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm
TSKgel SuperMultiporeHZ-H, 6 µm, 4.6 mm ID × 15 cm
Mobile phase: THF
Flow rate: 0.35 mL/min
Detection: UV @ 254 nm
Temperature: 25 °C
Samples: PStQuick polystyrene standards

Acrylic Resin

Figure 20 demonstrates that inflection points are no longer observed with columns packed from particles prepared by multi-pore technology.

Figure 20: Comparison for separation of acrylic resin

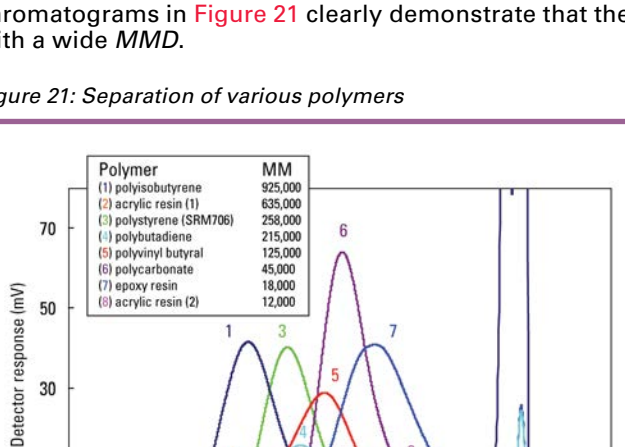


Columns:	**A. TSKgel SuperMultiporeHZ-M, 4.6 mm ID × 15 cm × 4** **B. TSKgel SuperHZ4000+3000+2500+2000, 4.6 mm ID × 15cm × 1**
Mobile phase:	THF
Detection:	RI
Temperature:	40 °C
Injection vol.:	10 µL
Samples:	acrylic resin

Various Polymers

Various polymers were analyzed on four TSKgel SuperMultiporeHZ-M columns in series. The superimposed chromatograms in Figure 21 clearly demonstrate that these new GPC columns can be utilized for the analysis of polymers with a wide *MMD*.

Figure 21: Separation of various polymers



Columns:	**SuperMultiporeHZ-M, 4 µm, 4.6 mm ID × 15 cm × 4**
Mobile phase:	THF
Flow rate:	0.35 mL/min
Detection:	RI
Temperature:	25 °C
Injection vol.:	10 µL
Sample conc.:	0.3%

TSKgel Alpha and SuperAW Size Exclusion Columns

TSKgel Alpha and SuperAW columns were developed for the GPC analysis of polymers of intermediate polarity. As in the TSKgel PW and PW$_{XL}$ columns, the particles in these TSKgel columns have a hydroxylated methacrylate polymer backbone, but they differ in that they are crosslinked to a higher degree to minimize swelling in polar organic solvents (methanol, acetonitrile, DMSO, isopropanol, THF, and HFIP). The TSKgel Alpha and SuperAW columns provide accurate molar mass determination and exhibit normal retention of polystyrene polymers in dimethyl formamide (DMF) solvent. Unlike TSKgel PW columns, which are stable to a 50% organic mixed with water at most, TSKgel SuperAW and Alpha columns are stable in a wide variety of organic solvents at concentrations up to 100%. TSKgel Alpha and SuperAW columns are offered in 5 discrete exclusion ranges and as a mixed bed column. Both column types can accommodate polymer standards up to several million Dalton molar mass.

- Use TSKgel Alpha columns when throughput is not critical, when sample mass is not limited, to collect fractions, and to obtain maximum number of plates (at the expense of analysis time). The main application area for TSKgel Alpha columns is the analysis of polymers that are soluble in polar organic solvents. Examples include cellulose derivatives, polyimide, and sodium dodecylsulfate (all in 10 mmol/L LiBr in DMF), cleansing gel in methanol, and degree of saponification of polyvinylalcohol in hexafluoroisopropanol (HFIP).

  The TSKgel Alpha Series consists of six columns with three particle sizes: 7, 10, and 13 µm. These columns span a wide MM separation range, from 100 to more than 1 x 10^6 Da, when using polyethylene oxide (PEO) as a MM standard. There is one mixed bed column within the TSKgel Alpha line, TSKgel Alpha-M, which has an extended linear calibration range and is suitable for samples with a broad MM distribution, as well as samples with unknown molar mass.

TSKgel Alpha columns include:

- TSKgel Alpha-2500
- TSKgel Alpha-3000
- TSKgel Alpha-4000
- TSKgel Alpha-5000
- TSKgel Alpha-6000
- TSKgel Alpha-M

- Use TSKgel SuperAW columns for high throughput applications, to reduce solvent consumption and to reduce solvent disposal cost. TSKgel SuperAW columns contains a similar chemistry as the TSKgel Alpha columns but offer the benefit of smaller particle sizes (4, 6, 7, and 9 µm), smaller column dimensions, and equivalent resolution. Reductions in analysis time and mobile phase consumption make TSKgel SuperAW columns ideal for high throughput applications.

  The TSKgel SuperAW column line consists of five columns and a mixed bed column. These high efficiency columns are only available in 6.0 mm ID × 15 cm dimensions.

TSKgel SuperAW columns include:

- TSKgel SuperAW2500
- TSKgel SuperAW3000
- TSKgel SuperAW4000
- TSKgel SuperAW5000
- TSKgel SuperAW6000
- TSKgel SuperAWM-H

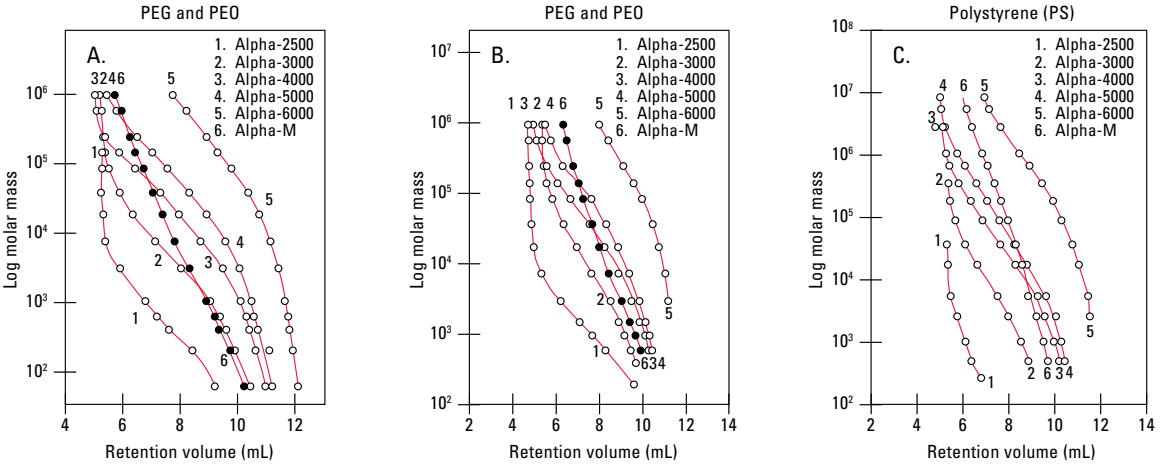
Attributes and Applications:

Product attributes of the TSKgel Alpha and SuperAW columns are shown in Table 12. These columns are for the analysis of polymers that are soluble in methanol, acetonitrile, DMSO, isopropanol, or THF and can also be used for water-soluble polymers. Figures 22-25 show the calibration curves for the TSKgel Alpha and SuperAW columns. Unlike TSKgel PW/PWXL columns, some of which are stable up to 50% organic mixed with water, TSKgel SuperAW and Alpha columns are stable in a wide variety of organic solvents at concentrations up to 100%. As shown in Figure 24, efficiency of all TSKgel SuperAW columns is maintained when changing solvents from water via acetonitrile, DMF, DMSO, THF to HFIP. Suitable solvents for TSKgel Alpha columns are shown in Figure 25.

Table 12: Product attributes

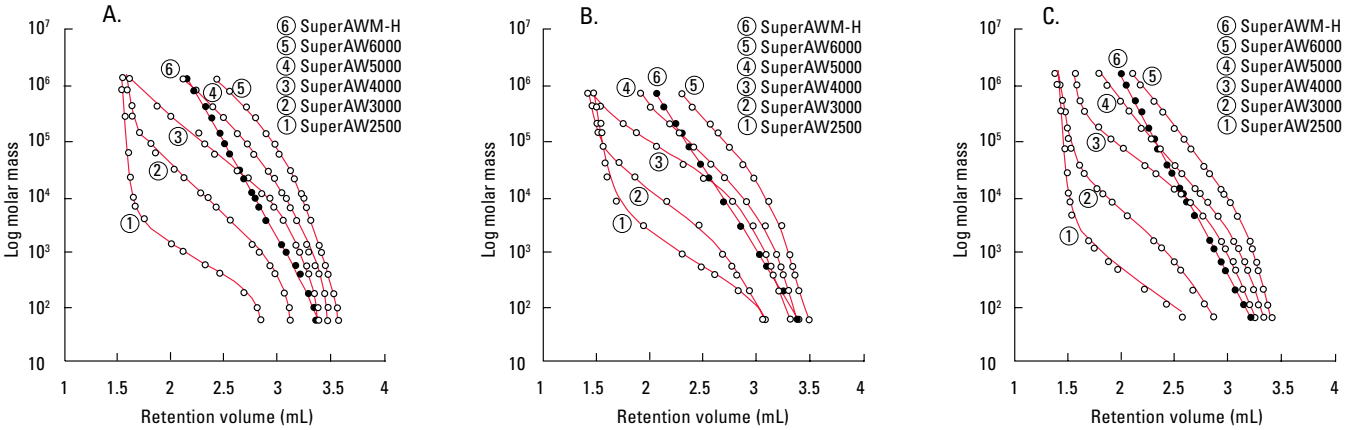
			Exclusion limit (Da) for various standards & eluents		
TSKgel column	**Particle size**	**Pore size**	**PEO in H_2O**	**PS in DMF with 10 mmol/L LiBr**	**PEG in MeOH with 10 mmol/L LiBr**
Alpha-2500	7 µm	2.5 nm	5,000	1×10^4	1×10^4
Alpha-3000	7 µm	15 nm	9×10^4	1×10^5	6×10^4
Alpha-4000	10 µm	45 nm	4×10^5	1×10^6	3×10^6
Alpha-5000	10 µm	100 nm	1×10^6	7×10^6	$>3 \times 10^5$
Alpha-6000	13 µm	>100 nm	$>1 \times 10^7$	$>1 \times 10^7$	$>3 \times 10^5$
Alpha-M	13 µm	mixed bed	$>1 \times 10^7$	$>1 \times 10^7$	$>3 \times 10^5$
SuperAW2500	4 µm	2.5 nm	5,000	1×10^4	1×10^4
SuperAW3000	4 µm	15 nm	9×10^4	1×10^5	6×10^4
SuperAW4000	6 µm	45 nm	4×10^5	1×10^6	3×10^6
SuperAW5000	7 µm	100 nm	1×10^6	7×10^6	$>3 \times 10^5$
SuperAW6000	9 µm	>100 nm	$>1 \times 10^7$	$>1 \times 10^7$	$>3 \times 10^5$
SuperAWM-H	9 µm	mixed bed	$>1 \times 10^7$	$>1 \times 10^7$	$>3 \times 10^5$

Figure 22: Polyethylene oxide, polyethylene glycol, and polystyrene calibration curves for TSKgel Alpha columns



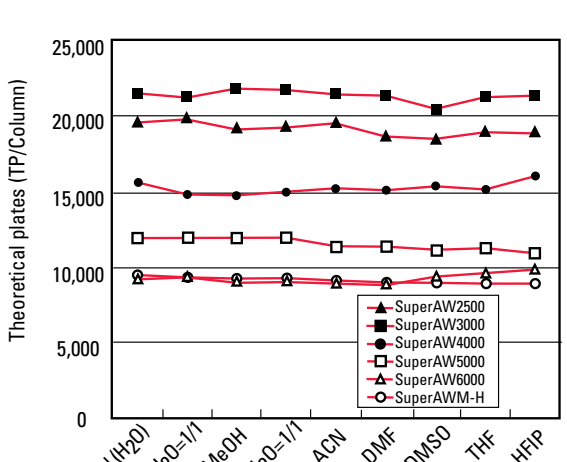
Column:	**TSKgel Alpha columns, 7.8 mm ID × 30 cm**
Mobile phase:	A. H_2O
	B. MeOH with 10 mmol/L LiBr
	C. DMF with 10 mmol/L LiBr
Flow rate:	1.0 mL/min
Detection:	RI
Temperature:	A. 25 °C B. 25 °C C. 40 °C
Samples:	A, B: polyethylene glycol (PEG) and polyethene oxide (PEO) standards
	C: polystyrene (PS) standards

Figure 23: Polyethylene oxide, polyethylene glycol, and ethylene glycol calibration curves for TSKgel SuperAW columns



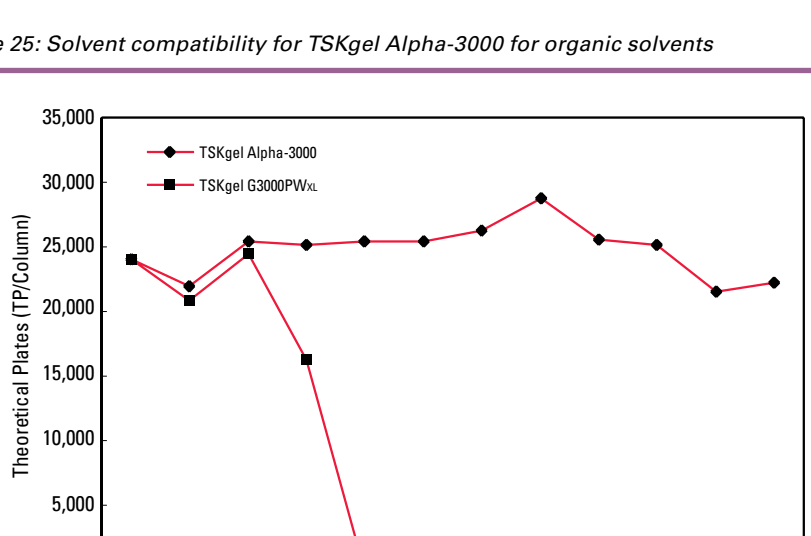
Column:	**TSKgel SuperAW columns, 6.0 mm ID × 15 cm**
Mobile phase:	A. H_2O
	B. MeOH with 10 mmol/L LiBr
	C. DMF with 10 mmol/L LiBr
Flow rate:	0.6 mL/min
Detection:	RI
Temperature:	25 °C
Samples:	polyethylene oxide (PEO), polyethylene glycol (PEG), and ethylene glycol (EG) standards

