TOSOH

EcoSEC SEC/GPC System

TOSOH BIOSCIENCE

ABOUT US

WITH A GLOBAL PERSPECTIVE.

TOSOH BIOSCIENCE GmbH, Separations Business Unit, Stuttgart, is an acknowledged global leader in the field of bioseparations. Established as TosoHaas in 1987, the original joint venture between Tosoh Corporation of Japan and the Rohm and Haas Company, USA, has become synonymous with advanced products and quality support. In the year 2000, Tosoh Corporation acquired a 100% controlling interest changing the name to TOSOH BIOSEP. In the course of unifying all Tosoh affiliates, the new Brand Name Tosoh Bioscience evolved. Today, the two branches, Bioseparations and Diagnostics operate with the same name Tosoh Bioscience - Separations Business Unit and accordingly Diagnostics Business Unit. Tosoh manufacturing sites in Japan provide products to the sales and support subsidiaries in the U.S. and Europe, ensuring full global coverage. Tosoh has a long and successful history in manufacturing instruments for gel permeation chromatography (GPC) for the Asian market. Based on the wide experience in GPC instrument design and GPC column technology, Tosoh has developed the new EcoSEC system to meet the market demands for high throughput, semi micro GPC.

1
TOSOH BIOSCIENCE GMBH
ZETTACHRING 6
70567 STUTTGART
GERMANY

T · + 49 (0) 711 · 13257 · 0
F · + 49 (0) 711 · 13257 · 89
INFO.TBG@TOSOH.COM
WWW.TOSOHBIOSCIENCE.COM

2
TOSOH BIOSCIENCE LLC
3604 HORIZON DRIVE,
SUITE 100
KING OF PRUSSIA, PA 19406, USA

T · +1 484-805-1219
F · +1 610-272-3028
INFO.TBL@TOSOH.COM
WWW.TOSOHBIOSCIENCE.COM

3
TOSOH CORPORATION
3-8-2 SHIBA, MINATO-KU
TOKYO 105-8623
JAPAN

T · +81 3-5427-5118
F · +81 3-5427-5198
INFO@TOSOH.CO.JP
WWW.TOSOHBIOSCIENCE.COM

4
TOSOH BIOSCIENCE SHANGHAI CO. LTD
ROOM 301, PLAZA B,
NO. 1289 YI SHAN ROAD
XU HUI DISTRICT
SHANGHAI, 200233, CHINA
T · +86 21-3461-0856
F · +86 21-3461-0858
INFO@TOSOH.COM.CN
WWW.SEPARATIONS.ASIA.TOSOHBIOSCIENCE.COM

5
TOSOH ASIA PTE. LTD
63 MARKET STREET #10-03
SINGAPORE 048942,
SINGAPORE

T · +65 6226-5106
F · +65 6226-5215
INFO@TOSOH.COM.SG
WWW.SEPARATIONS.ASIA.TOSOHBIOSCIENCE.COM

TOSOH HISTORY

1935	FOUNDING OF TOYO SODA MANUFACTURING CO., LTD.
1936	OPERATION OF NANYO MANUFACTURING COMPLEX BEGINS
1971	SCIENTIFIC INSTRUMENTS DIVISION FORMED, FIRST GPC COLUMN USING TSKgel DEVELOPED BY TOSOH
1974	HIGH PERFORMANCE LIQUID CHROMATOGRAPHY COLUMN PLANT IS COMPLETED
1979	TOSOH DEVELOPS TOYOPEARL MEDIA
1983	TOSOH DEVELOPS HYDROPHOBIC INTERACTION MEDIA
1987	TOSOHAAS US OPERATIONS FORMED IN MONTGOMERYVILLE
1989	TOSOHAAS GMBH OPERATIONS FORMED IN STUTTGART
1995	TOSOH NANYO GEL FACILITY RECEIVES ISO 9001
2000	IN NOVEMBER FORMER TOSOHAAS US OPERATIONS BECOME TOSOH BIOSEP LLC, A 100% SUBSIDIARY OF TOSOH CORPORATION
2001	IN JANUARY FORMER TOSOHAAS GMBH EUROPEAN OPERATIONS BECOME TOSOH BIOSEP GMBH, A 100% SUBSIDIARY OF TOSOH CORPORATION
2002/ 2003	TOSOH CORPORATION ANNOUNCES THAT ALL TOSOH AFFILIATED SCIENTIFIC AND DIAGNOSTIC SYSTEM RELATED COMPANIES IN EUROPE, WILL BE UNIFIED UNDER THE NEW NAME TOSOH BIOSCIENCE
2008	ECOSEC , THE 7TH GENERATION GPC SYSTEM IS INTRODUCED GLOBALLY
2009	TOSOH BIOSCIENCE GMBH CELEBRATES ITS 20TH ANNIVERSARY IN STUTTGART
2010	TOSOH CELEBRATES ITS 75TH YEAR IN BUSINESS WITH THE OPENING OF FIVE NEW PLANTS, AND CONTINUED RAPID EXPANSION IN CHINA
2011	TOSOH BIOSCIENCE CELEBRATES 40 YEARS OF OPERATION
2012	TOSOH RELEASES FIRST TOYOPEARL MIXED-MODE RESIN TOYOPEARL MX-TRP-650M

TOSOH BIOSCIENCE

EcoSEC
GPC SYSTEM

ALL-IN-ONE-SYSTEM

INCREASED THROUGHPUT

- Semi-micro (4.6 mm ID) columns cut run time in half, doubling your throughput
- Autosampler for unattended operation
- Stable RI baseline in THF within 2 hours of startup

LOWER SOLVENT COST

- Low dead volume requires 85% less solvent compared to conventional GPC systems
- Superior performance
- Unmatched baseline stability due to unique dual flow RI design
- Excellent retention time reproducibility due to advanced temperature controlled pumps
- Day to day, system to system, location to location consistency

UNPARALLELED VERSATILITY

- Column switching valve
- Easy to use, intuitive software
- Optional UV detector
- Optional Viscometer and Multi-Angle Light Scattering detector
- Optional 3rd party software allows system control, data handling, and connectivity to external detectors and other lab systems

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.

EcoSEC GPC SYSTEM

TABLE 1

Component	Description	Benefit
SEMI-MICRO DESIGN	The EcoSEC GPC System is engineered for low volume by reduced tubing lengths, low dead volume flow cells, and small stroke pumps.	Maintains the efficiency of semi-micro columns (4.6. mm ID x 15 cm). The result is no loss of resolution at 1/6 the flow rate and 50% of the run time compared to conventional (7.8 mm ID x 30 cm) columns.
CONTROL PANEL	Allows the system to be controlled manually 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 capactiy (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 accomodate 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.	Open for measuring UV-absorbing polymers.
LIGHT SCATTERING DETECTOR (optional)	Up to 7 angle MALS design.	Absolute molar mass determination on the widest range of polymer sizes.
VISCOMETRY DETECTOR (optional)	Unique 80/20 split between sample and reference flow paths.	Reduces run times by up to 50% compared to 50/50 split flow path detectors.

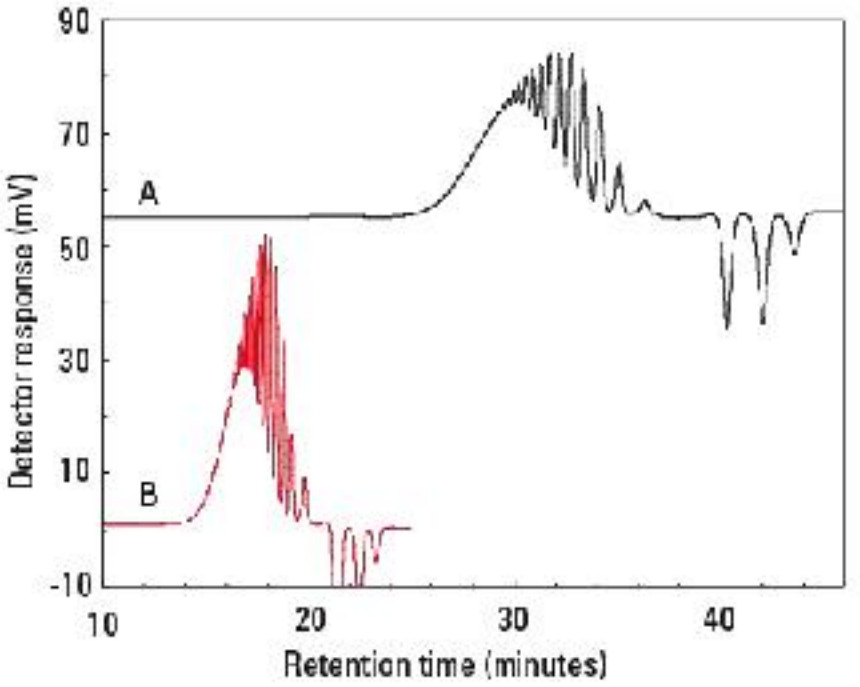
EcoSEC GPC SYSTEM

As shown in Figure 1, 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.

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.

Figure 2 shows an example of an oligomer (A-500) separation using four TSKgel SuperHZ2000 GPC columns in tandem performed using an EcoSEC GPC System and a competitive 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.

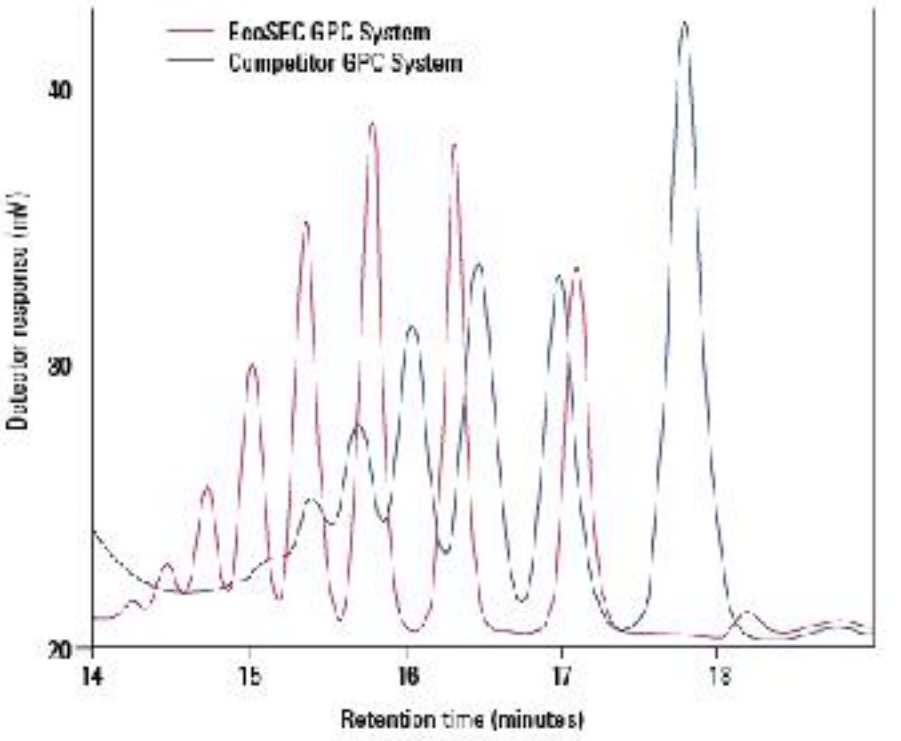
FIGURE 1



COMPARING SEMI-MICRO AND CONVENTIONAL GPC COLUMNS

Column: A. Conventional column, 7.8 mm ID x 30 cm x 4
B. TSKgel SuperMultiporeHZ-N, 4.6 mm ID x 15 cm x 4
Sample: poly(teramethylene ether glycol) (PTMEG 650), 10 µg/µL; Mobile phase: THF; Flow rate: A. 1.0 mL/min, B. 0.35 mL/min; Detection: RI; Temperature: 40°C; Injection vol.: A. 50 µL, B. 10 µL

FIGURE 2



COMPARISON OF RESOLUTION OF A SEMI-MICRO COLUMN RUN ON AN EcoSEC GPC SYSTEM AND A CONVENTIONAL GPC SYSTEM

Column: TSKgel SuperHZ2000, 4. 6mm ID x 15 cm x 4; Mobile phase: THF; Flow rate: 0.3 5mL/min; Detection: RI; Temperature: 40°C; Injection vol.: 10 µL; Sample: styrene oligomer (A-500), 0.2 mg/mL

TABLE 2

ANNUAL SOLVENT COST SAVING WITH SEMI-MICRO COLUMNS AND THE EcoSEC GPC SYSTEM

Solvent	Competitive GPC System	EcoSEC GPC System	Savings
THF cost (€ 30/L)/year	3.600	1.188	2.376
THF disposal cost (€ 30/L)/year	3.600	1.188	2.376
HFIP* cost (€ 1.500/L)/year	180.000	59.400	120.600
HFIP disposal cost (€ 60/L)/year	7.200	2.376	4.824

* THF: tetrahyrofuran; HFIP: hexafluoroisopropanol

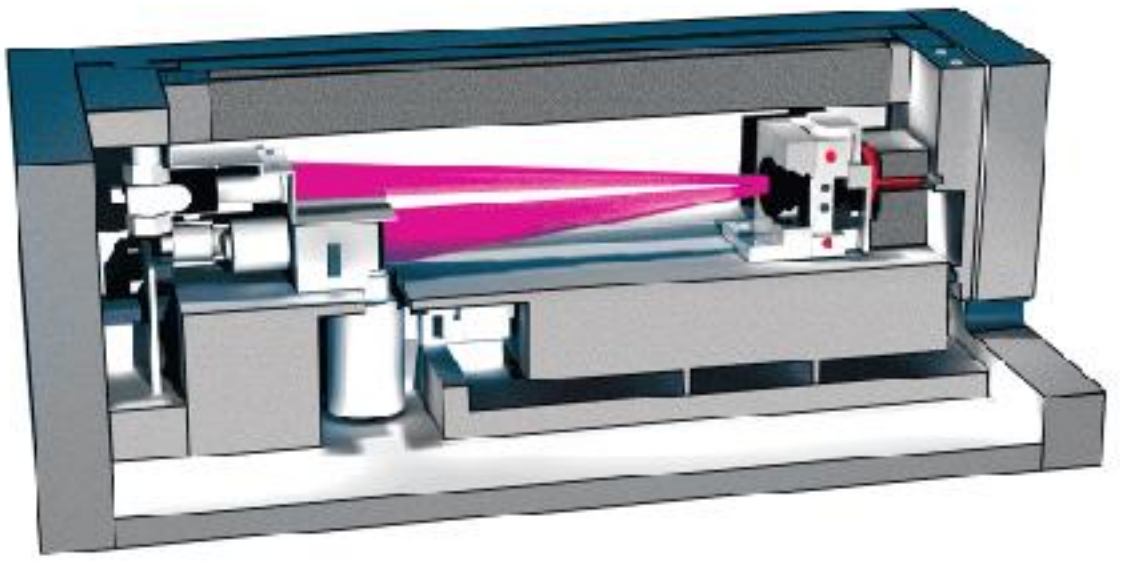
EcoSEC GPC SYSTEM

SUPERIOR PERFORMANCE

UNMATCHED BASELINE STABILITY

REFRACTOMETER

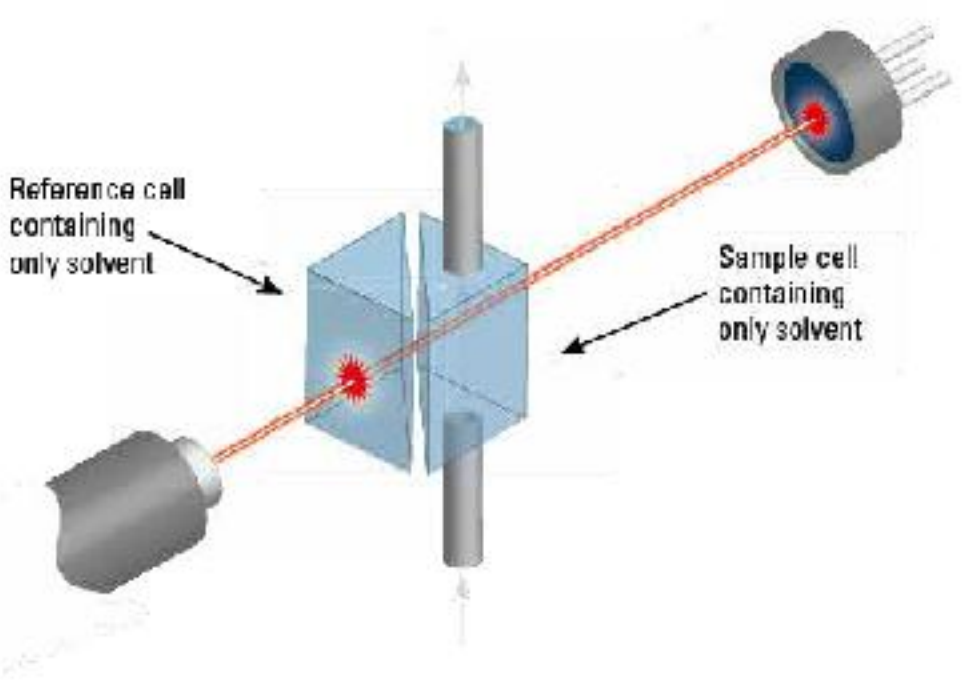
- Dual flow cell design
- Continous correction of RI baseline drift due to solvent instability
- Improved molar mass precision and accuracy
- Rapid baseline stability at startup



BASIC PRINCIPLE OF REFRACTIVE INDEX DETECTION

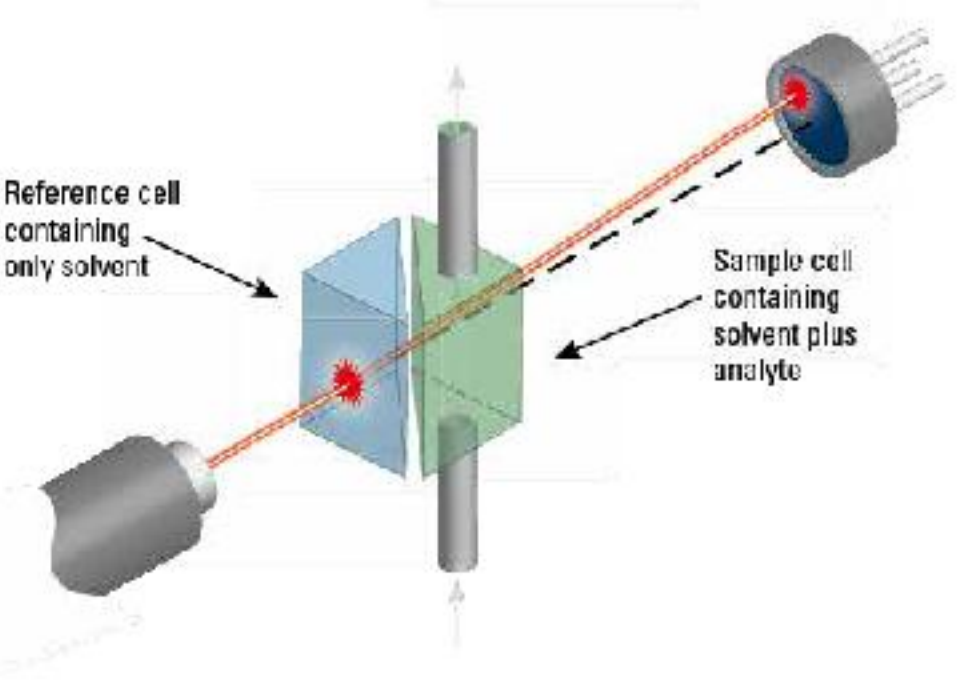
The most common type of differential refractive index detector is a deflection-type detector employing the principles of Snell's law of refraction. In this type of detector, light emitted from a source is transmitted through the flow cell of the RI detector and strikes a detector element. The flow cell is constructed in such a way that there are two separate, triangular pyramid shaped compartments (sides): (1) the reference side, consisting of stagnant pure solvent; and (2) the sample side, containing a flowing stream of analyte in the same solvent as in the reference side. As the light passes through the reference side into the sample side, the direction in which the light is travelling is changed, e.g., the path is bent. The amount of bending that takes place depends on the nature of the flow cell, the wavelength of the light being used, and the temperature and the concentration of analytes in the cell. The light then strikes a mirror and reflects back through the cell to two photodiodes mounted on a single chip. The two photodiodes will produce equal signals if the contents of the reference and sample sides have the same refractive indices as each other (Figure 3). In contrast, if the reference and sample sides have different refractive indices, a voltage difference will result between the photodiodes because the two photodiodes detect the difference in light intensity due the bending of the light beam, as shown in Figure 4. The difference in refractive indices between the two sides produces a voltage difference proportional to the concentration of the analyte in solution.

FIGURE 3



DEPICTION OF RI DETECTOR FLOW CELL WHEN THE CONTENTS OF THE REFERECNE AND SAMPLE SIDES HAVE THE SAME REFRACTIVE INDICES AS EACH OTHER

FIGURE 4



DEPICTION OF RI DETECTOR FLOW CELL WHEN THE CONTENTS OF THE REFERENCE AND SAMPLE SIDES HAVE DIFFERENT REFRACTIVE INDICES

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 5).