Figure 24: Column efficiency of TSKgel SuperAW columns



Column:	**TSKgel SuperAW columns, 6.0 mm ID × 15 cm**
Mobile phase:	H_2O
Flow rate:	0.6 mL/min
Detection:	RI
Temperature:	25 °C
Injection vol.:	5 µL (2.5 g/L)
Sample:	ethylene glycol

Figure 25: Solvent compatibility for TSKgel Alpha-3000 for organic solvents



Conditions of solvent change

Flow Rate:	1.0 mL/min
Temperature:	25 °C
Time for purge:	8 h

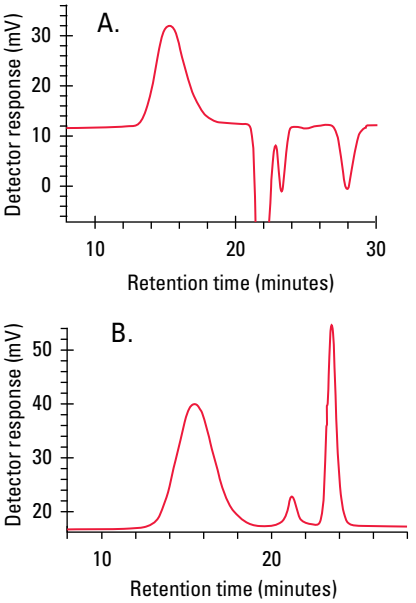
Conditions for TP measurement

Flow Rate:	1.0 mL/min
Detection:	RI
Temperature:	25 °C
Sample:	ethylene glycol

Cellulose Derivatives

The versatility of using TSKgel Alpha columns with various polar solvents is illustrated in Figure 26 for the analysis of cellulose derivatives. A TSKgel Alpha-M column was used to separate ethylcellulose with the polar solvent DMF and ethylhydroxyethyl cellulose with methanol.

Figure 26: Analysis of cellulose derivatives

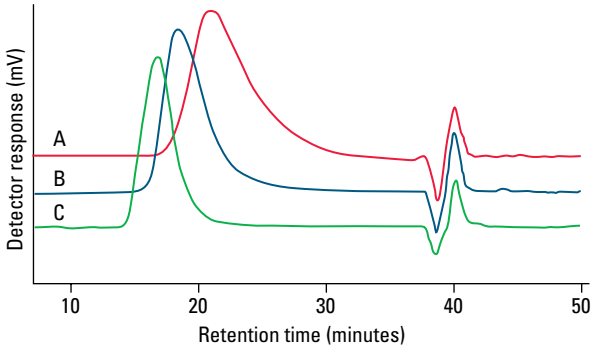


Column:	**TSKgel Alpha-M, 13 µm, 7.8 mm ID × 30 cm**
Mobile phase:	A. DMF with 10 mmol/L LiBr B. MeOH with 10 mmol/L LiBr
Flow rate:	0.5 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	50 µL
Sample:	A. ethyl cellulose, 0.1% B. ethyl hydroxyethyl cellulose, 0.1%

Polyvinylalcohol Characterization

The separation of polyvinylalcohol with different degrees of saponification is shown in Figure 27. This separation was performed with a TSKgel Alpha-5000 and a TSKgel Alpha-3000 column in series using a hexafluoroisopropanol (HFIP) mobile phase.

Figure 27: Analysis of polyvinylalcohol with different degrees of saponification

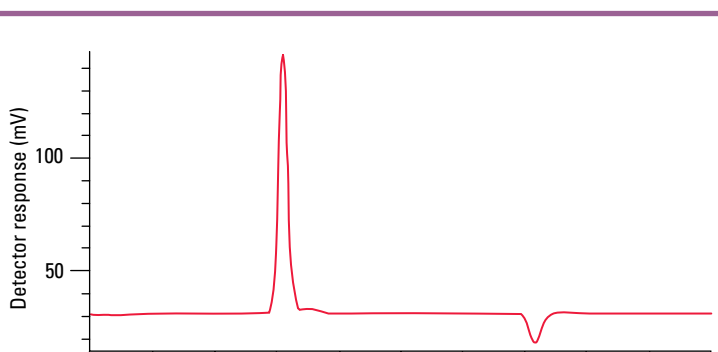


Column:	**TSKgel Alpha-5000 and Alpha-3000, 7.8 mm ID × 30 cm in series**
Mobile phase:	hexafluoroisopropanol (HFIP)
Flow rate:	0.5 mL/min
Detection:	RI
Temperature:	40 °C
Samples:	degree of saponification of polyvinyl alcohol: A. 75% B. 88% C. 100%

Glyceryl tri(2-ethylhexanoate)

Glyceryl tri(2-ethylhexanoate) is used as a plastic lubricant and as a cosmetic base. The analysis of this compound using a TSKgel Alpha-2500 column is shown in Figure 28.

Figure 28: Analysis of glyceryl tri(2-ethylhexanoate)

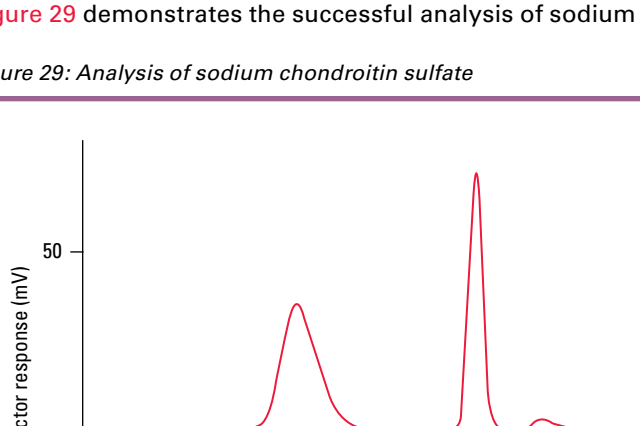


Column:	**TSKgel Alpha-2500, 7 µm, 7.8 mm ID × 30 cm**
Mobile Phase:	methanol
Flow rate:	0.5 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol:	50 µL
Sample:	glyceryl tri-2-ethylhexanoate
Sample load:	0.10%

Sodium Chondroitin Sulfate

Figure 29 demonstrates the successful analysis of sodium chondroitin sulfate on a TSKgel SuperAWM-H column.

Figure 29: Analysis of sodium chondroitin sulfate



Column:	**TSKgel SuperAWM-H, 9 µm, 6.0 mm ID × 15 cm × 2**
Mobile phase:	H_2O with 0.2 mol/L sodium nitrate
Flow rate:	0.6 mL/min
Detection:	RI
Temperature:	25 °C
Injection vol.:	5 µL (2.5 g/L)
Sample:	chondroitin sulfate

TSKgel PW Series Size Exclusion Columns

TSKgel PW and PWXL columns are recommended for analyses of water-soluble polymers and are prepared from hydrophilic polymethacrylate resin. TSKgel PWXL-CP columns are prepared from the same base resin as the TSKgel PWXL columns and were specifically developed for the analysis of water-soluble cationic polymers. TSKgel SuperMultiporePW columns are packed with particles containing a wide range of pore sizes for the analysis of water-soluble polymers with a wide molar mass range.

Stable from pH 2 to 12, TSKgel PW series columns can be used in mobile phases of water or buffer (up to 20% methanol/80% aqueous) and can tolerate temperatures up to 80 ˚C (50 ˚C for TSKgel G-DNA-PW column).

- Use TSKgel PW columns when analysis time is not critical, when sample mass is not limited, to collect fractions, or to obtain maximum number of plates (at the expense of analysis time). Particle sizes range from 12 µm for the smaller pore size columns (12.5 nm) to 17 µm for the larger pore size columns (20 nm - >100 nm).

  The TSKgel GMPW column, within the TSKgel PW column line, is a mixed bed column containing a mixture of different pore sizes that has an extended linear calibration range, suitable for samples with a broad MM distribution as well as unknown samples.

  A TSKgel G6000PW column is available in PEEK column hardware, TSKgel BioAssist G6PW, when ultra-low sample adsorption is required, such as in virus analysis.

- Use higher efficiency TSKgel PWXL columns for optimal resolution, to reduce analysis time or in sample-limited applications. TSKgel PWXL columns have smaller particle sizes than TSKgel PW columns, resulting in improved resolution.

  The TSKgel PWXL product line also offers specialty columns for analyzing carbohydrate oligomers (TSKgel G-Oligo-PW) and DNA and RNA fragments of 500-5000 base pairs (TSKgel G-DNA-PW). TSKgel GMPWXL is a mixed bed scouting column for aqueous water-soluble linear polymers. Its pore volume is accessible to polymers ranging from molar masses of 1,000 up to 8.0×10^6 Da.

- Cationic groups were introduced on the surface of the TSKgel PWXL-CP packing material to prevent adsorption of cationic polymers and allow elution under low salt conditions. These columns show high theoretical plate numbers, linear calibration curves and excellent durability. The base resin is the same as that used in the TSKgel PWXL columns.

  Three columns are available within the TSKgel PWXL-CP line, each with a different particle size, separation range and exclusion limit, allowing polymers within a wide molar mass range to be separated and characterized.

- A wide molar mass range can be analyzed with the three different TSKgel SuperMultiporePW columns, from high molar mass water-soluble polymers to oligomers. The packing material in the TSKgel SuperMultiporePW columns is more hydrophilic than that of TSKgel PWXL columns, which further reduces the chance of adsorption of hydrophilic polymers.

The range of pore sizes in which TSKgel PW and TSKgel PWXL columns are available permits a wide spectrum of water-soluble substances to be analyzed. The properties and molar mass separation ranges for all TSKgel PW series columns are summarized in Table 13.

The mechanism of SEC separation is based on the difference of apparent molecular size with no additional interaction between the column matrix and the sample molecules. In practice, however, a small number of weakly charged groups on the surface of all TSKgel PW series packings can cause changes in elution order from that of an ideal system. Fortunately, the mobile phase composition can vary greatly with TSKgel PW series columns to be compatible with a wide range of neutral, polar, anionic, and cationic samples. Table 14 lists appropriate mobile phases for GFC of major polymer types on TSKgel PW series columns.

For some nonionic, nonpolar polymers, such as polyethylene glycols, ideal size exclusion behavior can be obtained by using distilled water as the mobile phase. More polar ionic polymers may exhibit abnormal peak shapes or minor peaks near the void volume when eluted with distilled water due to ionic interactions between the sample and residual charged groups on the resin surface. To eliminate ionic interactions, a neutral salt such as sodium nitrate or sodium sulfate should be added to the aqueous eluent. Generally, a salt concentration of 0.1 mol/L to 0.5 mol/L is needed to overcome undesirable ionic interactions.

TSKgel PW resins are more hydrophobic than polysaccharide gels such as cross-linked dextran. Depending on the sample, this can lead to hydrophobic interaction as a secondary retention mechanism. The extent of hydrophobic interaction increases as the salt concentration of the eluent increases, but it can be reduced by the addition of an organic modifier such as acetonitrile. Water-soluble organic solvents are frequently used as modifiers to suppress hydrophobic interactions between the sample and the resin surface.

Table 13: Properties and separation ranges of TSKgel PW, PW$_{XL}$, PW$_{XL}$-CP, and SuperMultiporePW columns

			Molar mass of samples (Da)
TSKgel column	**Particle size**	**Pore size**	**Polyethylene glycols & oxides**
SuperMultiporePW-N	4 µm	20 nm	300 – 5 × 10^4
SuperMultiporePW-M	5 µm	100 nm	500 – 1 × 10^6
SuperMultiporePW-H	8 µm	>100 nm	1,000 – 1 × 10^7
G2000PW	12 µm	12.5 nm	<3,000
G2500PW	12 µm and 17 µm	12.5 nm	<3,000
G3000PW	12 µm and 17 µm	20 nm	<5 × 10^4
G4000PW	17 µm	50 nm	<3 × 10^5
G5000PW	17 µm	100 nm	<1 × 10^6
G6000PW BioAssist G6PW	17 µm	>100 nm	<8 × 10^6
GMPW	17 µm	mixed pore sizes	1,000 – 8 × 10^6
G2500PW$_{XL}$	7 µm	12.5 nm	<3,000
G3000PW$_{XL}$	7 µm	20 nm	<5 × 10^4
G4000PW$_{XL}$	10 µm	50 nm	<3 × 10^5
G5000PW$_{XL}$	10 µm	100 nm	<1 × 10^6
G6000PW$_{XL}$	13 µm	>100 nm	<8 × 10^6
G-DNA-PW	10 µm	>100 nm	<8 × 10^6
GMPW$_{XL}$	13 µm	mixed pore sizes	1,000 – 8 × 10^6
SuperOligoPW	3 µm	12.5 nm	<3,000
G-Oligo-PW	7 µm	12.5 nm	<3,000
G3000PW$_{XL}$-CP	7 µm	20 nm	200 – 5 × 10^4
G5000PW$_{XL}$-CP	10 µm	100 nm	400 – 5 × 10^5
G6000PW$_{XL}$-CP	13 µm	>100 nm	1,000 – 1 × 10^7
Columns: TSKgel PW columns, 7.5 mm ID × 60 cm TSKgel PW$_{XL}$, G-Oligo-PW and G-DNA-PW columns, 7.8 mm ID × 30 cm TSKgel SuperMultiporePW and SuperOligoPW columns, 6.0 mm ID × 15 cm Mobile phase: polyethylene glycols and oxides (PEOs): distilled water Flow rate: 1.0 mL/min, except for TSKgel SuperMultiporePW and SuperOligoPW columns: 0.6 mL/min			

Modifiers are also used for optimizing the elution of both charged and neutral hydrophobic polymers. Typical examples for a variety of sample types are given in Table 14. All TSKgel PW series packings are compatible with 20% aqueous solutions of methanol, ethanol, propanol, acetonitrile, dimethyl formamide, dimethyl sulfoxide, formic acid, and acetic acid. In addition, these columns can be operated in 50% aqueous acetone.