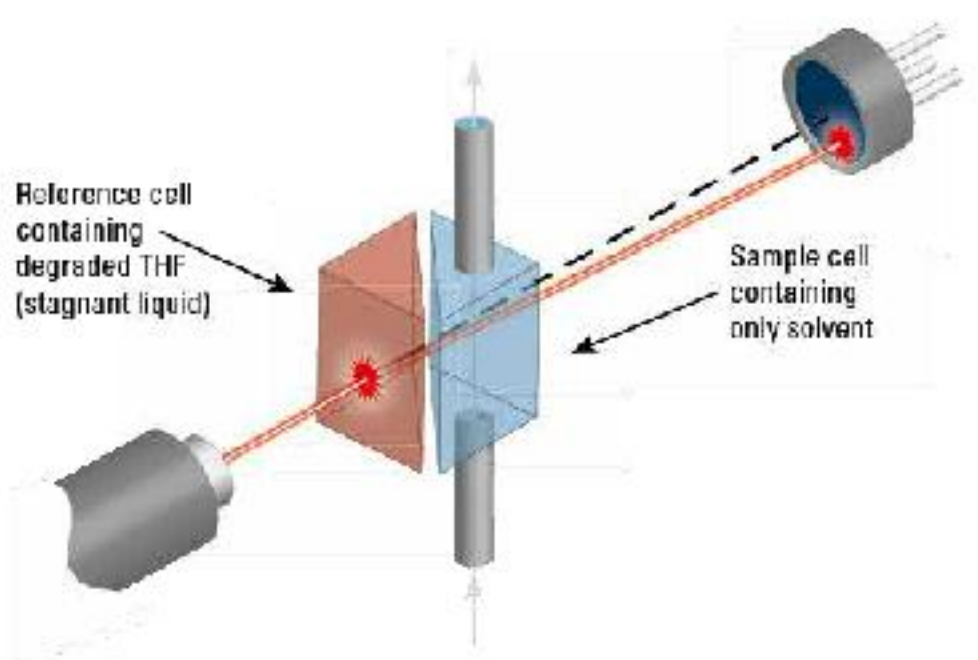
The unique dual flow design of the EcoSEC GPC System results in superb RI baseline stability and reduced RI baseline drift. In a conventional RI detector, over time, the refractive index of the stagnant pure solvent in the reference side will slowly change and the two photodiodes will no longer produce 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 6). 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.

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].

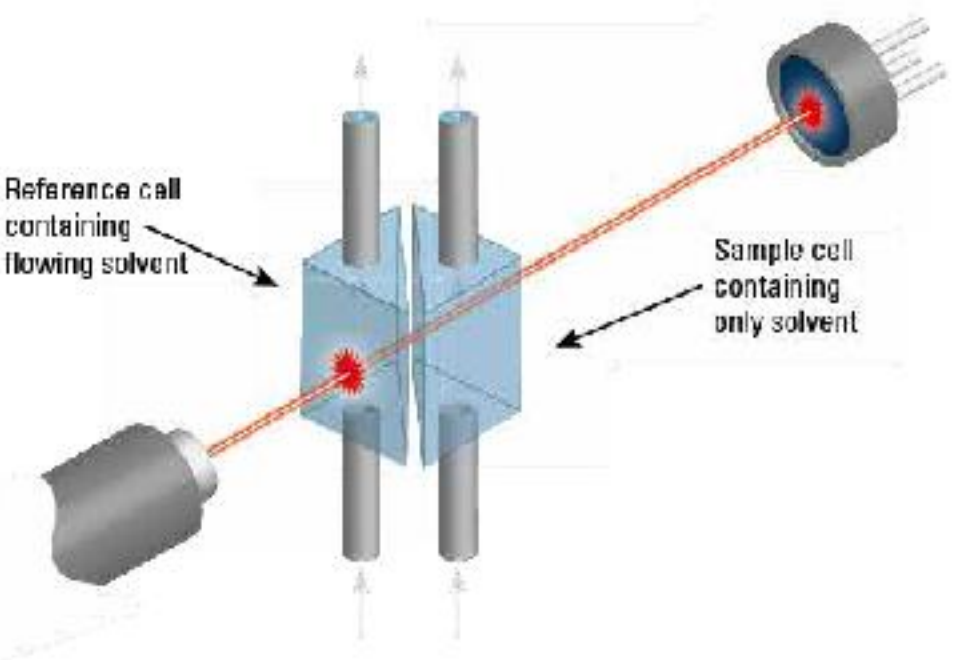
[1]Tcjir, W.J.; Rudin, A.; and Fyfe, C.A.; Journal of Polymer Science: Polymer Physics Edition, Volume 20, Issue 8, 1443-1451

FIGURE 5



DEPICTION OF DUAL FLOW RI DETECTOR IN THE EcoSEC SYSTEM, SHOWING THE COMPENSATION OF THE CHANGES IN REFRACTIVE INDEX OF THE SOLVENT OVER TIME

FIGURE 6



DEPICTION OF RI DETECTOR FLOW CELL SHOWING THE EFFECTS OF THF DEGRADATION IN THE STAGNANT REFERENCE SIDE OF A CONVENTIONAL GPC SYSTEM

EcoSEC GPC SYSTEM

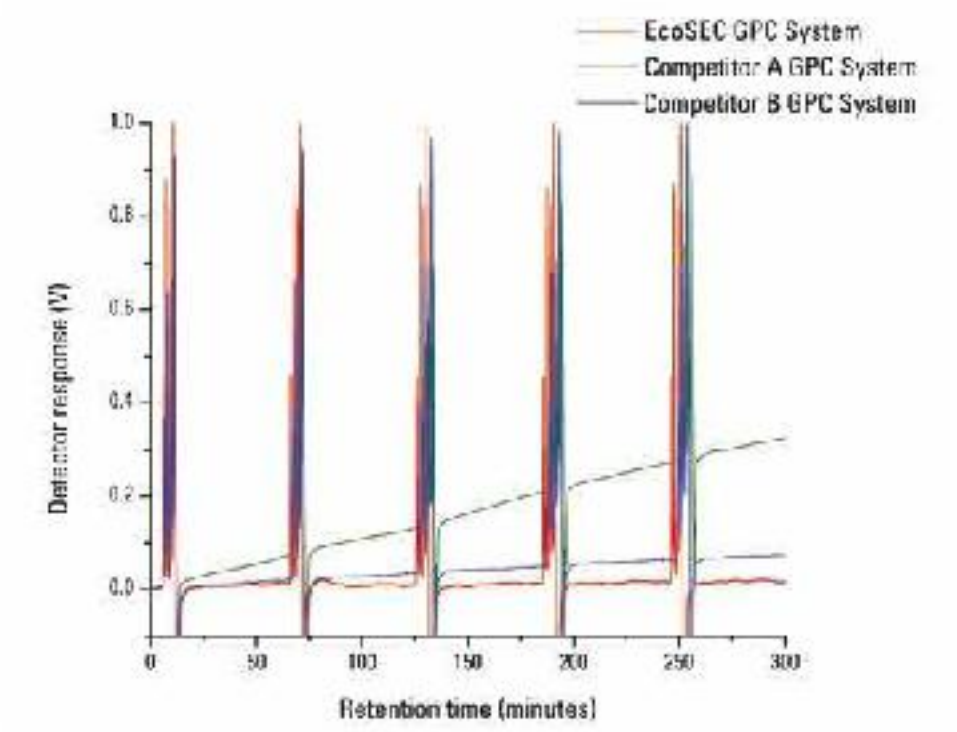
A study was done to demonstrate the superb baseline stability of the EcoSEC GPC System compared to that of two conventional GPC systems over a five hour time period. As shown in Figure 7, five consecutive injections of polystyrene standards with run times deliberately extended to one hour without auto 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.

COMPARISON OF BASELINE STABILITY

Five injections of dicyclohexyl phthalate were made on the EcoSEC GPC System and a conventional GPC instrument with a stagnant reference cell. The chromatograms were overlaid and are shown in Figures 8A and 8B.

With the EcoSEC GPC System, superposition of five consecutive chromatograms shows negligible baseline drift, as compared to the same experiment repeated with a competitor's GPC system having a non flow-through reference cell.

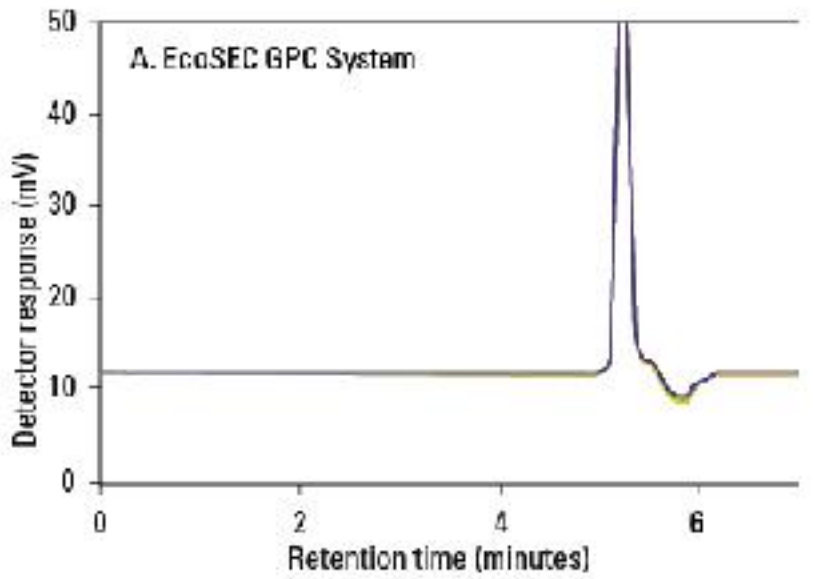
FIGURE 7

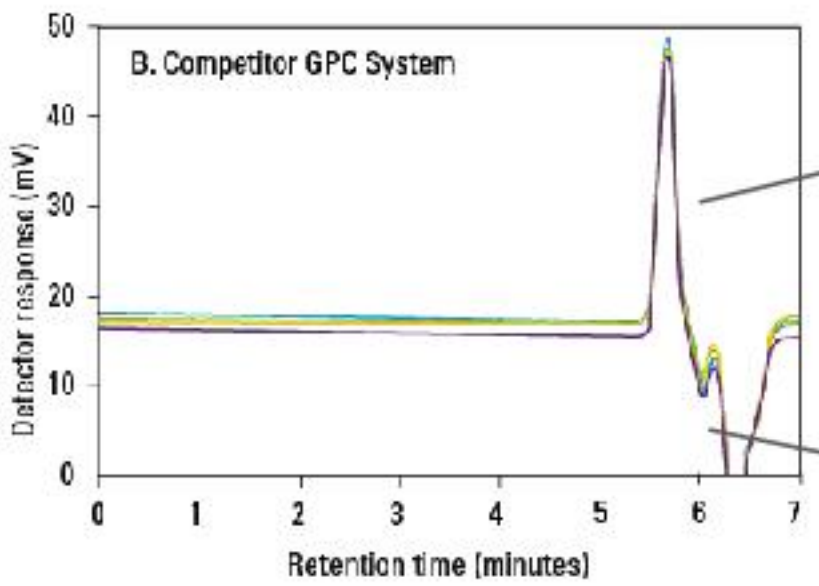


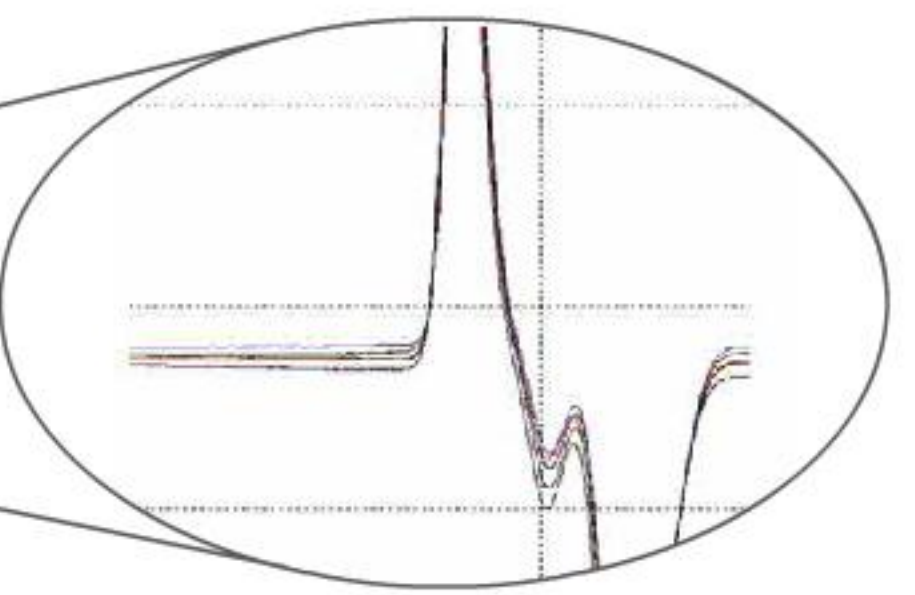
COMPARISON OF BASELINE DRIFT OF THE DUAL FLOW REFACTIVE INDEX DETECTOR OF THE EcoSEC GPC SYSTEM AND TWO CONVENTIONAL GPC SYSTEMS

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

FIGURE 8







COMPARISON OF BASELINE STABILITY

Column: TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm; Sample: dicyclohexyl phthalate; Mobile phase: THF; Flow rate: 0.35 mL/min; Detection: RI; Temperature: 40°C; Inj. volume: 10 µL

EcoSEC GPC SYSTEM

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 sixty 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 Figure 9 below. The elution volume drift of the EcoSEC GPC System was about 20% lower than that of the conventional GPC system.

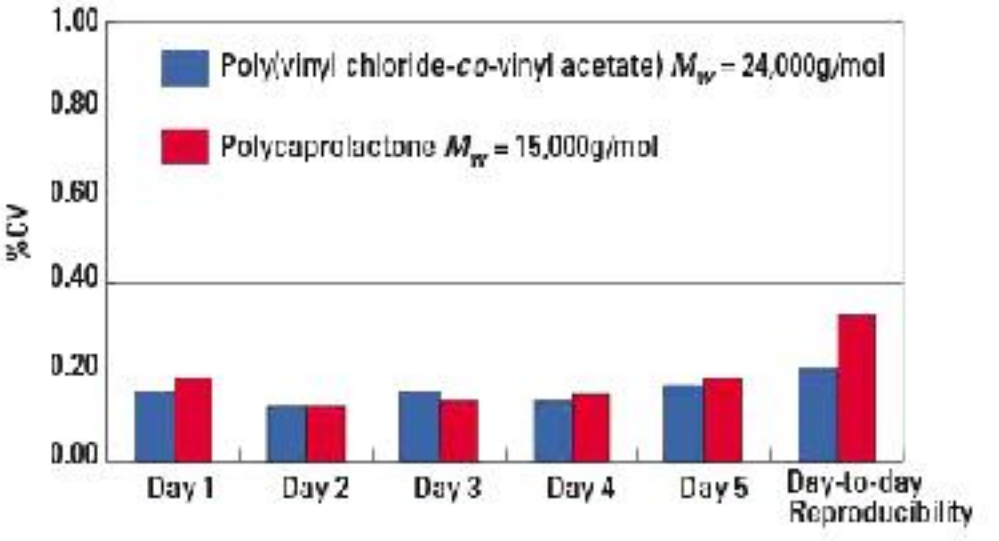
The results shown demonstrate that the engineering design concepts of the EcoSEC GPC System result in a high degree of reproducibility of elution times and molar mass determinations.

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.

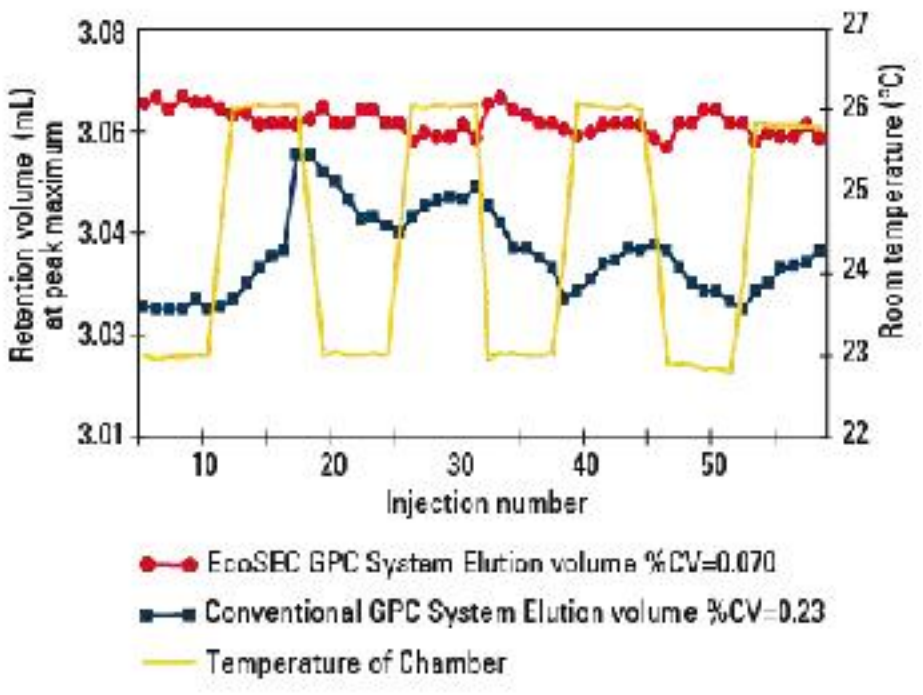
Figure 10 demonstrates the superiority of the EcoSEC GPC System for the determination of weight average molar masses. Figure 11 shows a comparison of M_w reproducibility for a sample injected 10 times a day for 5 days on the EcoSEC GPC System compared to a conventional GPC. The reproducibility of the EcoSEC GPC System was superior by a factor of 3 to that of the conventional GPC system.

FIGURE 10



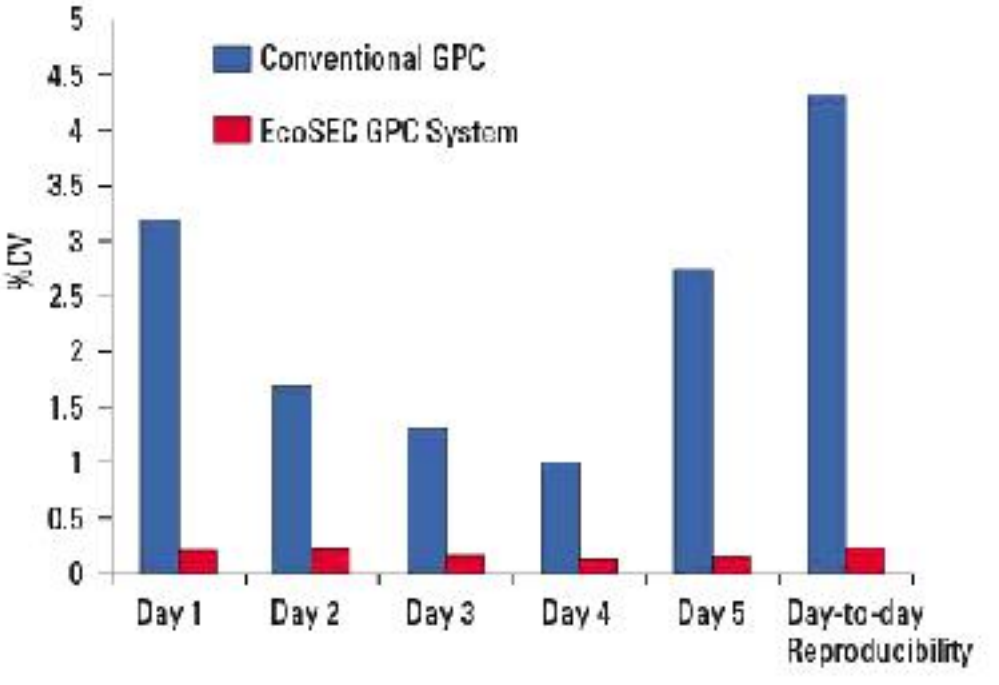
REPRODUCIBILITY OF M_w ANALYSIS
Column: TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm x 2; Sample: poly (vinyl chloride-co-vinyl acetate); Mobile phase: THF; Flow rate: 0.35 mL/min ; Temp.: 40 °C; Detection: RI; Inj.volume: 10 µL

FIGURE 9



MOBILE PHASE DELIVERY REPRODUCIBILITY WITH AMBIENT TEMPERATURE CHANGES
Column: TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm x 2; Sample: polystyrene standard; Mobile phase: THF; Flow rate: 0.35 mL/min, Detection: RI; Temperature: 40°C; Inj.volume: 10 µL
Temperature was cycled 23°C-26°C in the testing chamber.

FIGURE 11



COMPARING M_w REPRODUCIBILITY OF THE EcoSEC GPC SYSTEM AND A CONVENTIONAL GPC SYSTEM
Column: TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm x 2; Sample: poly (vinyl chloride-co-vinyl acetate); Mobile phase: THF; Flow rate: 0.35 mL/min ; Temp.: 40 °C; Detection: RI; Inj.volume: 10 µL

EcoSEC GPC SYSTEM

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 12. The high-precision of the EcoSEC GPC System results in minimal variation among instruments and from day-to-day.

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 3.