Table 14: Recommended mobile phases for GFC of water-soluble polymers on TSKgel PW, PW$_{XL}$, PW$_{XL}$-CP, and SuperMultiporePW columns

Type of polymer	Typical sample	Suitable mobile phase
Nonionic hydrophilic	polyethylene glycol	Distilled water
	soluble starch, methyl cellulose, pullulan	0.01 mol/L NaOH
	dextran, hydroxyethyl cellulose	20% DMSO (dimethyl sulfoxide)
	polyvinyl alcohol, polyacrylamide	Buffer or salt solution (e.g. 0.1-0.5 mol/L $NaNO_3$)
Nonionic hydrophobic	polyvinylpyrrolidone	Buffer or salt solution with organic solvent (e.g. 20% CH_3CN in 0.1 mol/L $NaNO_3$)
Anionic hydrophilic	sodium chondroitin sulfate, sodium alginate, carboxymethyl cellulose, sodium polyacrylate, sodium hyaluronate	Buffer or salt solution (e.g. 0.1 mol/L $NaNO_3$)
Anionic hydrophobic	sulfonated lignin sodium salt, sodium polystyrenesulfonate	Buffer or salt solution with organic solvent (e.g. 20% CH_3CN in 0.1 mol/L $NaNO_3$)
Cationic hydrophilic	glycol chitosan, DEAE-dextran, poly(ethylene imine), poly(trimethylaminoethyl methacrylate) iodide salt	0.5 mol/L acetic acid with 0.3 mol/L Na_2SO_4 or 0.8 mol/L $NaNO_3$
Cationic hydrophobic	poly(4-vinylbenzyltrimethylammonium chloride), poly(N-methyl-2-vinylpyridinium) iodide salt	0.5 mol/L acetic acid with 0.3 mol/L Na_2SO_4
Amphoteric hydrophilic	peptides, proteins, poly- and oligosaccharides, DNA, RNA	Buffer or salt solution (e.g. 0.1 mol/L $NaNO_3$)
Amphoteric hydrophobic	blue dextran, collagen, gelatin, hydrophobic proteins, hydrophobic peptides	Buffer or salt solution with organic solvent (e.g. 20% CH_3CN in 0.1 mol/L $NaNO_3$ or 35-45% CH_3CN in 0.1% TFA)

TSKgel PW Size Exclusion Columns

TSKgel PW columns are composed of spherical, hydrophilic polymethacrylate beads. Particle sizes range from 12 µm for the smaller pore size columns to 17 µm for the larger pore size columns. Stable from pH 2 to 12, TSKgel PW columns can be used in mobile phases of water or buffer (up to 20% methanol/80% aqueous) and can tolerate temperatures up to 80 ˚C.

The TSKgel PW column line consists of the following columns:

- TSKgel G2000PW
- TSKgel G2500PW
- TSKgel G3000PW
- TSKgel GMPW
- TSKgel G4000PW
- TSKgel G5000PW
- TSKgel G6000PW

The mixed bed column, TSKgel GMPW, has an extended linear calibration range, suitable for samples with a broad MM distribution, as well as for unknown samples. The pore volume can be accessed by polymers ranging in molar mass from 1,000 to 8.0 × 10^6 Da. By quickly categorizing the MM profile of an unknown sample, the column enables a fast selection of the best TSKgel PW column for routine analysis.

Attributes and Applications

Product attributes of all eight TSKgel PW columns are shown in Table 15. All TSKgel PW columns have a base material of hydroxylated polymethacrylate, can be used in a maximum of 20% organic, and are shipped in water. The main application area for TSKgel PW columns is the analysis of water-soluble polymers, such as celluloses, acrylamides, glycols, dextrans, polyvinylalcohol, and oligosaccharides. TSKgel G2000PW, the larger particle size equivalent of TSKgel G-Oligo-PW, is most suitable for semi-preparative and preparative isolation of oligosaccharides. Representative application examples for the PW columns are illustrated in Table 16. The calibration curve for polyethylene glycol and oxides for the TSKgel PW columns is shown in Figure 30.

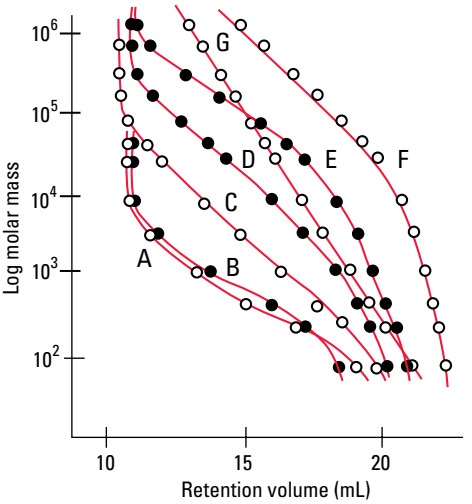
Table 15: Product attributes

TSKgel column	Particle size (mean)	Pore size (mean)	Calibration range
G2000PW	12 µm	12.5 nm	Up to 3,000 Da (polyethylene glycols and oxides)
G2500PW	12 µm and 17 µm	12.5 nm	Up to 3,000 Da (polyethylene glycols and oxides)
G3000PW	12 µm and 17 µm	20 nm	Up to 5.0 × 10^4 Da (polyethylene glycols and oxides)
G4000PW	17 µm	50 nm	Up to 3.0 × 10^5 Da (polyethylene glycols and oxides)
G5000PW	17 µm	100 nm	Up to 1.0 × 10^6 Da (polyethylene glycols and oxides)
G6000PW	17 µm	>100 nm	Up to 8.0 × 10^6 Da (polyethylene glycols and oxides)
GMPW	17 µm	mixed pore sizes	1,000 - 8.0 × 10^6 Da (polyethylene glycols and oxides)

Table 16: Representative application examples for TSKgel PW columns

Classification	Examples
1. Synthetic polymers • Nonionic • Cationic • Anionic	• PEG, polyglycerin,v polyacrylamide • Polyethyleneimine, polyvinylpyrolidine • Poly (sodium acrylate), Poly (sodium styrene sulfonate)
2. Polysaccharides and derivatives	• Standard dextran, clinical dextran, pullulan, inulin, heparin, chitosan • Carboxymethylcellulose
3. Very large biopolymers • Polynucleotides • Viruses • Proteins	• DNA fragments • TMV, SBMV, TBSV • Lipoprotein (VLDL, LDL), apoferritin, gelatin, sea worm chlorocruorin
4. Small molecules • Oligomers • Others	• oligosaccharides (dextran hydrolysate, cyclodoxtrin • hydrolysate), cyclodextrins • oligopeptides • oligonucleotides

Figure 30: Polyethylene glycol and oxide calibration curves for TSKgel PW columns

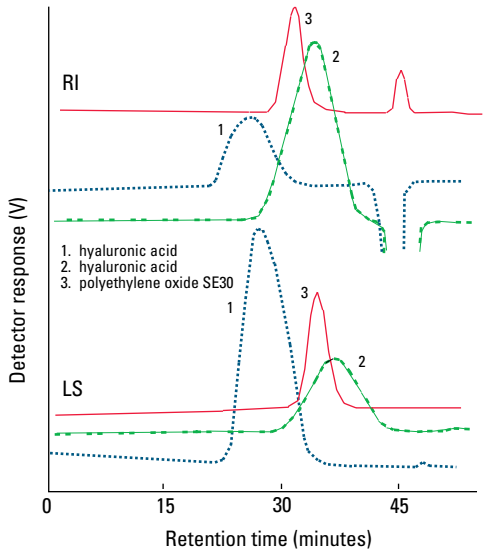


Column:	**A. G2000PW, B. G2500PW, C. G3000PW, D. G4000PW, E. G5000PW, F. G6000PW, G. GMPW, all 7.5 mm ID × 60 cm**
Mobile phase:	distilled H_2O
Flow rate:	1.0 mL/min
Detection:	RI

Oligosaccharides

TSKgel PW columns are recommended for polysaccharide analysis due to their ability to separate a wide molar mass distribution. An effective separation of the anionic hydrophilic glucosaminoglycan, hyaluronic acid, is shown in Figure 31 on a TSKgel G6000PW and TSKgel G4000PW column in series with a 0.2 mol/L sodium chloride mobile phase. To obtain shorter analysis time and similar resolution, we recommend using TSKgel G3000PWXL and G4000PWXL columns in series.

Figure 31: Analysis of polysaccharides

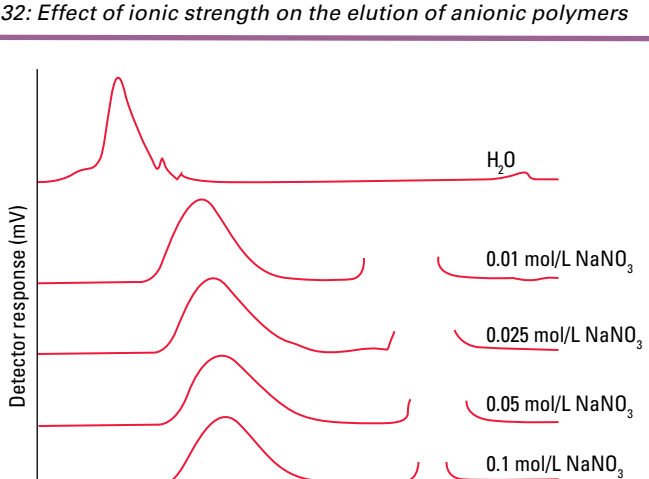


Columns:	**TSKgel G6000PW + G4000PW, two 7.5 mm ID × 60 cm columns in series**
Mobile phase:	H_2O with 0.2 mol/L NaCl
Flow rate:	0.9 mL/min
Temperature:	40 °C
Sample:	hyaluronic acid, polyethylene oxide

Polymers

Sodium polyacrylate, an anionic polymer, is effectively separated on two TSKgel GMPW columns in Figure 32. The addition of 0.01 mol/L $NaNO_3$ results in normal elution and peak shape overcoming the ionic repulsion between the anionic sample and the resin.

Figure 32: Effect of ionic strength on the elution of anionic polymers



Column:	**TSKgel GMPW, 17 µm, 7.5 mm ID × 60 cm × 2**
Mobile phase:	H_2O with 0.01 mol/L, 0.025 mol/L, 0.05 mol/L or 0.1 mol/L $NaNO_3$
Flow rate:	0.5 mL/min
Detection:	RI
Sample:	0.5 mL of 0.05-0.1% of the sodium salt of polyacrylic acid, an anionic polymer

TSKgel PWXL Size Exclusion Columns

TSKgel PWXL columns are composed of spherical, hydrophilic polymethacrylate beads. The smaller particle size of TSKgel PWXL columns provide 1.7x higher resolution than their TSKgel PW columns counterpart, making TSKgel PWXL columns more suitable for analytical purposes. Four specialty columns are included in the TSKgel PWXL column line.

The TSKgel G-DNA-PW column is designed for the separation of large polynucleotides such as DNA and RNA fragments of 500 - 5,000 base pairs. This column is a smaller particle size version of the TSKgel G6000PWXL column. The TSKgel G-Oligo-PW column is designed for high resolution separations of aqueous nonionic and cationic oligomers, and oligosaccharides such as hydrolyzed cyclodextrins. Because of the presence of cationic groups on the gel matrix, this column is not suitable for separating anionic polymers. The TSKgel G-Oligo-PW column has a PEG and PEO calibration curve identical to that of the TSKgel G2500PWXL column. The mixed-mode column, TSKgel GMPWXL, has an extended linear calibration range, suitable for samples with a broad MM distribution and unknowns.

The TSKgel SuperOligoPW column is designed for the determination of molar mass of aqueous oligomers, particularly oligosaccharides, and low molar mass aqueous polymers. The combination of the decreased particle size and semi-micro dimensions of the TSKgel SuperOligoPW column enables high speed separation with high resolution and lowered solvent consumption. Since the packing material in the TSKgel SuperOligoPW columns is more hydrophilic compared with TSKgel G-Oligo-PW columns, an even wider range of water-soluble polymers can be analyzed without the need to add organic solvent to the eluent.

The following TSKgel PWXL columns are offered:

- TSKgel G2500PWXL
- TSKgel G3000PWXL
- TSKgel G4000PWXL
- TSKgel G5000PWXL
- TSKgel G6000PWXL
- TSKgel G-DNA-PW
- TSKgel GMPWXL
- TSKgel G-Oligo-PW
- TSKgel SuperOligoPW

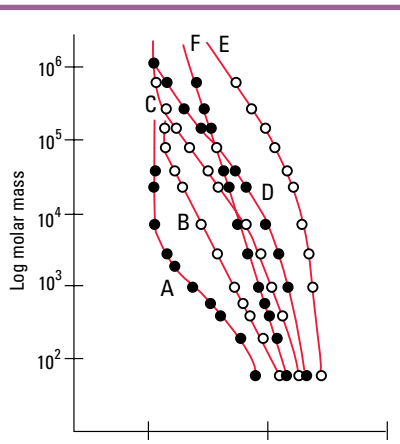
Attributes and Applications

The main application area for TSKgel PWXL columns is the analysis of water-soluble polymers, such as celluloses, acrylamides, glycols, dextrans, polyvinylalcohol, and oligosaccharides. Because of the presence of cationic groups on the base bead of TSKgel G2500PWXL, this column is not suited for separating anionic polymers. Product attributes of all of the TSKgel PWXL columns are shown in Table 17. All TSKgel PWXL columns have a base material of hydroxylated polymethacrylate, can be used in a maximum of 20% organic and are shipped in water. Figures 33-37 show the calibration curves for all of the TSKgel PWXL columns.

Table 17: Product attributes

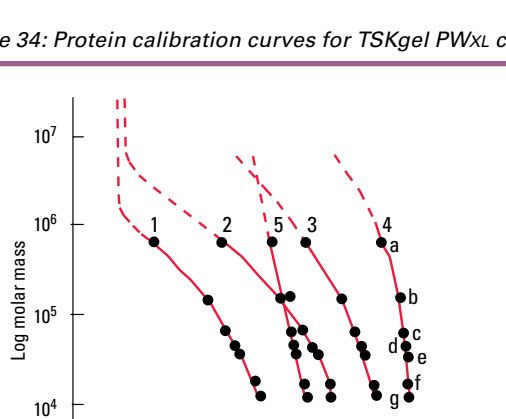
TSKgel column	Particle size (mean)	Pore size (mean)	Calibration range
G2500PWXL	7 µm	12.5 nm	<3,000 Da (polyethylene glycols and oxides)
G3000PWXL	7 µm	20 nm	<4.0×10^4 Da (polyethylene glycols and oxides)
G4000PWXL	10 µm	50 nm	2,000 - 3.0×10^5 Da (polyethylene glycols and oxides)
G5000PWXL	10 µm	100 nm	4,000 - 8.0×10^5 Da (polyethylene glycols and oxides)
G6000PWXL	13 µm	>100 nm	4.0×10^4 - 8.0×10^6 Da (polyethylene glycols and oxides)
G-DNA-PW	10 µm	>100 nm	4.0×10^4 - 8.0×10^6 Da (polyethylene glycols and oxides)
GMPWXL	13 µm	mixed pore sizes	1,000 - 8.0×10^6 Da (polyethylene glycols and oxides)
G-Oligo-PW	7 µm	12.5 nm	Up to 3,000 Da (polyethylene glycols and oxides)
SuperOligoPW	3 µm	12.5 nm	<3,000 Da (PEO,PEG/H_2O)

Figure 33: Polyethylene glycol and oxide calibration curves for TSKgel PWXL columns



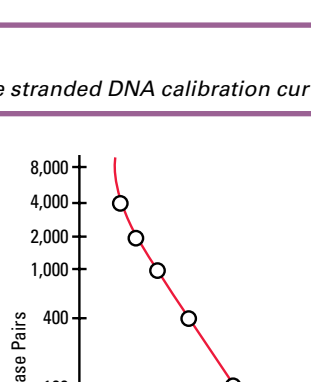
Column:	**A. G2500PWXL, B. G3000PWXL, C. G4000PWXL, D. G5000PWXL, E. G6000PWXL, F. GMPWXL, all 7.8 mm ID × 30 cm**
Mobile phase:	distilled H_2O
Flow rate:	1.0 mL/min
Detection:	RI

Figure 34: Protein calibration curves for TSKgel PWXL columns



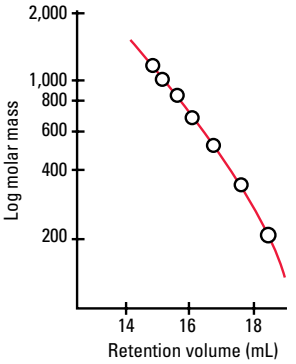
Column:	**1. TSKgel G3000PWXL** **2. TSKgel G4000PWXL** **3. TSKgel G5000PWXL** **4. TSKgel G6000PWXL** **5. TSKgel GMPWXL** **all 7.8 mm ID × 30 cm**
Mobile phase:	0.2 mol/L phosphate buffer, pH 6.8
Flow rate:	1.0 mL/min
Detection:	UV @ 280 nm
Sample:	a. thyroglobulin (6.6×10^5 Da) b. γ-globulin (1.5×10^5 Da) c. albumin (6.7×10^4 Da) d. ovalbumin (4.3×10^4 Da) e. β-lactoglobulin (3.6×10^4 Da) f. myoglobin (1.69×10^4 Da) g. cytochrome C (1.24×10^4 Da)

Figure 35: Double stranded DNA calibration curves for TSKgel G-DNA-PW column



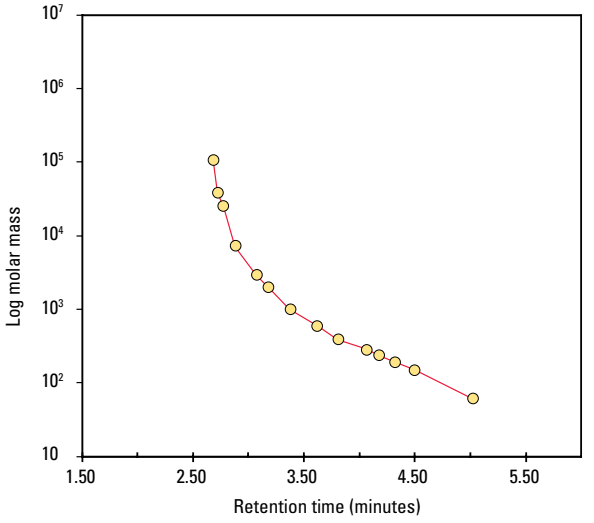
Column:	**TSKgel G-DNA-PW, 10 µm, 7.8 mm ID × 30 cm × 4**
Mobile phase:	H_2O with 0.3 mol/L NaCl in 0.1 mol/LTris-HCl, pH 7.5, + 1 mmol/L EDTA
Flow rate:	0.15 mL/min
Detection:	UV @ 260 nm
Sample:	*Eco* RI and *Bst* NI-cleaved pBR322 DNA, void volume determined with λ-DNA

Figure 36: Oligosaccharide calibration curve for TSKgel G-Oligo-PW column



Column:	**TSKgel G-Oligo-PW, 7 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	distilled H_2O
Flow rate:	1.0 mL/min
Detection:	UV @ 260 nm
Sample:	hydrolyzed β-cyclodextrin

Figure 37: Polyethylene glycol, oxide and ethylene glycol calibration curve for TSKgel SuperOligoPW column

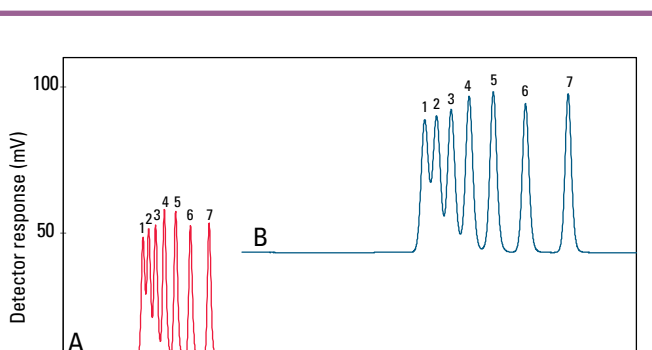


Columns:	**TSKgel SuperOligoPW, 3 µm, 6.0 mm ID × 15 cm**
Mobile phase:	H_2O
Flow rate:	0.60 mL/min
Detection:	RI
Temperature:	25 °C
Samples:	PEO, PEG and ethylene glycol

Oligosaccharides

Figure 38 demonstrates the high speed analysis of maltose oligomers using a TSKgel SuperOligoPW column compared to a TSKgel G-Oligo-PW column. The faster analysis time is due to the semi-micro dimensions (6.0 mm ID × 15 cm) and the small particle size (3 µm) of the TSKgel SuperOligoPW column compared to the 7.8 mm ID × 30 cm size and 7 µm particle size of the TSKgel G-Oligo-PW column.

Figure 38: Analysis of maltose oligomers

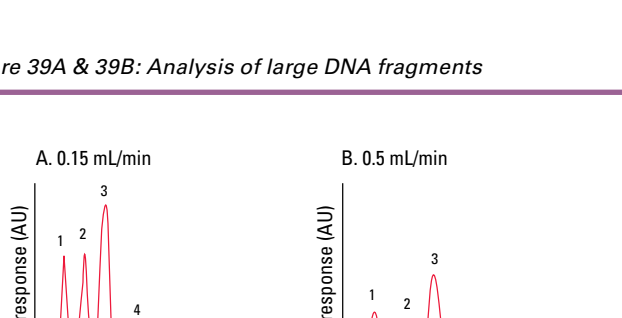


Columns:	**A. TSKgel SuperOligoPW, 3 µm, 6.0 mm ID × 15 cm × 4** **B. TSKgel G-Oligo-PW, 7 µm, 7.8 mm ID × 30 cm × 4**
Mobile phase:	H_2O
Flow rate:	A: 0.6 mL/min B: 1.0 mL/min
Detection:	RI
Temperature:	40 °C
Injection vol.:	A: 10 µL B: 50 µL
Samples:	1. maltoheptose 2. maltohexose 3. maltopentose 4. maltotetraose 5. maltotriose 6. maltose 7. glucose

Large DNA fragments

For the separation of large DNA fragments greater than 1,000 base pairs, a four column system is typically required. Baseline resolution of DNA fragments up to 7,000 base pairs can be achieved, provided there is a two-fold difference in the chain length of the fragments. Figure 39A shows the elution of double stranded DNA fragments, obtained from pBR322 DNA cleaved by both EcoRI and BstNI, on four TSKgel G-DNA-PW columns in series. The eluted peaks were collected and subjected to polyacrylamide gel electrophoresis, which showed almost complete separation of the 1060, 1857, and 4362 base pair fragments. Although lower flow rates typically yield better separations of most fragments, the resolution of the 1857 and 4362 base pair fragments was slightly greater at the higher flow rate, as shown in Figure 39B.

Figure 39A & 39B: Analysis of large DNA fragments



Column:	**TSKgel G-DNA-PW, 10 µm, 7.8 mm ID × 30 cm × 4**
Mobile phase:	H_2O with 0.3 mol/L NaCl in 0.1 mol/L Tris-HCl, pH 7.5, + 1 mmol/L EDTA
Flow Rate:	A. 0.15 mL/min B. 0.5 mL/min
Detection:	UV @ 260 nm
Samples:	60 µL of Eco RI and Bst NI - cleaved pBR322 DNA, base pairs: 1. 4362 2. 1857 3. 1060 & 928 4. 383 5. 121 6. 13

TSKgel PWXL-CP Size Exclusion Columns

TSKgel PWXL-CP columns were specifically developed for the analysis of water-soluble cationic polymers. Composed of polymethacrylate beads, cationic groups are introduced on the surface of the TSKgel PWXL-CP packing material to prevent adsorption of cationic polymers and allow elution under low salt conditions. These columns show high theoretical plate numbers, linear calibration curves, and high durability because the base resin is the same as that used in the TSKgel PWXL columns.

Three columns are available within the TSKgel PWXL-CP series, each with a different particle size, separation range, and exclusion limit, allowing polymers within a wide molar mass range to be separated and characterized.

- TSKgel G3000PWXL-CP
- TSKgel G5000PWXL-CP
- TSKgel G6000PWXL-CP

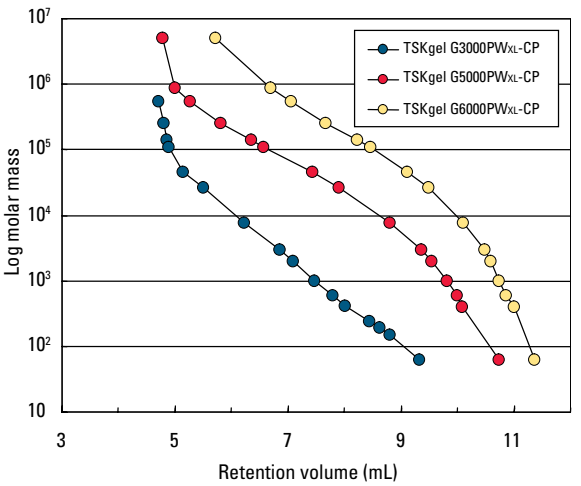
Attributes and Applications:

Table 18 shows the product attributes for each of the three TSKgel PWXL-CP columns. Figure 40 shows calibration curves produced with standard polyethylene oxide and polyethylene glycol in a 0.1 mol/L aqueous solution of sodium nitrate.

Table 18: Product attributes

TSKgel column	G3000PWXL-CP	G5000PWXL-CP	G6000PWXL-CP
Base material	polymethacrylate	polymethacrylate	polymethacrylate
Particle size	7 µm	10 µm	13 µm
Pore size	20 nm	100 nm	>100 nm
Exclusion limit	1.0×10^5 Da	1.0×10^6 Da	2.0×10^7 Da
Separation range (PEO, PEG)	200 ~ 5.0×10^4 Da	400 ~ 5.0×10^5 Da	1,000 ~ 1.0×10^7 Da
Theoretical plates	16,000	10,000	7,000

Figure 40: Polyethylene glycol and oxide calibration curves for TSKgel PWXL-CP columns

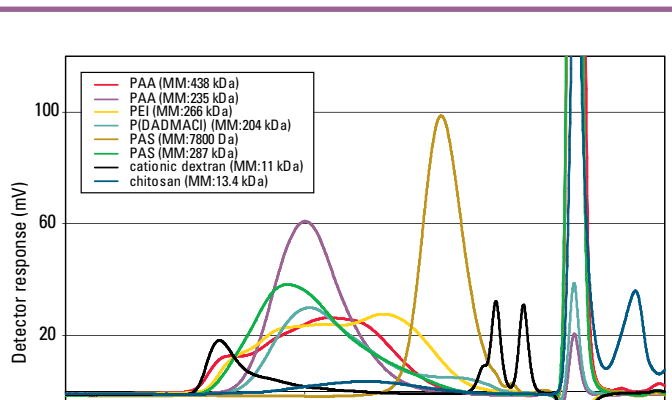


Columns:	**TSKgel G3000PWXL-CP, 7 µm, 7.8 mm ID × 30 cm** **TSKgel G5000PWXL-CP, 10 µm, 7.8 mm ID × 30 cm** **TSKgel G6000PWXL-CP, 13 µm, 7.8 mm ID × 30 cm**
Mobile phase:	H_2O with 0.1 mol/L $NaNO_3$
Flow Rate:	1 mL/min
Detection:	RI
Temperature:	25 °C
Samples:	polyethylene oxides (PEO) standards polyethylene glycols (PEG) standards

Cationic Polymers

Various cationic polymers with different functional groups and molar masses were injected on the three TSKgel PW$_{XL}$-CP columns (TSKgel G6000PW$_{XL}$-CP, G5000PW$_{XL}$-CP, and G3000PW$_{XL}$-CP) connected in series. Figure 41 demonstrates that these SEC columns can be utilized for the analysis of a wide variety of cationic polymers.