Reproducibility from system-to-system and location-to-location is exceptional with the EcoSEC GPC System. Coefficients of variations for all mass determinations 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 3

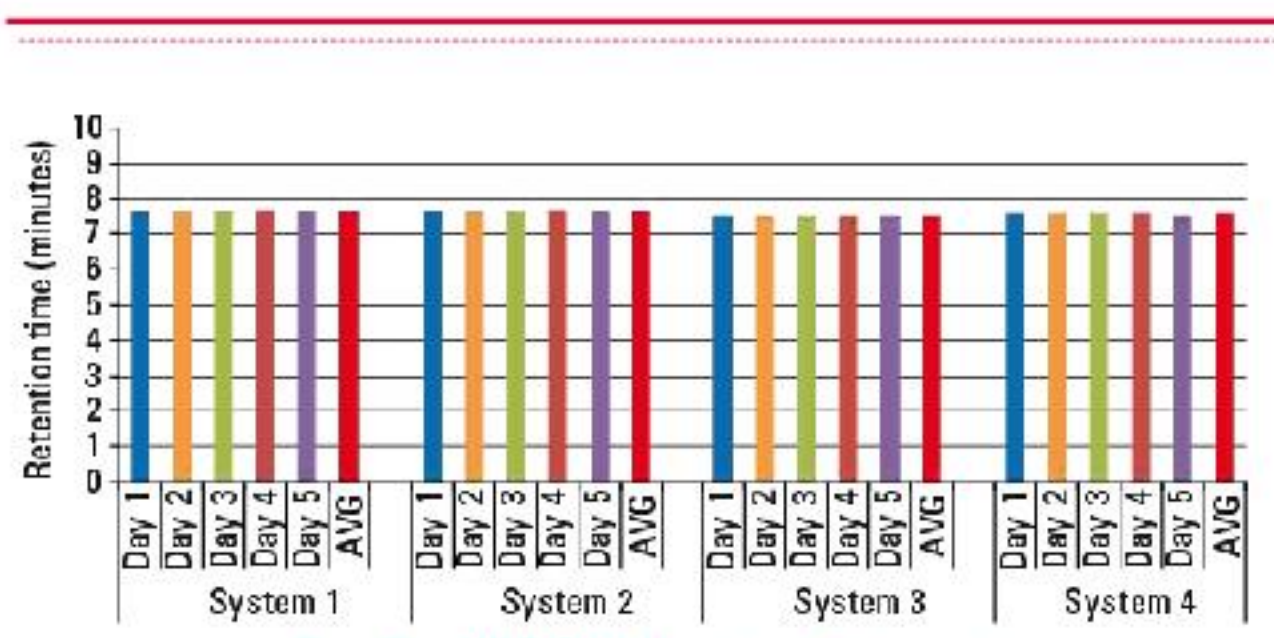
SITE-TO-SITE REPRODUCIBILITY

	M_n	M_w	W_z
Site A	13,800	29,800	53,700
Site B	13,700	29,900	54,300
Site C	13,600	29,800	53,200
Site D	13,700	30,200	54,100
Average	13,700	29,900	53,800
Deviation	70	160	420
%CV	0.52	0.55	0.78

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

Column: TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm x 2; Sample: poly (vinyl chloride-co-vinyl-acetate); Mobile phase: THF; Flow rate: 0.35 mL/min, Detection: RI; Temperature: 40°C; Inj.volume: 10 µL
Average of values measured with each instrument (n=10).

FIGURE 12



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

DAY-TO-DAY REPRODUCIBILITY
Column: TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm x 2; Sample: poly (vinyl chloride-co-vinyl acetate); Mobile phase: THF; Flow rate: 0.35 mL/min ; Temp.: 40 °C; Detection: RI; Inj.volume: 10 µL

EcoSEC GPC SYSTEM

RAPID COLUMN SWITCHING

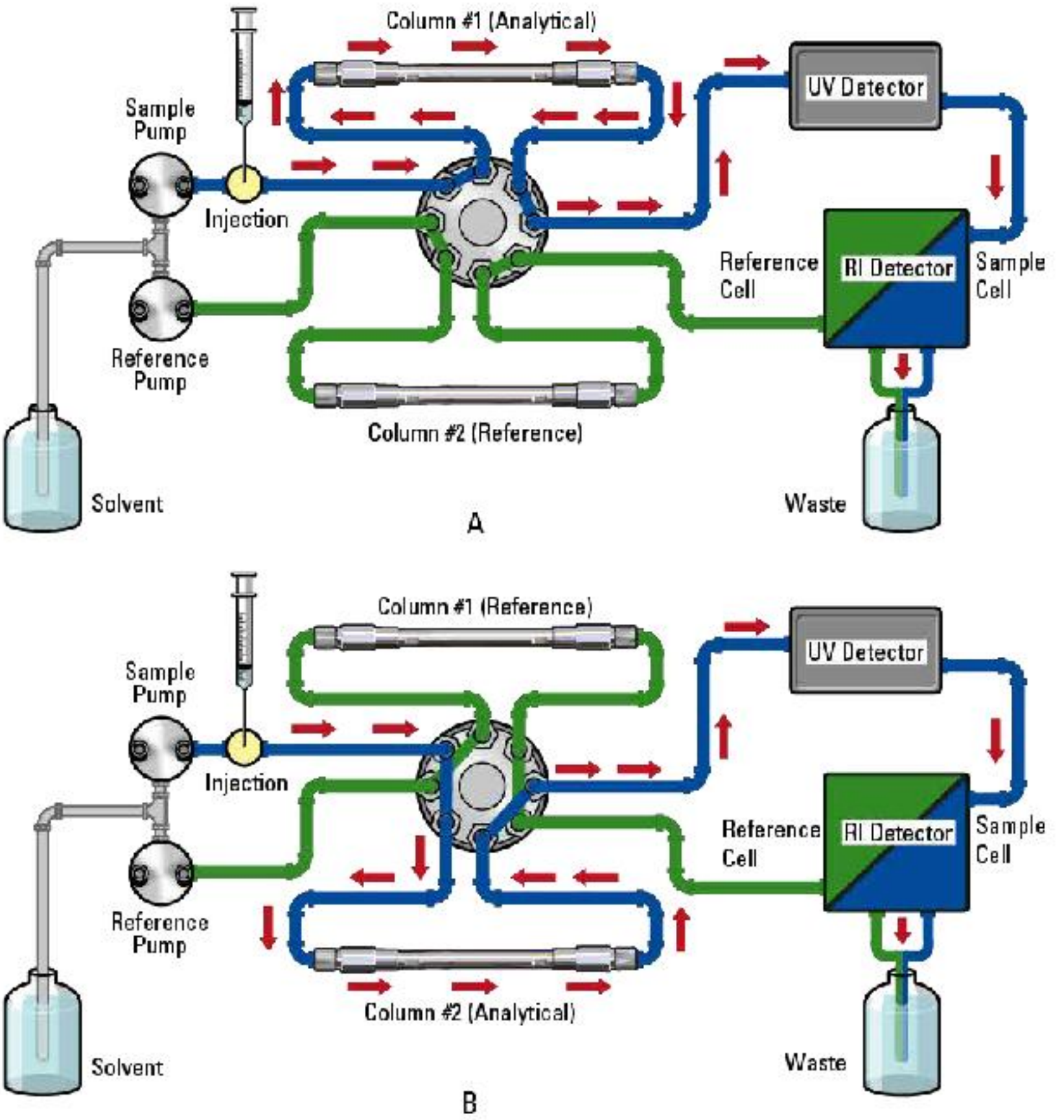
COLUMN SWITCHING VALVE

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

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 measurement column while column 1 will be in the flow path to the reference side of the RI detector flow cell.

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 13



A.) FLOW PATH WITH COLUMN 1 AS THE ANALYTICAL COLUMN B.) FLOW PATH WITH COLUMNS 2 AS THE ANALYTICAL COLUMN

EcoSEC GPC SYSTEM

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 1 x 10^{-7} RIU/hr 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 after 80 - 90 minutes. When a new column set is brought online using the column switching valve, the baseline stabilizes after 15 minutes.

(Experimental conditions: THF, 35°C, 0.35 mL/min, 20 minute warm-up at 50% flow rate). Figure 14 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 14

OVERLAY OF REFRACTIVE INDEX DETECTOR SIGNALS DURING EQUILIBRATION FOLLOWING A COLUMN CHANGE USING THE COLUMN SWITCHING VALVE (BLUE) AND WITHOU USE OF THE COLUMNS SWITCHING VALVE (RED)

CONFIGURATION OPTIONS

UV DETECTOR

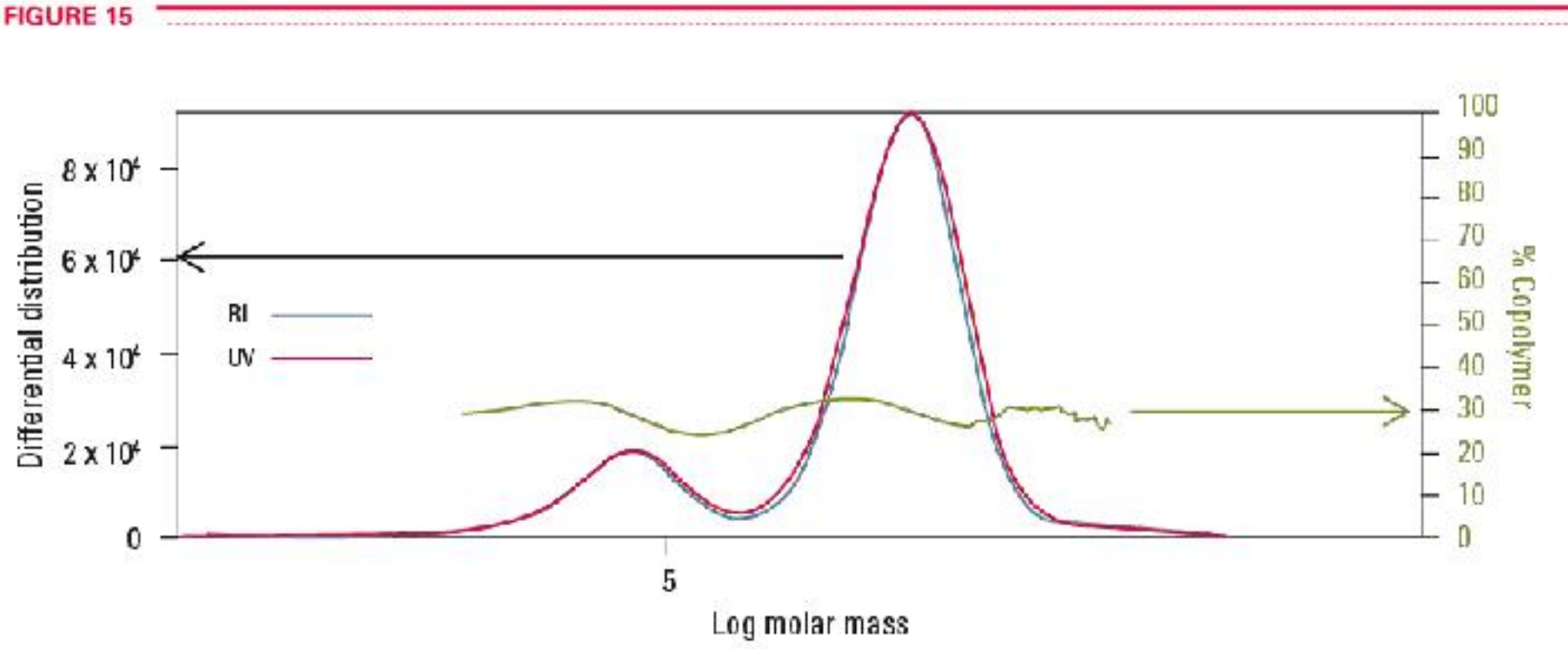
- Variable UV; 195 – 350 nm
- Semi-micro flow cell (2 µL)
- Factory installed option

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.