Figure 41: Analysis of cationic polymers



Columns:	**TSKgel G3000PW$_{XL}$-CP, 7 µm, 7.8 mm ID × 30 cm** **TSKgel G5000PW$_{XL}$-CP, 10 µm, 7.8 mm ID × 30 cm** **TSKgel G6000PW$_{XL}$-CP, 13 µm, 7.8 mm ID × 30 cm**
Mobile phase:	H_2O with 0.1 mol/L $NaNO_3$
Flow Rate:	1 mL/min
Detection:	RI
Temperature:	25 °C
Sample Load:	3 g/L, 100 µL

TSKgel SuperMultiporePW Size Exclusion Columns

The innovative multi-pore particle synthesis technology*, pioneered by Tosoh scientists, is incorporated into TSKgel SuperMultiporePW columns for water-soluble polymer analysis. Three semi-micro columns varying in linear range are available within this series, enabling high speed and high resolution analysis with lowered solvent consumption. The base material of each TSKgel SuperMultiporePW column is polymethacrylate.

A wide molar mass range can be analyzed with the three different TSKgel SuperMultiporePW columns, from high molar mass water-soluble polymers to oligomers. The packing material in the TSKgel SuperMultiporePW columns is more hydrophilic than that of TSKgel PWXL series columns, which further reduces the chance of adsorption of hydrophilic polymers.

- TSKgel SuperMultiporePW-N
- TSKgel SuperMultiporePW-M
- TSKgel SuperMultiporePW-H

*Using this proprietary technology, Tosoh can manufacture particles, each containing 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.

Attributes and Applications:

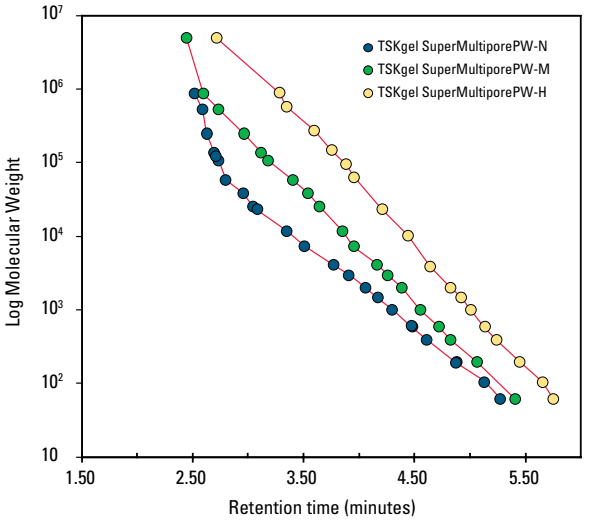
Table 19 shows the product attributes for each of the three TSKgel SuperMultiporePW columns. Figure 42 shows polyethylene glycol, oxide and ethylene glycol calibration curves for each of the TSKgel SuperMultiporePW columns.

Table 19: Product attributes

TSKgel column	SuperMultiporePW-N	SuperMultiporePW-M	SuperMultiporePW-H
Base material	polymethacrylate	polymethacrylate	polymethacrylate
Particle size	4 µm*	5 µm*	8 µm*
Pore size	20 nm	100 nm	>100 nm
Exclusion limit (PEO, PEG/H_2O)	1.0×10^5 - 1.5×10^5 Da	6.0×10^5 - 1.5×10^6 Da	-
Separation range	300 ~ 5.0×10^4 Da	500 ~ 1.0×10^6 Da	1,000 ~ 1.0×10^7 Da
Theoretical plates/15cm column	>16,000	>12,000	>7,000

* Particle size distribution is monodisperse.

Figure 42: Polyethylene glycol, oxide, and ethylene glycol calibration curves for TSKgel SuperMultiporePW columns



Columns:	**TSKgel SuperMultiporePW-N, 4 µm, 6.0 mm ID × 15 cm** **TSKgel SuperMultiporePW-M, 5 µm, 6.0 mm ID × 15 cm** **TSKgel SuperMultiporePW-H, 8 µm, 6.0 mm ID × 15 cm**
Mobile phase:	H_2O
Flow rate:	0.60 mL/min
Detection:	RI
Temperature:	25 °C
Samples:	polyethylene oxides (PEO) standards polyethylene glycols (PEG) standards ethylene glycol (EG) standards

Ordering Information - TSKgel H columns

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
16131	TSKgel G1000H$_{XL}$, 5 µm, 1.5 nm	polymer	Stainless Steel	7.8	30
16134	TSKgel G2000H$_{XL}$, 5 µm, 2 nm	polymer	Stainless Steel	7.8	30
16135	TSKgel G2500H$_{XL}$, 5 µm, 3 nm	polymer	Stainless Steel	7.8	30
16136	TSKgel G3000H$_{XL}$, 6 µm, 7.5 nm	polymer	Stainless Steel	7.8	30
16137	TSKgel G4000H$_{XL}$, 5 µm, 20 nm	polymer	Stainless Steel	7.8	30
16138	TSKgel G5000H$_{XL}$, 9 µm, 65 nm	polymer	Stainless Steel	7.8	30
16139	TSKgel G6000H$_{XL}$, 9 µm, >65 nm	polymer	Stainless Steel	7.8	30
16140	TSKgel G7000H$_{XL}$, 9 µm, >65 nm	polymer	Stainless Steel	7.8	30
16141	TSKgel GMH$_{XL}$, 9 µm, mixed bed	polymer	Stainless Steel	7.8	30
16652	TSKgel GMH$_{XL}$-L, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
18403	TSKgel MultiporeH$_{XL}$-M, 5 µm	polymer	Stainless Steel	7.8	30
07113	TSKgel Guard Column for 7.8 mm ID TSKgel G1000H$_{XL}$-G4000H$_{XL}$ columns, 8 µm	polymer	Stainless Steel	6	4
13727	TSKgel Guard Column for 7.8 mm ID TSKgel G5000H$_{XL}$-GMH$_{XL}$ & GMH$_{XL}$-L columns, 13 µm	polymer	Stainless Steel	6	4
18404	TSKgel Guard Column for TSKgel MultiporeH$_{XL}$-M column, 5 µm	polymer	Stainless Steel	6	4
17352	TSKgel G1000H$_{HR}$, 5 µm, 1.5 nm	polymer	Stainless Steel	7.8	30
17353	TSKgel G2000H$_{HR}$, 5 µm, 2 nm	polymer	Stainless Steel	7.8	30
17354	TSKgel G2500H$_{HR}$, 5 µm, 3 nm	polymer	Stainless Steel	7.8	30
17355	TSKgel G3000H$_{HR}$, 5 µm, 7.5 nm	polymer	Stainless Steel	7.8	30
17356	TSKgel G4000H$_{HR}$, 5 µm, 20 nm	polymer	Stainless Steel	7.8	30
17357	TSKgel G5000H$_{HR}$, 5 µm, 65 nm	polymer	Stainless Steel	7.8	30
17358	TSKgel G6000H$_{HR}$, 5 µm, >65 nm	polymer	Stainless Steel	7.8	30
17359	TSKgel G7000H$_{HR}$, 5 µm, >65 nm	polymer	Stainless Steel	7.8	30
17362	TSKgel GMH$_{HR}$-L, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
17392	TSKgel GMH$_{HR}$-M, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
18055	TSKgel GMH$_{HR}$-N, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
17360	TSKgel GMH$_{HR}$-H, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
17361	TSKgel GMH$_{HR}$-H (S), 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
17393	TSKgel GMH$_{HR}$-M (S), 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
18399	TSKgel GMH$_{HR}$-H (20), 20 µm, mixed bed	polymer	Stainless Steel	7.8	30
18398	TSKgel GMH$_{HR}$-H (30), 30 µm, mixed bed	polymer	Stainless Steel	7.8	30
18420	TSKgel GMH$_{HR}$-H HT, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
18393	TSKgel GMH$_{HR}$-H (S) HT, 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
18392	TSKgel GMH$_{HR}$-H (20) HT, 20 µm, mixed bed	polymer	Stainless Steel	7.8	30
18391	TSKgel GMH$_{HR}$-H (30) HT, 30 µm, mixed bed	polymer	Stainless Steel	7.8	30
18395	TSKgel G2000H$_{HR}$ (20) HT, 20 µm, 2 nm	polymer	Stainless Steel	7.8	30
22888	TSKgel GMH$_{HR}$-H (20) HT2, 20 µm, mixed bed	polymer	Stainless Steel	7.8	30
22887	TSKgel GMH$_{HR}$-H (30) HT2, 30 µm, mixed bed	polymer	Stainless Steel	7.8	30
22889	TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, mixed bed	polymer	Stainless Steel	7.8	30

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
22890	TSKgel G2000H$_{HR}$ (20) HT2, 20 µm, 2 nm	polymer	Stainless Steel	7.8	30
17368	TSKgel Guard Column for 7.8 mm ID TSKgel G1000H$_{HR}$-G4000H$_{HR}$ & GMH$_{HR}$-L columns, 5 µm	polymer	Stainless Steel	6	4
17369	TSKgel Guard Column for 7.8 mm ID TSKgel G5000H$_{HR}$-G7000H$_{HR}$ & GMH$_{HR}$-M;-N;-H columns, 5 µm	polymer	Stainless Steel	6	4
17367	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (S), -M (S) columns, 13 µm	polymer	Stainless Steel	7.5	7.5
18402	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (20), -H (30) columns, 30 µm	polymer	Stainless Steel	7.5	7.5
18397	TSKgel Guard Column for 7.8 mm ID TSKgel GMH$_{HR}$-H (S) HT column, 13 µm	polymer	Stainless Steel	7.5	7.5
18396	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (20) HT & GMH$_{HR}$-H (30) HT columns, 30 µm	polymer	Stainless Steel	7.5	7.5
22891	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (20) HT2 & GMH$_{HR}$-H (30) HT2 columns, 30 µm	polymer	Stainless Steel	7.5	7.5
22892	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (S) HT2 column, 13 µm	polymer	Stainless Steel	7.5	7.5
17990	TSKgel SuperH1000, 3 µm, 1.5 nm	polymer	Stainless Steel	6	15
17991	TSKgel SuperH2000, 3 µm, 2 nm	polymer	Stainless Steel	6	15
17992	TSKgel SuperH2500, 3 µm, 3 nm	polymer	Stainless Steel	6	15
17993	TSKgel SuperH3000, 3 µm, 7.5 nm	polymer	Stainless Steel	6	15
17994	TSKgel SuperH4000, 3 µm, 20 nm	polymer	Stainless Steel	6	15
17995	TSKgel SuperH5000, 3 µm, 65 nm	polymer	Stainless Steel	6	15
17996	TSKgel SuperH6000, 5 µm, >65 nm	polymer	Stainless Steel	6	15
17997	TSKgel SuperH7000, 5 µm, >65 nm	polymer	Stainless Steel	6	15
17998	TSKgel SuperHM-L, 3 µm, mixed bed	polymer	Stainless Steel	6	15
17999	TSKgel SuperHM-N, 3 µm, mixed bed	polymer	Stainless Steel	6	15
18000	TSKgel SuperHM-M, 3 µm, mixed bed	polymer	Stainless Steel	6	15
18001	TSKgel SuperHM-H, 3 µm, mixed bed	polymer	Stainless Steel	6	15
18002	TSKgel Guard Column for 6 mm ID TSKgel SuperH1000-SuperH4000 columns, 3 µm	polymer	Stainless Steel	4.6	3.5
18003	TSKgel Guard Column for 6 mm ID TSKgel SuperH5000-7000;HM-L;-N;-M;-H columns, 3 µm	polymer	Stainless Steel	4.6	3.5
19309	TSKgel SuperHZ1000, 3 µm, 1.5 nm	polymer	Stainless Steel	4.6	15
19310	TSKgel SuperHZ2000, 3 µm, 2 nm	polymer	Stainless Steel	4.6	15
19311	TSKgel SuperHZ2500, 3 µm, 3 nm	polymer	Stainless Steel	4.6	15
19312	TSKgel SuperHZ3000, 3 µm, 7.5 nm	polymer	Stainless Steel	4.6	15
19313	TSKgel SuperHZ4000, 3 µm, 20 nm	polymer	Stainless Steel	4.6	15
19660	TSKgel SuperHZM-N, 3 µm, mixed bed	polymer	Stainless Steel	4.6	15
19662	TSKgel SuperHZM-M, 3 µm, mixed bed	polymer	Stainless Steel	4.6	15
19664	TSKgel SuperHZM-H, 10 µm, mixed bed	polymer	Stainless Steel	4.6	15
19302	TSKgel SuperHZ1000, 3 µm, 1.5 nm	polymer	Stainless Steel	6	15

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
19303	TSKgel SuperHZ2000, 3 µm, 2 nm	polymer	Stainless Steel	6	15
19304	TSKgel SuperHZ2500, 3 µm, 3 nm	polymer	Stainless Steel	6	15
19305	TSKgel SuperHZ3000, 3 µm, 7.5 nm	polymer	Stainless Steel	6	15
19306	TSKgel SuperHZ4000, 3 µm, 20 nm	polymer	Stainless Steel	6	15
19661	TSKgel SuperHZM-N, 3 µm, mixed bed	polymer	Stainless Steel	6	15
19663	TSKgel SuperHZM-M, 3 µm, mixed bed	polymer	Stainless Steel	6	15
19665	TSKgel SuperHZM-H, 10 µm, mixed bed	polymer	Stainless Steel	6	15
19314	TSKgel Guard Column for 4.6 mm ID TSKgel SuperHZ1000-4000 and HZM-N & -M columns, 3 µm	polymer	Stainless Steel	4.6	2
19668	TSKgel Guard Column for 4.6 mm ID TSKgel SuperHZM-H column, 10 µm	polymer	Stainless Steel	4.6	2
19666	TSKgel Guard Column for 6 mm ID TSKgel SuperHZ1000-4000 and HZM-N & -M columns, 3 µm	polymer	Stainless Steel	4.6	3.5
19667	TSKgel Guard Column for 6 mm ID TSKgel SuperHZM-H column, 10 µm	polymer	Stainless Steel	4.6	3.5
21815	TSKgel SuperMultiporeHZ-N, 3 µm, 8 nm	polymer	Stainless Steel	4.6	15
21885	TSKgel SuperMultiporeHZ-H, 6 µm, >14 nm	polymer	Stainless Steel	4.6	15
21488	TSKgel SuperMultiporeHZ-M, 4 µm, 14 nm	polymer	Stainless Steel	4.6	15
21816	TSKgel SuperMPHZ-N Guard, 3 µm	polymer	Stainless Steel	4.6	2
21886	TSKgel SuperMPHZ-H Guard, 6 µm	polymer	Stainless Steel	4.6	2
21489	TSKgel SuperMPHZ-M Guard, 4 µm	polymer	Stainless Steel	4.6	2

Ordering Information - TSKgel SuperAW and Alpha columns

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
19315	TSKgel SuperAW2500, 4 µm, 2.5 nm	polymer	Stainless Steel	6	15
19316	TSKgel SuperAW3000, 4 µm, 15 nm	polymer	Stainless Steel	6	15
19317	TSKgel SuperAW4000, 6 µm, 45 nm	polymer	Stainless Steel	6	15
19318	TSKgel SuperAW5000, 7 µm, 100 nm	polymer	Stainless Steel	6	15
19319	TSKgel SuperAW6000, 9 µm, >100 nm	polymer	Stainless Steel	6	15
19320	TSKgel SuperAWM-H, 9 µm, mixed bed	polymer	Stainless Steel	6	15
19321	TSKgel Guard Column for 6.0 mm ID TSKgel SuperAW2500-4000 columns, 7 µm	polymer	Stainless Steel	4.6	3.5
19322	TSKgel Guard Column for 6.0 mm ID TSKgel SuperAW5000-AWM-H columns, 13 µm	polymer	Stainless Steel	4.6	3.5
18339	TSKgel Alpha-2500, 7 µm, 2.5 nm	polymer	Stainless Steel	7.8	30
18340	TSKgel Alpha-3000, 7 µm, 15 nm	polymer	Stainless Steel	7.8	30
18341	TSKgel Alpha-4000, 10 µm, 45 nm	polymer	Stainless Steel	7.8	30
18342	TSKgel Alpha-5000, 10 µm, 100 nm	polymer	Stainless Steel	7.8	30
18343	TSKgel Alpha-6000, 13 µm, >100 nm	polymer	Stainless Steel	7.8	30
18344	TSKgel Alpha-M, 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
18345	TSKgel Guard Column for 7.8 mm ID TSKgel Alpha-2500-Alpha-M columns, 13 µm	polymer	Stainless Steel	6	4

Ordering Information - TSKgel PW columns

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
20024	TSKgel BioAssist G6PW, 17 µm, >100 nm	polymer	PEEK	7.8	30
05761	TSKgel G2000PW, 12 µm, 12.5 nm	polymer	Stainless Steel	7.5	30
05105	TSKgel G2000PW, 12 µm, 12.5 nm	polymer	Stainless Steel	7.5	60
08028	TSKgel G2500PW, 12 µm, 12.5 nm	polymer	Stainless Steel	7.5	30
08029	TSKgel G2500PW, 12 µm, 12.5 nm	polymer	Stainless Steel	7.5	60
05762	TSKgel G3000PW, 12 µm, 20 nm	polymer	Stainless Steel	7.5	30
05106	TSKgel G3000PW, 12 µm, 20 nm	polymer	Stainless Steel	7.5	60
05763	TSKgel G4000PW, 17 µm, 50 nm	polymer	Stainless Steel	7.5	30
05107	TSKgel G4000PW, 17 µm, 50 nm	polymer	Stainless Steel	7.5	60
05764	TSKgel G5000PW, 17 µm, 100 nm	polymer	Stainless Steel	7.5	30
05108	TSKgel G5000PW, 17 µm, 100 nm	polymer	Stainless Steel	7.5	60
05765	TSKgel G6000PW, 17 µm, >100 nm	polymer	Stainless Steel	7.5	30
05109	TSKgel G6000PW, 17 µm, >100 nm	polymer	Stainless Steel	7.5	60
08026	TSKgel GMPW, 17 µm, mixed bed	polymer	Stainless Steel	7.5	30
08027	TSKgel GMPW, 17 µm, mixed bed	polymer	Stainless Steel	7.5	60
16248	TSKgel G2500PW, 17 µm, 12.5 nm	polymer	Stainless Steel	21.5	30
16249	TSKgel G3000PW, 17 µm, 20 nm	polymer	Stainless Steel	21.5	30
08030	TSKgel G2500PW, 17 µm, 12.5 nm	polymer	Stainless Steel	21.5	60
06763	TSKgel Guard Column for 7.5 mm ID TSKgel G2000PW columns, 13 µm	polymer	Stainless Steel	7.5	7.5
06762	TSKgel Guard Column for 7.5 mm ID TSKgel G2500PW-GMPW columns, 13 µm	polymer	Stainless Steel	7.5	7.5
06758	TSKgel Guard Column for 21.5 mm ID TSKgel G2500-G3000PW columns, 17 µm	polymer	Stainless Steel	21.5	7.5
08020	TSKgel G2500PW$_{XL}$, 7 µm, 12.5 nm	polymer	Stainless Steel	7.8	30
08021	TSKgel G3000PW$_{XL}$, 7 µm, 20 nm	polymer	Stainless Steel	7.8	30
08022	TSKgel G4000PW$_{XL}$, 10 µm, 50 nm	polymer	Stainless Steel	7.8	30
08023	TSKgel G5000PW$_{XL}$, 10 µm, 100 nm	polymer	Stainless Steel	7.8	30
08024	TSKgel G6000PW$_{XL}$, 13 µm, >100 nm	polymer	Stainless Steel	7.8	30
08025	TSKgel GMPW$_{XL}$, 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
08032	TSKgel G-DNA-PW, 10 µm, >100 nm	polymer	Stainless Steel	7.8	30
08031	TSKgel G-Oligo-PW, 7 µm, 12.5 nm	polymer	Stainless Steel	7.8	30
22792	TSKgel SuperOligoPW, 3 µm, 12.5 nm	polymer	Stainless Steel	6	15
08033	TSKgel Guard Column for 7.8 mm ID TSKgel G2500PW$_{XL}$-GMPW$_{XL}$ columns, 12 µm	polymer	Stainless Steel	6	4
08033	TSKgel Guard Column for 7.8 mm ID TSKgel G-DNA-PW column, 12 µm	polymer	Stainless Steel	6	4
08034	TSKgel Guard Column for 7.8 mm ID TSKgel G-Oligo-PW column, 13 µm	polymer	Stainless Steel	6	4
22796	TSKgel Guard Column for 6 mm ID TSKgel SuperOligoPW column, 4 µm	polymer	Stainless Steel	4.6	3.5

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
21873	TSKgel G3000PWXL-CP, 7 µm, 20 nm	polymer	Stainless Steel	7.8	30
21874	TSKgel G5000PWXL-CP, 10 µm, 100 nm	polymer	Stainless Steel	7.8	30
21875	TSKgel G6000PWXL-CP, 13 µm, >100 nm	polymer	Stainless Steel	7.8	30
21876	TSKgel Guard Column for 7.8 mm ID TSKgel G3000-G6000PWXL-CP columns, 13 µm	polymer	Stainless Steel	6	4
22789	TSKgel SuperMultiporePW-N, 4 µm, 20 nm	polymer	Stainless Steel	6	15
22790	TSKgel SuperMultiporePW-M, 5 µm, 100 nm	polymer	Stainless Steel	6	15
22791	TSKgel SuperMultiporePW-H, 8 µm, >100 nm	polymer	Stainless Steel	6	15
22794	TSKgel SuperMP(PW)-M Guard, 8 µm	polymer	Stainless Steel	4.6	3.5
22793	TSKgel SuperMP(PW)-N Guard, 5 µm	polymer	Stainless Steel	4.6	3.5
22795	TSKgel SuperMP(PW)-H Guard, 12 µm	polymer	Stainless Steel	4.6	3.5
08035	TSKgel Top-Off for PWXL and G-DNA-PW, 10 µm, 1 g	polymer			

TSKgel High Temperature GPC Columns

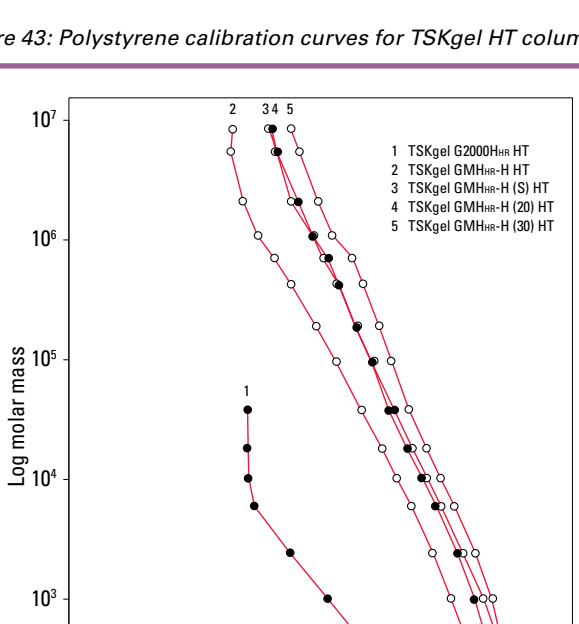
TSKgel H_{HR} HT and HT2 high temperature columns are recommended for the analysis of organic-soluble polymers and are packed with spherical particles composed of polystyrene cross-linked with divinylbenzene (PS-DVB). The "GM" prefix denotes a column packed with particles of different pore sizes blended to provide an extended linear calibration curve. The TSKgel HT columns are for high temperature applications (≤ 140 °C) while the TSKgel HT2 columns are used in ultra-high temperature (up to 220 °C) applications.

Table 20 lists the attributes of the TSKgel H_{HR} HT columns which are for high temperature applications up to 140 °C. Figure 43 shows the polystyrene calibration curves for each of the TSKgel H_{HR} HT columns.

Table 20: Properties and separation ranges for TSKgel HT columns

TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
GMH_{HR}-H HT	5 µm	mixed pore sizes	4.0 × 10^8 Da	140 °C
GMH_{HR}-H (S) HT	13 µm	mixed pore sizes	4.0 × 10^8 Da	140 °C
GMH_{HR}-H (20) HT	20 µm	mixed pore sizes	4.0 × 10^8 Da	140 °C
GMH_{HR}-H (30) HT	30 µm	mixed pore sizes	4.0 × 10^8 Da	140 °C
$G2000H_{HR}$ (20) HT	20 µm	2 nm	1.0 × 10^4 Da	140 °C

Figure 43: Polystyrene calibration curves for TSKgel HT columns



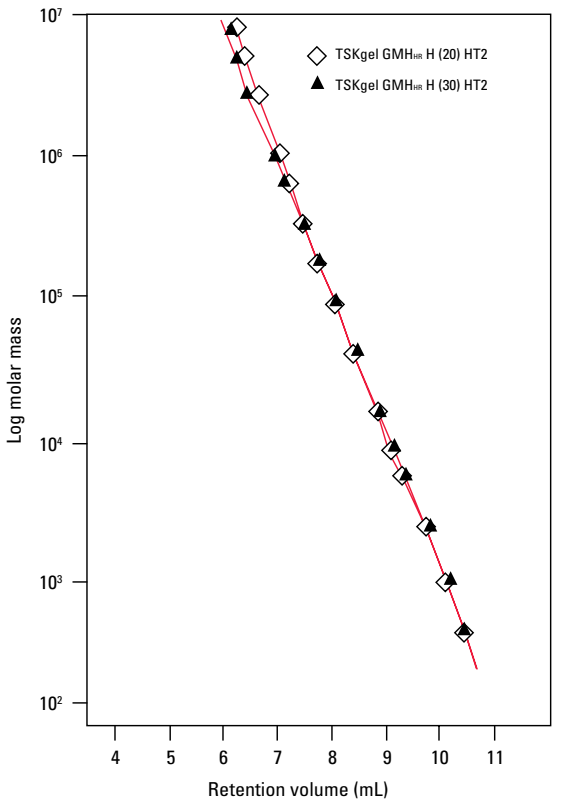
Columns: **TSKgel $G2000H_{HR}$ (20) HT, 20 µm, 7.8 mm ID × 30 cm**
TSKgel GMH_{HR}-H HT, 5 µm, 7.8 mm ID × 30 cm
TSKgel GMH_{HR}-H (S) HT, 13 µm, 7.8 mm ID × 30 cm
TSKgel GMH_{HR}-H (20) HT, 20 µm, 7.8 mm ID × 30 cm
TSKgel GMH_{HR}-H (30) HT, 30 µm, 7.8 mm ID × 30 cm
Mobile phase: ODCB with 0.05% BHT
Flow rate: 1.0 mL/min
Detector: RI (EcoSEC High Temperature GPC System)
Temperature: 135 °C
Injection vol.: 300 µL
Sample: polystyrene standards

The TSKgel high temperature column series also includes four columns for the analysis of polymers at ultra-high temperatures (up to 220 °C). The TSKgel H_{HR} HT2 columns are specifically designed for the analysis of organic-soluble polymers at extremely elevated temperatures. The attributes of the TSKgel HT2 column series are listed in Table 21. Figure 44 shows the polystyrene calibration curves for each of the TSKgel H_{HR} HT2 columns.