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 15, 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 15



COPOLYMER ANALYSIS OF POLYSTYRENE-b-POLYBUTENE

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

CONFIGURATION OPTIONS

TABLE 4

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	3×10^{-4} AU/h (254 nm, air in cell, response: 1.0 s)
Noise	2.5×10^{-5} AU/h (254 nm, air in cell, response: 1.0 s)
Flow cell volume	2 µL
Safety mechanism	Liquid leakage sensor; lighting time monitoring

EcoSEC GPC SYSTEM

ECOSEC 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	Air detection in wash solution and sample solution, Needle lock when the sample table is not loaded, Monitoring of 6-way and 4-way valve rotations

COLUMN OVEN	
Temperature range	Ambient plus 10°C to 60°C
Capacity	7.8 mm ID x 30 cm x 8 columns
Accuracy	+/- 0.5°C
Precision	+/- 0.2°C

RI DETECTOR	
Type	Bryce or 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} RI units (RIU)
Drift	1×10^{-7} RIU/h (THF, 1.0 mL/min)
Dynamic range	+/- 2.5×10^{-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) x 500 (D) x 550 (H) mm = 2.2′ x 1.6′ x 1.8′
Weight	95 kg = 210 lbs

APPLICATIONS

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 5, percent standard deviations are more than 10 x lower than values previously reported for polyamides in HFIP[2].

Percent relative standard deviation of the polydispersity index (PDI) ranged from 0.1 to 0.5%, permitting one to report PDIs within three significant figures. The high precision of the EcoSEC GPC System allows for the detailed study of polymerization reactions.

Sample chromatograms from 4 selected poly-ß-alkylalanoid samples run on an EcoSEC GPC System using two TSKgel GMH_{HR}-M, 5 µm, 4.6 mm ID x 15 cm columns are shown in Figure 16. 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 MW polymer component in the sample.

TABLE 5

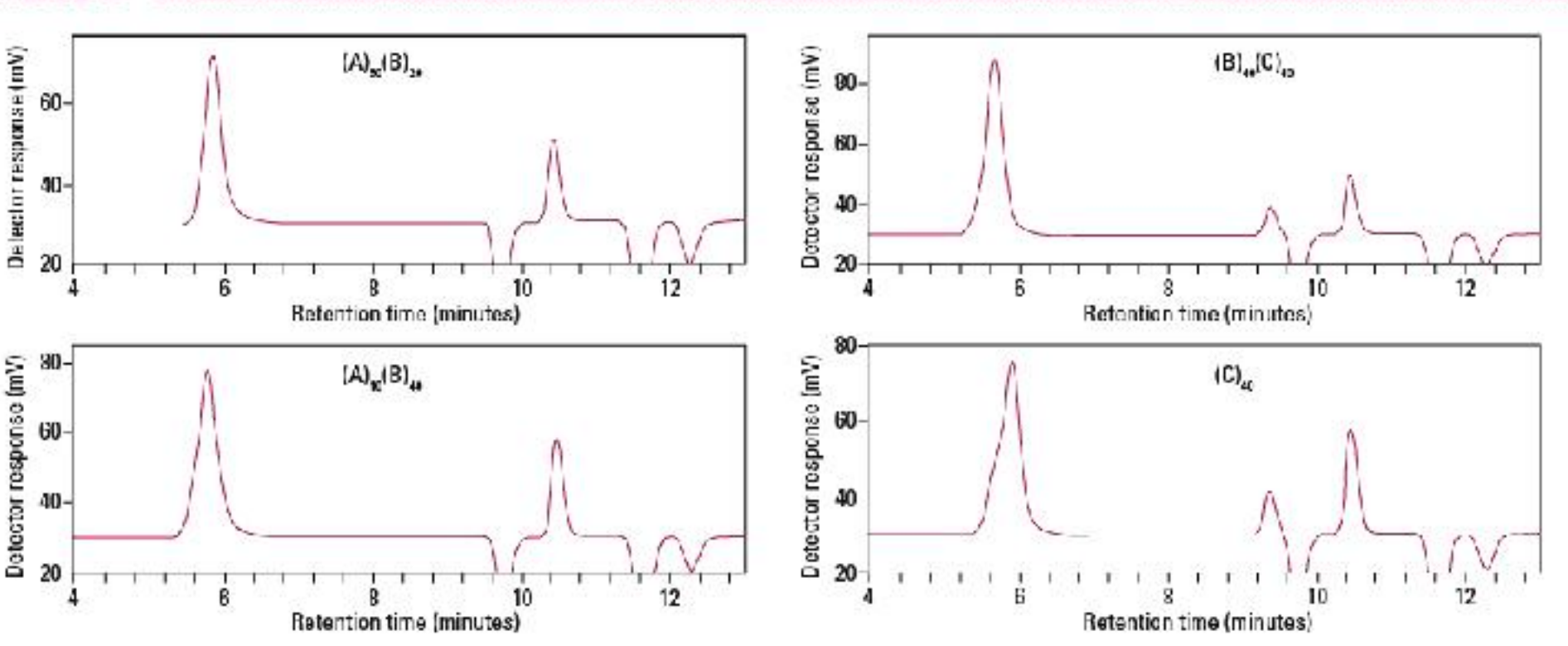
AVERAGED VALUES FROM THREE CONSECUTIVE INJECTIONS AND THE PERCENT RELATIVE STANDARD DEVIATIONS

Sample[a]	M_n[b]		M_w[b]		PDI[b]	
		Rel std dev		Rel std dev		Rel std dev
$(A)_{10}(B)_{40}$	26,500 ± 10	0.04%	30,300 ± 30	0.11%	1.14 ± 0.01	0.09%
$(A)_{60}(B)_{20}$	33,300 ± 170	0.52%	40,700 ± 28	0.07%	1.22 ± 0.01	0.50%
$(A)_{40}(B)_{40}$	48,700 ± 220	0.45%	60,900 ± 160	0.26%	1.25 ± 0.01	0.10%
$(C)_{40}$	30,100 ± 50	0.18%	36,400 ± 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.

FIGURE 16



POLY-β-ALKYLALANOID SAMPLES

TSKgel GMH_{HR}-M, 5 µm, 4.6 mm ID x 15 cm x 2 packed in HFIP; Samples: selection of poly-β-alkylalanoid samples

Mobile Phase: HFIP containing 5 mmol/L sodium trifluoroacetate; Flow rate: 0.35 mL/min; Detection: RI; Temperature: 40°C Inj. vol.: 10 µL

[2] 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

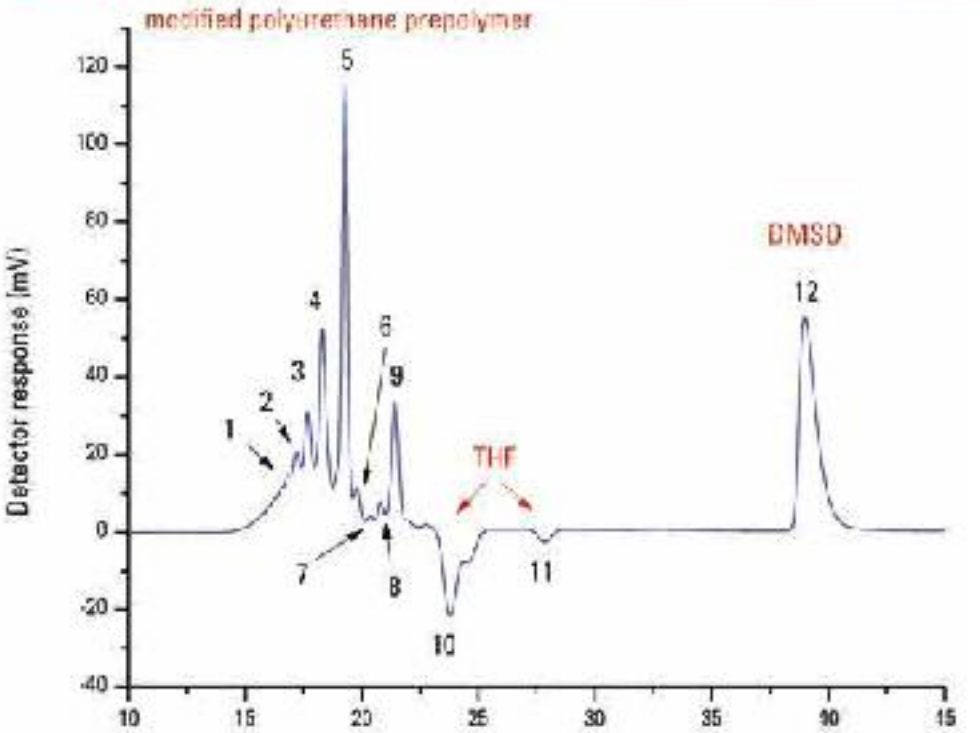
APPLICATIONS

MODIFIED POLYURETHANE PREPOLYMER ANALYSIS

An EcoSEC GPC System was used to analyze an isocyanate modified polyurethane prepolymer with residual DMSO. As shown in Figure 17, separation of the sample by GPC results in ten positive chromatographic peaks and two negative chromatographic peaks. The first nine chromatographic peaks correspond to components of the modified polyurethane prepolymer while the two negative chromatographic peaks are indicative of the solvent, THF. The latest eluting peak is a result of the residual DMSO present in the sample and is retained by a non-SEC retention mechanism, as it elutes after the void volume of the column.

The molar mass averages M_n, M_w, and M_z of the sample, given in Table 6, were determined via a polystyrene relative calibration curve. The sample was found to have a weight average molar mass M_w ranging from 4,199 to 178 g/mol. The polydispersity index (PDI) shown in Table 6 for the entire sample, e.g., peaks 1 through 9, was 2.26 while the individual components of the polyurethane prepolymer had PDI values ranging from 1.01 to 1.09. From the PDI values it can be concluded that collectively the sample is polydispersed with respect to molar mass, but the nine visible components within the sample are virtually monodispersed with respect to molar mass.