Table 21: Properties and separation ranges for TSKgel HT2 columns

TSKgel column	Particle size	Pore size	Exclusion limit	Max. temp.
GMH_{HR}-H (20) HT2	20 µm	mixed pore sizes	4.0 × 10^8 Da	220 °C
GMH_{HR}-H (30) HT2	30 µm	mixed pore sizes	4.0 × 10^8 Da	220 °C
GMH_{HR}-H (S) HT2	13 µm	mixed pore sizes	4.0 × 10^8 Da	220 °C
G2000H_{HR} (20) HT2	20 µm	2 nm	1.0 × 10^4 Da	220 °C

Figure 44: Polystyrene calibration curves for TSKgel HT2 columns

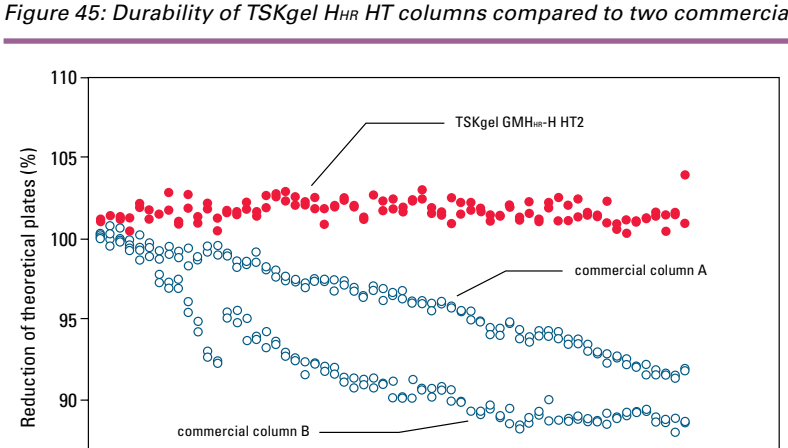


Columns: **TSKgel GMH_{HR}-H (20) HT2, 20 µm, 7.8 mm ID × 30 cm**
TSKgel GMH_{HR}-H (30) HT2, 30 µm, 7.8 mm ID × 30 cm
Mobile phase: ODCB with 0.05% BHT
Flow rate: 1.0 mL/min
Detector: RI (EcoSEC High Temperature GPC System)
Temperature: 135 °C
Sample: polystyrene standards

Performance Stability

Figure 45 demonstrates the performance stability of the TSKgel GMH$_{HR}$-H HT columns compared to other commercially available high temperature GPC columns during repetitive temperature changes. The TSKgel H$_{HR}$ HT columns and two commercially available high temperature GPC columns were subjected to drastic changes in temperature by raising the temperature for 2 hours followed by lowering the temperature for two hours for a total of 60 cycles. The number of theoretical plates was shown to remain constant for the TSKgel H$_{HR}$ HT columns and to decrease by 15% for the two commercially available high temperature GPC columns; thus revealing the superior performance stability of the TSKgel H$_{HR}$ HT columns.

Figure 45: Durability of TSKgel H$_{HR}$ HT columns compared to two commercially available high temperature GPC columns



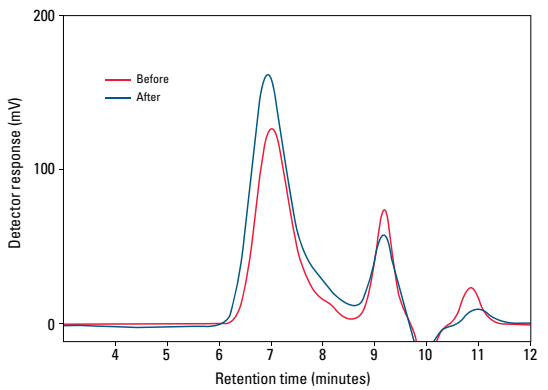
Column:	**TSKgel GMH$_{HR}$-H HT2, 5 µm, 7.8 mm ID × 30 cm × 2**
Mobile phase:	ODCB with 0.05% BHT
Flow rate:	1 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	40 to 145 °C

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.

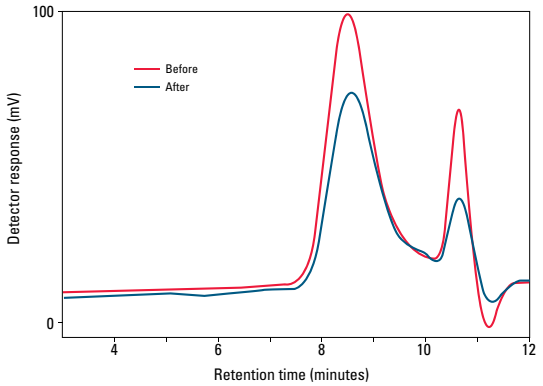
A durability and stability study of a TSKgel GMH$_{HR}$-H (S) HT high temperature GPC column was performed and the results were compared to another commercially available column for polymer analysis at 220 °C. The deterioration of the commercially available high temperature GPC column is observed in the GPC elution profiles, Figure 46, 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 47.

Figure 46: 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 µm, 7.8 mm ID × 30 cm
Mobile phase:	1-CN
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	220 °C
Injection vol.:	200 µL
Sample:	synthetic polymer

Figure 47: 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 µm, 7.8 mm ID × 30 cm**
Mobile phase:	1-CN
Flow rate:	1.0 mL/min
Detector:	RI (EcoSEC High Temperature GPC System)
Temperature:	220 °C
Injection vol.:	200 µL
Sample:	synthetic polymer

Ordering Information - TSKgel High Temperature GPC Columns

Part #	Description	Matrix	Housing	ID (mm)	Length (cm)
18420	TSKgel GMH$_{HR}$-H HT, 5 µm, mixed bed	polymer	Stainless Steel	7.8	30
18393	TSKgel GMH$_{HR}$-H (S) HT, 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
18392	TSKgel GMH$_{HR}$-H (20) HT, 20 µm, mixed bed	polymer	Stainless Steel	7.8	30
18391	TSKgel GMH$_{HR}$-H (30) HT, 30 µm, mixed bed	polymer	Stainless Steel	7.8	30
18395	TSKgel G2000H$_{HR}$ (20) HT, 20 µm, 2 nm	polymer	Stainless Steel	7.8	30
18397	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (S) HT column, 30 µm	polymer	Stainless Steel	7.5	7.5
18396	TSKgel Guard Column for TSKgel GMH$_{HR}$- H (20) HT & GMH$_{HR}$-H (30) HT columns, 30 µm	polymer	Stainless Steel	7.5	7.5
22888	TSKgel GMH$_{HR}$-H (20) HT2, 20 µm, mixed bed	polymer	Stainless Steel	7.8	30
22887	TSKgel GMH$_{HR}$-H (30) HT2, 30 µm, mixed bed	polymer	Stainless Steel	7.8	30
22889	TSKgel GMH$_{HR}$-H (S) HT2, 13 µm, mixed bed	polymer	Stainless Steel	7.8	30
22890	TSKgel G2000H$_{HR}$ (20) HT2, 20 µm, 2 nm	polymer	Stainless Steel	7.8	30
22891	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (20) HT2 & GMH$_{HR}$-H (30) HT2 columns, 30 µm	polymer	Stainless Steel	7.5	7.5
22892	TSKgel Guard Column for TSKgel GMH$_{HR}$-H (S) HT2 column, 13 µm	polymer	Stainless Steel	7.5	7.5


TOSOH
TSKgel
TSKgel GMHhr-H (30) HT2
TOSOH
TSKgel
TSKgel GMHhr-H (30) HT2

Standards, Components and Replacement Parts

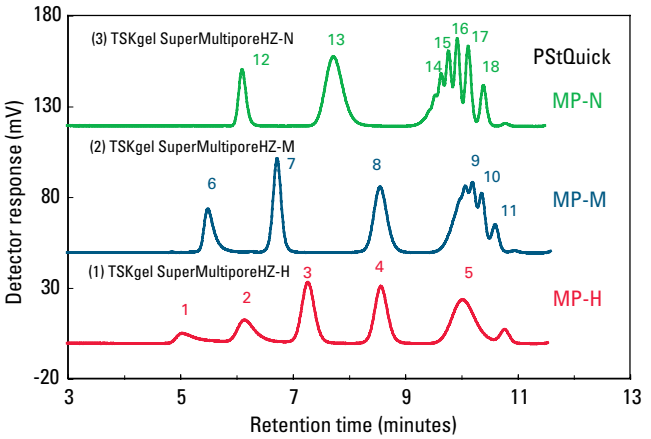
- Tosoh Bioscience offers bulk quantities of polystyrene and polyethylene oxide standards, as well as pre-mixed quantities of polystyrene polymers, for calibration of GPC columns.

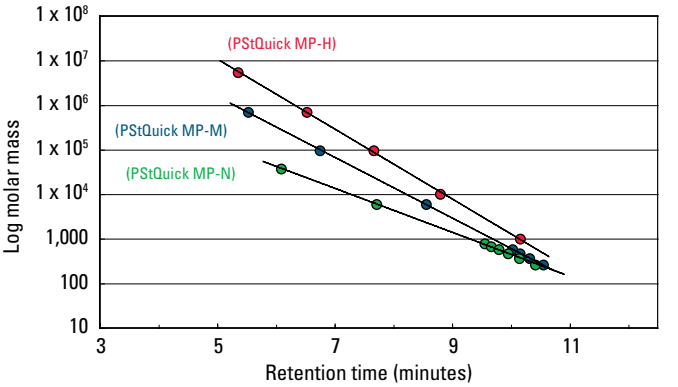
- Components and replacement parts are available for the EcoSEC GPC System and EcoSEC High Temperature GPC System.

PStQuick GPC Polystyrene Calibration Standards

PStQuick polystyrene calibration standards contain pre-mixed quantities of polystyrene polymers in autosampler vials for the calibration of GPC columns. Addition of solvent is all that is required for easy preparation and analysis. 12 different kits containing polystyrene polymers of various molar masses are available. Of the 12 kits, 9 are individual kits, each containing 3 to 5 polystyrene polymers. The remaining 3 are composite kits containing 2 or 3 of the individual kits.

Figure 1: Chromatograms and calibration curves obtained using the PStQuick MP series





PStQuick MP-H	PStQuick MP-M	PStQuick MP-N
1. M_w: 5.48 × 10^6	6. M_w: 7.06 × 10^5	12. M_w: 3.79 × 10^4
2. M_w: 7.06 × 10^5	7. M_w: 9.64 × 10^4	13. M_w: 5,970
3. M_w: 9.64 × 10^4	8. M_w: 5,970	14. M_w: 682
4. M_w: 1.02 × 10^4	9. M_w: 474	15. M_w: 578
5. M_w: 1,010	10. M_w: 370	16. M_w: 474
	11. M_w: 266	17. M_w: 370
		18. M_w: 266

Columns: **SuperMultiporeHZ-H, 6 µm, 4.6mm ID x 15cm x 2**
SuperMultiporeHZ-M, 4 µm, 4.6mm ID x 15cm x 2
SuperMultiporeHZ-N, 3 µm, 4.6mm ID x 15cm x 2
Mobile phase: THF
Flow rate: 0.35 mL/min
Detection: UV @ 254 nm (UV-8020 microcell)
Temperature: 25 °C
Injection vol.: 10 µL
Sample: PStQuick MP series

Contents of each kit

For example, PStQuick Kit-M contains 20 vials each of grades C and D.

Polystyrene Calibration Mixtures	TSKgel column	A	B	C	D	E	F	G	H	Vials
PStQuick MP-N	SuperMultiporeHZ-N								●	60
PStQuick MP-M	SuperMultiporeHZ-M							●		60
PStQuick MP-H	SuperMultiporeHZ-H		●							60
PStQuick Kit -H (High MW)	Mixed Bed H-type	●	●	●						60 (3 x 20)
PStQuick Kit -M (Medium MW)	Mixed Bed M-type			●	●					40 (2 x 20)
PStQuick Kit -L Low MW)	Mixed Bed N-type					●	●			40 (2 x 20)

Nominal MW of Kit Components

For example, grade B contains polystyrene polymers of nominal molecular weights 5,480,000 - 706,000, 96,400 - 10,200 and 1,000. In the above Table it is shown that grade B is part of PStQuick MP-H and PStQuick Kit-H

Polystyrene MW	A	B	C	D	E	F	G	H
8,420,000	●							●
5,480,000		●						
2,890,000			●					
1,090,000	●			●				
706,000		●					●	
355,000			●		●			
190,000	●			●		●		
96,400		●					●	
37,900			●		●			●
18,100	●			●		●		
10,200		●						
5,970			●		●		●	●
2,500	●			●		●		
1,000		●			●			
500			●			●	●	●

Ordering Information - PStQuick Polystyrene calibration standards

To calibrate TSKgel SuperMultiporeHZ columns

Part #	Description	Remarks	Calibration Range	Contents	Vials
21912	PStQuick MP-N	For SuperMultiporeHZ-N	530 to 4.4 x 10^4	A-500, A-5000, F-4	60
21913	PStQuick MP-M	For SuperMultiporeHZ-M	530 to 8.0 x 10^5	A-500, A-5000, F-10, F-80	60
21914	PStQuick MP-H	For SuperMultiporeHZ-H	950 to 5.5 x 10^6	A-1000, F-1, F-10, F-80, F-550	60

To calibrate TSKgel H-type mixed bed columns

Part #	Description	Remarks	Calibration Range	Contents	Vials
21915	PStQuick Kit-L	For H-type – N grade	530 to 4.2 x 10^5	PStQuick E, F	40**
21916	PStQuick Kit-M	For H-type – M grade	530 to 2.9 x 10^6	PStQuick C, D	40**
21917	PStQuick Kit-H	For H-type – H grade	530 to 8.4 x 10^6	PStQuick A, B, C	60*

*20 of each type x 3, **20 of each type x 2

To calibrate other TSKgel GPC columns

Part #	Description	Remarks	Calibration Range	Contents	Vials
21911	PStQuick A	For Other GPC columns	2,800 to 8.4 x 10^6	A-2500, F-2, F-20, F-128, F-850	20
21910	PStQuick B	For Other GPC columns	950 to 5.5 x 10^6	A-1000, F-1, F-10, F-80, F-550	20
21909	PStQuick C	For Other GPC columns	530 to 2.9 x 10^6	A-500, A-5000, F-4, F-40, F-288	20
21908	PStQuick D	For Other GPC columns	2,800 to 1.3 x 10^6	A-2500, F-2, F-20, F-128	20
21907	PStQuick E	For Other GPC columns	950 to 4.2 x 10^5	A-1000, A-5000, F-4, F-40	20
21906	PStQuick F	For Other GPC columns	530 to 1.9 x 10^5	A-500, A-2500, F-2, F-20	20

TSKgel Polystyrene Calibration Standards

TSKgel polystyrene bulk calibration standards are used to calibrate GPC columns for subsequent analysis of unknown samples. The standards range from 400 to 2.1 × 10^7 Da.