FIGURE 17



MODIFIED POLYURETHANE PREPOLYMER SAMPLE
Column: TSKgel SuperH3000, 6.0 mm ID x 15 cm x 2; Sample: modified polyurethane prepolymer, 10 mg/mL ; Mobile phase: THF; Flow rate: 0.30 mL/min; Detector: RI; Temperature: 35°C; Injection volume: 20 µL

TABLE 6

MOLAR MASS AVERAGES AND POLYDISPERSITY INDEX FOR MODIFIED POLYURETHANE PREPOLYMER SAMPLE IN THF AT 0.3 mL/min

Peak	M_n	M_w	M_z	PDI[a]
1	4,199 ± 46[b]	4,606 ± 67	5,214 ± 109	1.09 ± 0.01
2	2,643 ± 19	2,655 ± 18	2,667 ± 18	1.01 ± 0.01
3	2,011 ± 16	2,024 ± 16	2,038 ± 16	1.01 ± 0.01
4	1,387 ± 10	1,403 ± 10	1,418 ± 10	1.01 ± 0.01
5	798 ± 4	808 ± 5	817 ± 5	1.01 ± 0.01
6	551 ± 9	554 ± 9	557 ± 9	1.01 ± 0.01
7	391 ± 9	394 ± 9	397 ± 10	1.01 ± 0.01
8	278 ± 3	280 ± 3	282 ± 3	1.01 ± 0.01
9	178 ± 1	181 ± 1	183 ± 1	1.01 ± 0.01
All	676 ± 9	1,531 ± 31	2,873 ± 83	2.26 ± 0.02

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

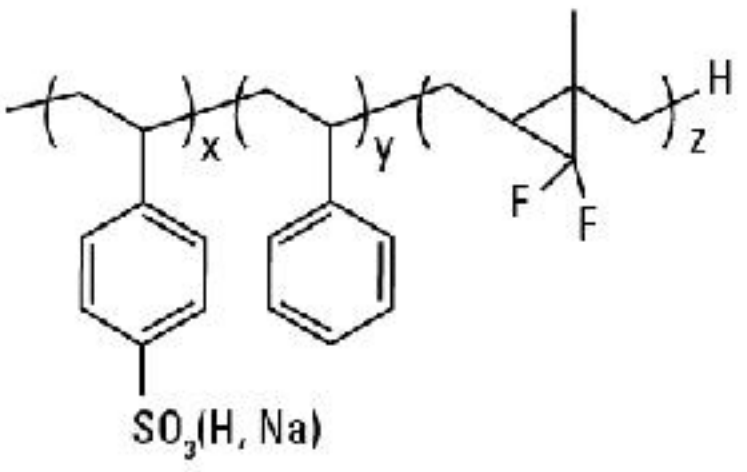
APPLICATIONS

ANALYSIS OF STYRENE AND ISOPRENE BLOCK COPOLYMERS BEFORE AND AFTER FLUORINATION

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 (sPS-b-fPI), Figure 18, were synthesized by anionic polymerization followed by fluorination and sulfonation[3].

The EcoSEC GPC System, equipped with TSKgel SuperMultipore® columns, was then used to determine the number-average molar mass, M_n, and the polydispersity index, PDI, of sPS-b-fPI, as well as that of the precursor polymer (PS-b-PI), Table 7. As seen in Figure 19, complete analysis of sPS-b-fPI was obtained in less than 10 minutes with excellent resolution using the EcoSEC GPC System.

FIGURE 18



STRUCTURE OF SULFONATED POLYSTYRENE-b-FLUORINATED POLYISOPRENE (sPS-b-FPI)

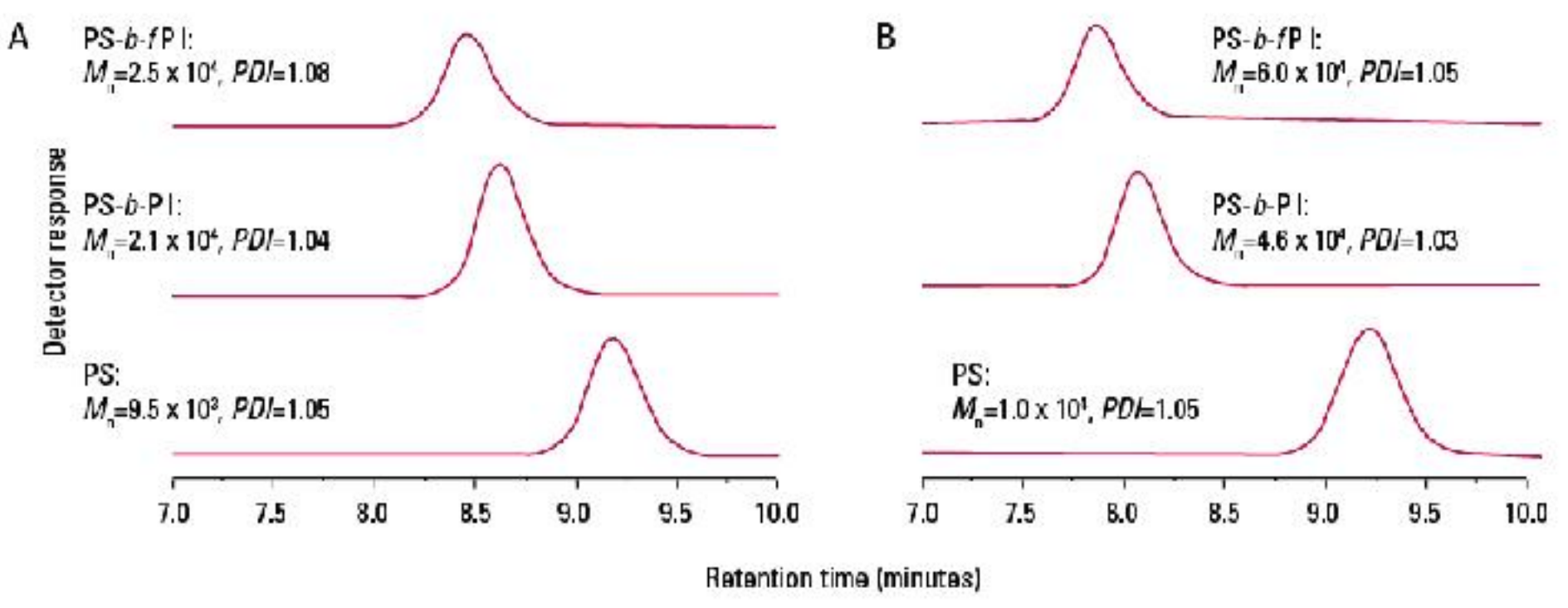
TABLE 7

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		sPS-b-fPI	
Series[a]	M_n(SEC)	PDI	M_n(SEC)	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 19



SULFONATED POLYSTYRENE-b-FLUORINATED POLYISOPRENE PRECURSOR SAMPLES

TSKgel SuperMultiporeHZ-M, 4.6 mm ID x 15 cm x 1; Samples: A. series 1, table 3 B, series 2, table 3; Mobile Phase: THF; Flow rate: 0.35 mL/min; Detection: RI; Temperature: 35°C Inj. vol.: 20 µL

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

TSKgel COLUMNS AND STANDARDS

TSKgel GPC COLUMNS

SEMI-MICRO 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.

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

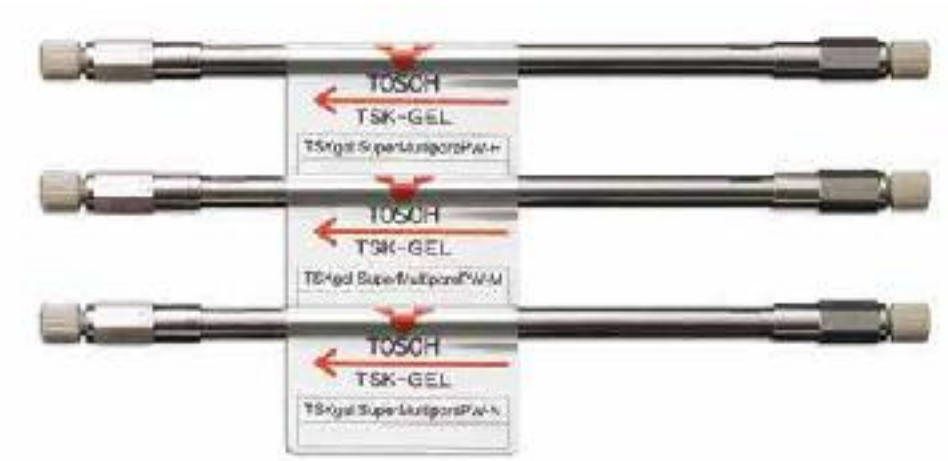
Semi-micro columns* (4.6 or 6.0 mm ID x 15 cm)

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

Conventional columns (7.8 mm ID x 30 cm)

- TSKgel H_{XL} columns for ultra-low adsorption
- TSKgel H_{HR} columns for low adsorption; max temp. 140°C

*EcoSEC GPC System recommended columns



GPC COLUMNS FOR POLYMERS SOLUBLE IN POLAR ORGANIC SOLVENTS

Semi-micro columns* (6.0 mm ID x 15 cm)

- TSKgel SuperAW columns

Conventional columns (7.8 mm ID x 30 cm)

- TSKgel Alpha columns

*EcoSEC GPC System recommended columns

GPC COLUMNS FOR POLYMERS SOLUBLE IN AQUEOUS SOLVENTS

Semi-micro columns* (6.0 mm ID x 15 cm)

- TSKgel SuperMultiporePW columns

*EcoSEC GPC System recommended columns

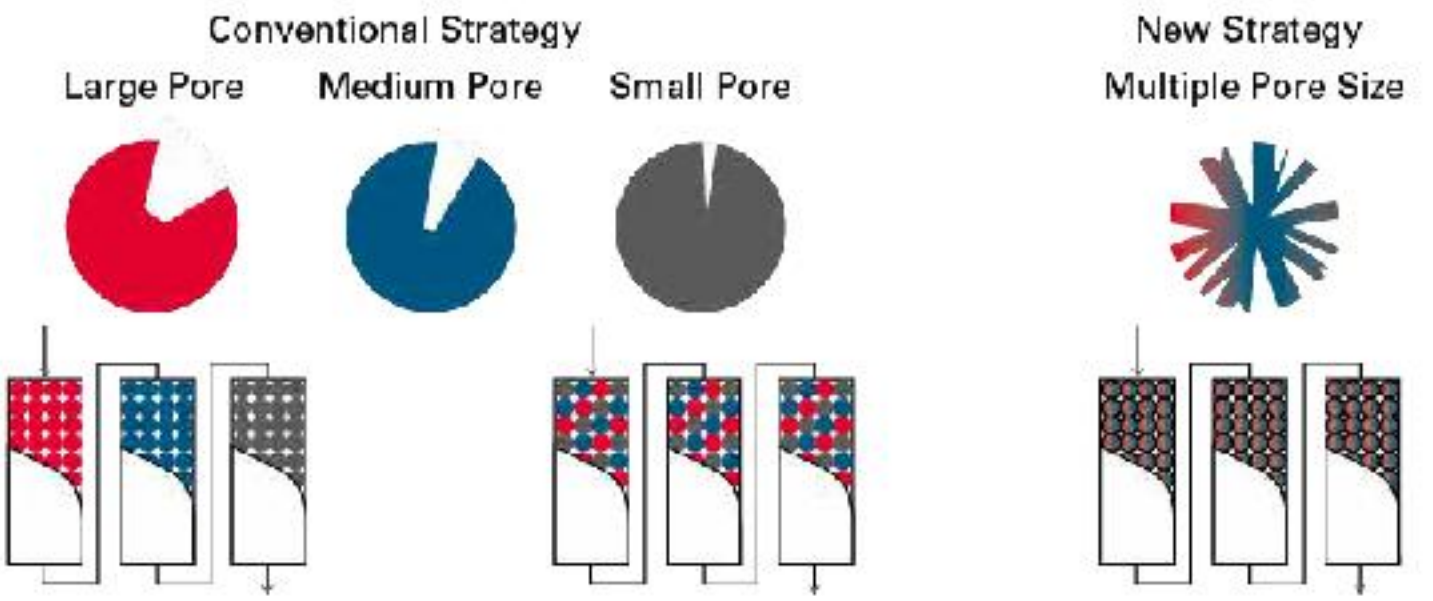
TSKgel COLUMNS AND STANDARDS

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 is to use multiple columns of different pore sizes linked together in series. A second is to use a column packed with a mixed-bed resin of different pore sizes at an optimized mix ratio. However, problems can occur with both of these methods, which include distortion of the chromatogram or deviations between the actual calibration curve and the calibration curve approximated from data obtained from the molar mass standards.