Ordering Information - TSKgel Polystyrene calibration standards

Part #	Description	Weight
05202	A-300, 400 Da	10 g
05203	A-500, 530 Da	10 g
05204	A-1000, 950 Da	10 g
05205	A-2500, 2,800 Da	5 g
05206	A-5000, 6,200 Da	5 g
05207	F-1, 1.0 x 10^4 Da	5 g
05208	F-2, 1.7 x 10^4 Da	5 g
05209	F-4, 4.4 x 10^4 Da	5 g
05210	F-10, 1.0 x 10^5 Da	5 g
05211	F-20, 1.9 x 10^5 Da	5 g
05212	F-40, 4.2 x 10^5 Da	5 g
05213	F-80, 7.8 x 10^5 Da	5 g
05214	F-128, 1.3 x 10^6 Da	1 g
05215	F-288, 2.9 x 10^6 Da	1 g
05216	F-380, 3.8 x 10^6 Da	1 g
05217	F-450, 4.5 x 10^6 Da	1 g
05218	F-550, 5.5 x 10^6 Da	1 g
05219	F-700, 6.8 x 10^6 Da	1 g
05220	F-850, 8.4 x 10^6 Da	1 g
05221	F-2000, 2.1 x 10^7 Da	1 g
06476	Oligomer Kit, A-500 thru F-128	12 x 1 g
06477	High MW Kit, F-10 thru F-2000	12 x 1 g

EcoSEC GPC System: Optional Components and Replacement Parts

Tosoh Bioscience offers the following replacement parts and optional components for the EcoSEC GPC System. In addition, preventative and basic maintenance kits are available for those parts that experience wear and tear due to normal usage.

Tosoh Bioscience offers extended service agreements and on-site periodic maintenance service calls. Please contact us for additional information or a quote for these services.

Part #	Description
Optional Components	
21792	UV-8320 Detector, 2 µL cell
21793	Column Switching Valve
18004	TSKgel SuperH-RC Reference Column
Autosampler Accessories	
06456	Needle, 1/16" OD, 45 mm Length, 90 degree , 12/pk
16414	Rotor Seal for 4-way Valve
16415	Rotor Seal for 6-way Valve
22015	Sample Rack
22020	Needle Assembly
22054	Syringe Assembly
05462	Sample Loop, SS, 50 µL
05679	Sample Loop, SS, 100 µL
05464	Sample Loop, SS, 500 µL
05672	Sample Loop, SS, 1000 µL
07035	Sample Loop, SS, 1500 µL
89239-030	Sample vial with disposable caps and septum, glass, 2 mL, 100/pk
17538	Drain Tube, Teflon, for Autoinjector
22016	Drain Block Seal
Pumps and Accessories	
06574	Mobile Phase Inlet Filter, SS, 5 µm pores
18517	Piston Seal, Polyethylene - for Aqueous
18524	Mold to Replace Piston Seal
18525	Shaft for Piston Seal Replacement
19056	Pump Head Sealing Gasket, PTFE, 2/pk
19190	Piston Seal, GFP - for Organics
19762	Piston, zirconium
21220	Syringe, 2500 µL, O-ring Seal
22011	Check Valve Assembly, Inlet
22012	Check Valve Assembly, Outlet
22047	Purge Pump Assembly
22048	Purge Syringe
22049	Degasser Chamber
22050	Vacuum Pump

Part #	Description
22053	Pump Assembly
22198	Piston seal (GFP) Short Lip Type - for Toluene
Detectors and Accessories	
22062	RI-8320 Detector, dual flow, 2.5 µL cell
21792	UV-8320 Detector, 2 µL cell
14243	Window for UV Detector Cell, 2/pk
17545	Micro Cell for UV, 4 mm pathlength, 2 µL
17556	Seal for UV Cell Window
17558	Retaining Nut for UV Detector Cell
18445	Deuterium Lamp
Tubing/Fittings and Accessories	
06039	Tubing, SS, 1/16" OD × 0.4 mm ID × 2 m Length
06160	Nut, SS, 1/16", 5/pk
06163	Union, Internal, 1/16" OD × 0.35 mm ID, 5/pk
06167	Tubing, SS, 1/16" OD × 0.1 mm ID × 2 m Length
06168	Tubing, SS, 1/16" OD × 0.2 mm ID × 2 m Length
06169	Tubing, SS, 1/16" OD × 0.6 mm ID × 2 m Length
06170	Tubing, SS, 1/16" OD × 0.8 mm ID × 2 m Length
06171	Tubing, SS, 1/16" OD × 1.0 mm ID × 2 m Length
06176	Ferrule, 2-piece, SS, 1/8", 10/pk
06186	Column-to-Column Connector, 1/16" OD × 0.4 mm ID × 7 cm Length
06448	Tubing, Teflon, 3 mm OD × 2 mm ID × 2 m Length
06587	Tubing, Teflon, 2 mm OD × 1 mm ID × 2 m Length
06630	Tubing, SS, 1/16" OD × 0.25 mm ID × 2 m Length
06815	Union, Teflon, for 1/4" OD tubing
07055	Tee, SS, 1/16" OD, 1 mm bore
07337	Union, SS, 1/16" OD, 1 mm bore, 5/pk
07539	Tee, SS, 1/16" OD, 0.4 mm bore
07540	Union, SS, for 1/16" OD SS to 1/8” Teflon
08278	Tee, Teflon, 1/4 × 28 UNF threads
08290	File, double edged, to cut SS tubing
08299	Nut, Long, Rheodyne, SS, 1/16", 5/pk
08851	Tubing, Silicon, 4 mm OD × 2 mm ID × 2 m Length
08878	Nut, Male, SS, 1/8", 5/pk
13652	Tee, SS, 1/4 × 28 UNF, for 1/8” OD Teflon
13656	Union, for SS and Teflon Tubing, 1 mm bore
14182	Adapter for Teflon Tubing, 2 mm OD, 10/pk
14186	Adapter for Teflon Tubing, 1/8" OD, 10/pk
14188	Adapter for Teflon Tubing, 1/16" OD, 5/pk
14189	Adapter Fitting for Teflon Tubing (p/n 14182), 2 mm OD, 10/pk
14191	Adapter Fitting for Teflon Tubing (p/n 14186), 1/8" OD, 10/pk

Part #	Description
16180	Ferrule, SS, 1/16", 10/pk
16481	Tubing, Silicon, 2.5mm OD × 1.5 mm ID × 200 cm Length
16745	Adapter Fitting for Teflon Tubing (p/n 14188), 1/16" OD, 5/pk
17714	Frit, 10 µm pores, for p/n 18444
18184	Column-to-Column Connector, 1/16" OD × 0.2 mm ID × 7 cm
18444	Inline Frit Filter Holder, SS, for p/n 17714
22005	Union, Internal, SS, 1/16" OD Short
22010	Low Dead Volume Tubing Assembly
22055	Ferrule, PEEK, for 0.3 mm ID Tubing
23276	Tubing for Degasser, Santoprene, 5 mm OD × 3 mm ID × 100 cm Length, Replaces p/n 17747
Basic Maintenance Kits	
44959	Basic Maintenance Kit with Standard GFP Seals for EcoSEC GPC System - includes p/ns 19190(×2), 19056(×1), 16415(×1), 06574(×1), 19762(×2), 21220(×1), 16414(×1), 17714(×2)
44958	Basic Maintenance Kit (Aqueous) with PE Seals for EcoSEC GPC System - includes p/ns 18517(×2), 19056(×1), 16415(×1), 06574(×1), 19762(×2), 21220(×1), 16414(×1), 17714(×2)
44957	Basic Maintenance Kit (Toluene) with Modified GFP Seals for EcoSEC GPC System - includes p/ns 22198(×2), 19056(×1), 16415(×1), 06574(×1), 19762(×2), 21220(×1), 16414(×1), 17714(×2)

EcoSEC High Temperature GPC System: Optional Components and Replacement Parts

Tosoh Bioscience offers the following replacement parts and optional components for the EcoSEC High Temperature GPC System. In addition, preventative and basic maintenance kits are available for those parts that experience wear and tear due to normal usage.

Tosoh Bioscience offers extended service agreements and on-site periodic maintenance service calls. Please contact us for additional information or a quote for these services.

Part #	Description
Optional Components	
23801	Sample Prep System
23804	Column Switching Valve
22893	TSKgel HHR HT-RC Reference Column
Autosampler Accessories	
05462	Sample Loop, SS, 50 µL
05679	Sample Loop, SS, 100 µL
05464	Sample Loop, SS, 500 µL
23809	Sample Vial, glass,10 mL, 30/pk
18107	HT Sample Vial, Transparent, 10 mL, PTFE cover, 30/pk
23810	HT Aluminum Sheets, 30 mm square, 100/pk
23811	HT Stainless Steel Mesh, 26 µm, 50mm square,100/pk
23812	HT Stainless Steel Mesh 96 µm, 50mm square,100/pk
Pumps and Accessories	
23817	HT Needle
23818	HT Needle Joint
23819	HT Sampler Syringe Assembly
23815	HT Purge Syringe Assembly
23816	HT Pump
18524	Mold to Replace Piston Seal
18525	Shaft for Piston Seal Replacement
19056	Pump Head Sealing Gasket, PTFE, 2/pk
19190	Piston Seal, GFP
22011	Check Valve Assembly, Inlet
22012	Check Valve Assembly, Outlet
22049	Degasser Chamber
22050	Vacuum Pump
23848	HT Waste Liquid Bottle for Sampler

Part #	Description
Solvent Related Accessories	
06574	Mobile Phase Inlet Filter, SS, 5 µm pores
13166	Line Filter
18118	Moisture Trap for 3 L Solvent Reservoir
06814	Solvent Bottle End Plug, 1/4", 10/pk
Tubing/Fittings and Accessories	
06160	Nut, SS, 1/16", 5/pk
08851	Tubing, Silicon, 4 mm OD × 2 mm ID × 2 m Length
16180	Ferrule, SS, 1/16", 10/pk
16566	Fingertight Fitting, 2/pk
17714	10 µm pores, for p/n 18444
18444	Inline Frit Filter Holder, SS, for p/n 17714
Valves and Accessories	
18069	Rotor Seal for 6-way Valve, Polyimide (PI)
23826	HT Temperature Sensor for CO
16415	Rotor Seal for 6-way Valve
Basic Maintenance Kits	
44959HT	Basic Mainentance Kit with Standard GFP Seals for EcoSEC High Temperature GPC System - includes p/ns 19190(×2), 19056(×1), 06574(×1), 19762(×2), 21220(×1), 18069(×1), 17714(×2)

Read all about it!

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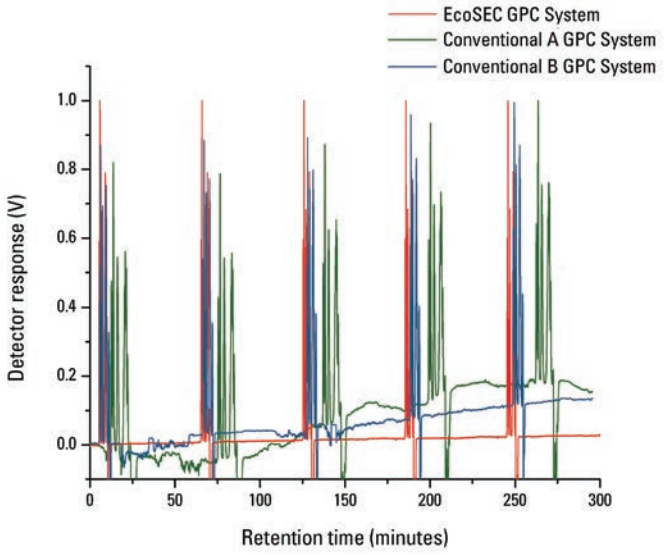

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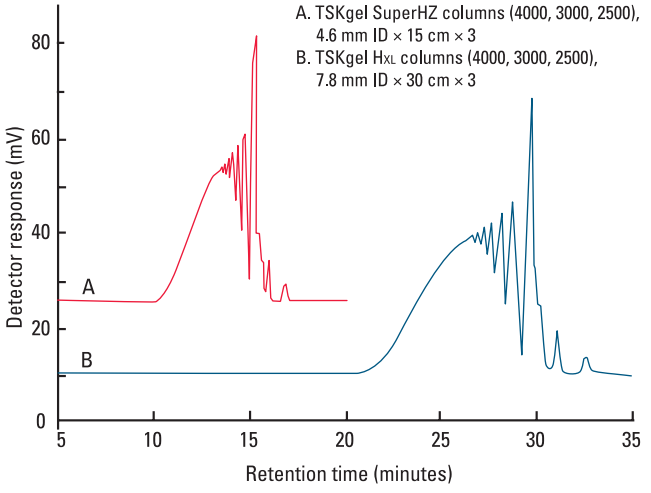

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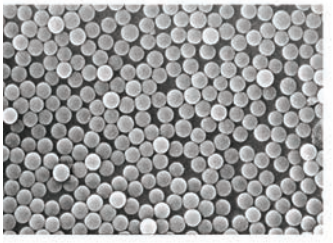

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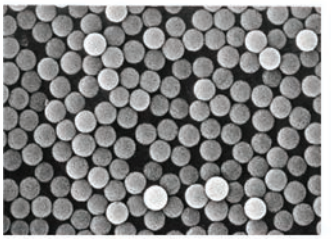

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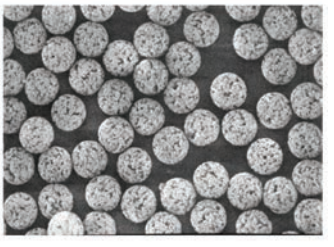

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