As is shown in Figure 20, a novel approach to solve this problem was developed by Tosoh scientists and is incorporated in TSKgel SuperMultiporeHZ columns. These columns are packed with small particles of uniform size 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. Their small ID (4.6 mm ID) and length (15 cm) reduces solvent consumption, results in quick run times, and offers high throughput capabilities.

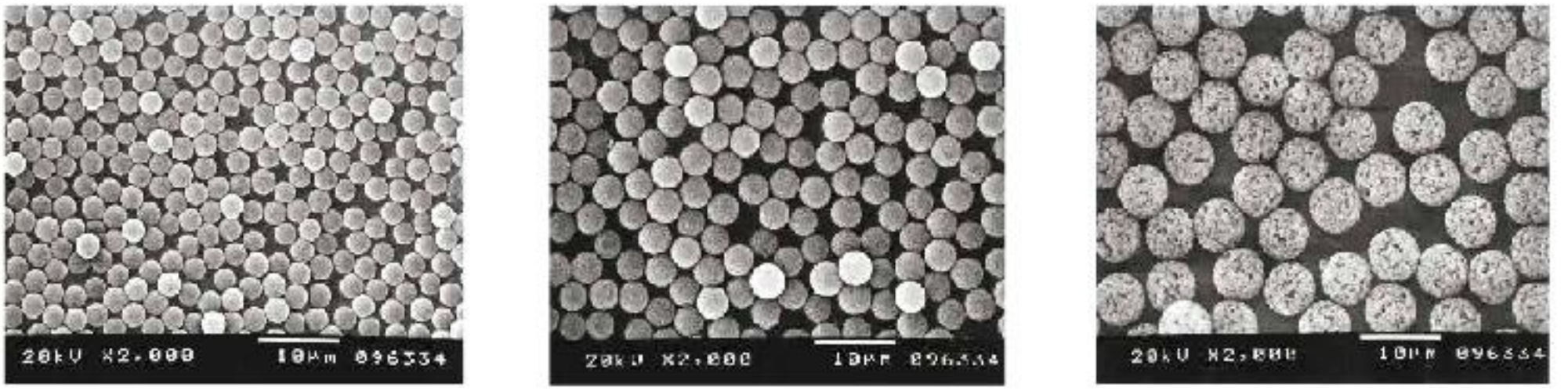
FIGURE 20



GRAPHICAL REPRESENTATIONS ILLUSTRATE THE MULTI-PORE PARTICLE SYNTHESIS TECHNOLOGY

TSKgel COLUMNS AND STANDARDS

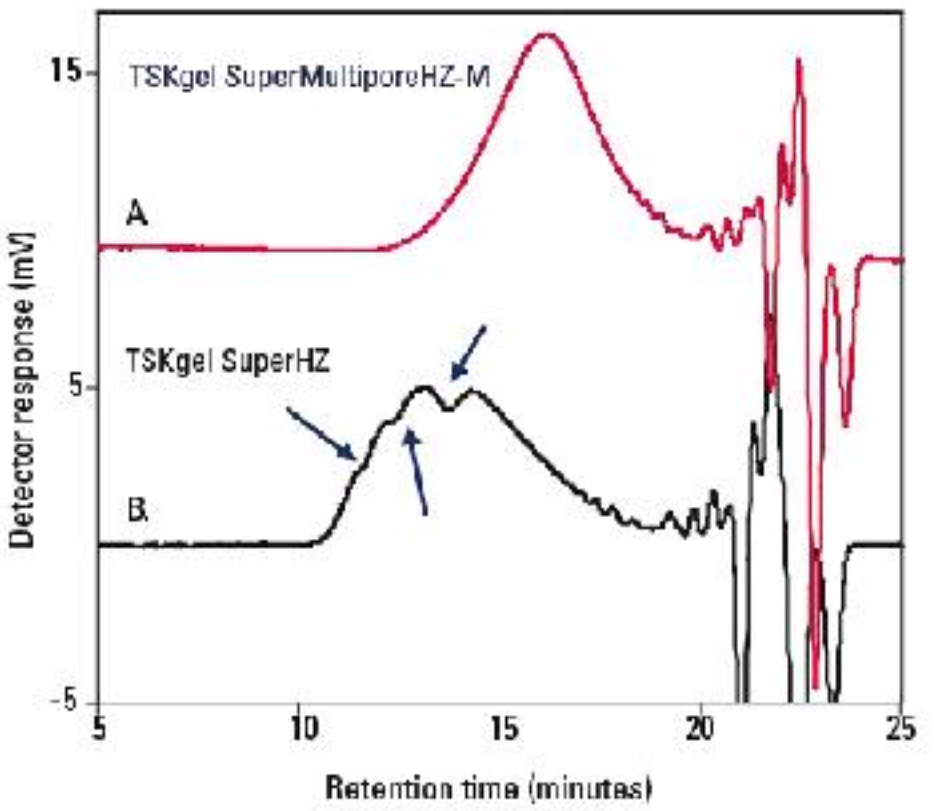
FIGURE 21



TSKgel SuperMultiporeHZ COLUMNS PACKED WITH MONODISPERSE PARTICLES

Figure 21 shows the monodispersity of the particle size distribution of TSKgel SuperMultiporeHZ columns compared to a conventional mixed-bed column.

FIGURE 22



COMPARISON OF TSKgel SuperMultiporeHZ-M AND TSKgel SuperHZ COLUMNS FOR THE SEPARATION OF ACRYLIC RESIN

Figure 22 demonstrates that inflection points are no longer observed with columns packed from particles prepared by multi-pore technology.

TSKgel COLUMNS AND STANDARDS

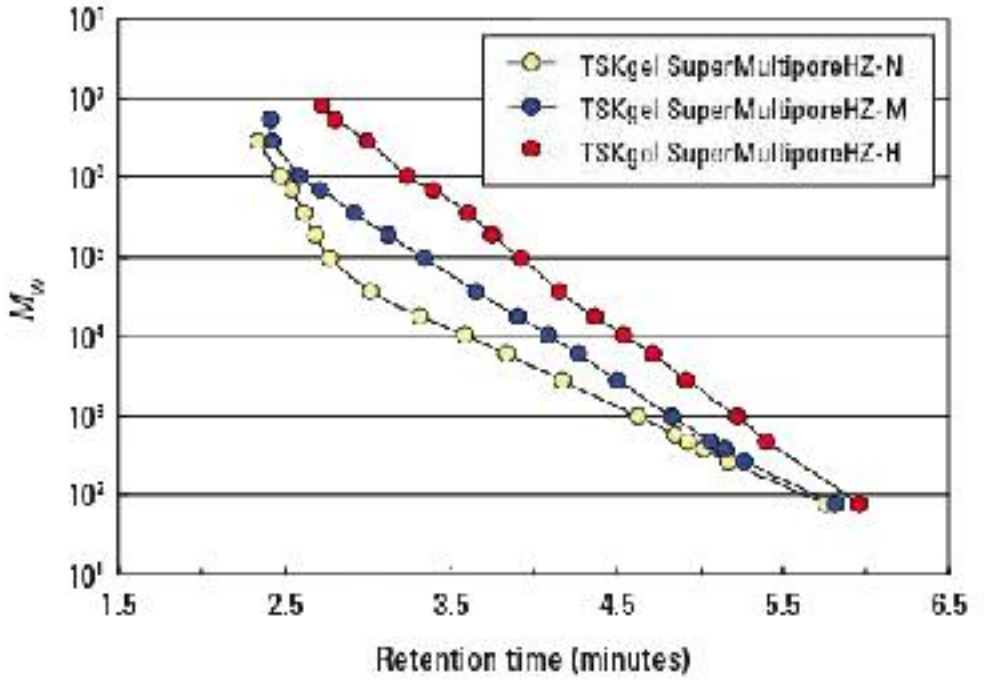
TSKgel SuperMultiporeHZ COLUMNS

TSKgel SuperMultiporeHZ columns combine ultra high performance GPC columns with low dead volume (4.6 mm ID x 15 cm) and feature multi-pore particles with a wide pore size distribution. The use of the multi-pore technology ensures that the calibration curves have excellent linearity. There are three columns available within the TSKgel SuperMultiporeHZ columns, each with a different particle size, separation range, and exclusion limit. These columns can separate and characterize polymers within a wide molar mass range.

As demonstrated in Figure 23, the TSKgel SuperMultiporeHZ columns have a highly linear calibration curve with a shallow slope, which indicates excellent reproducibility across various average molar mass values.

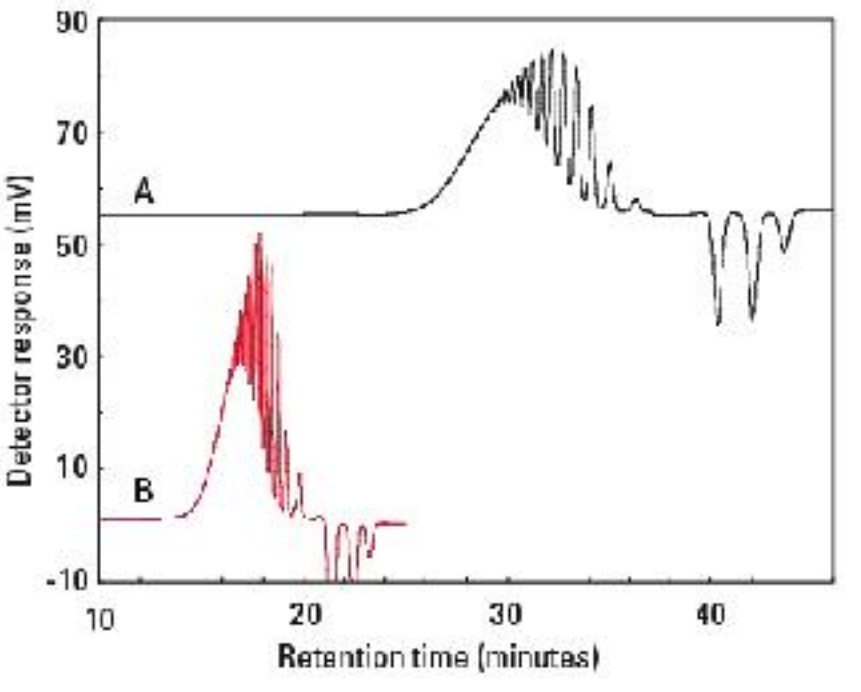
The TSKgel SuperMultiporeHZ-N columns provide the same or higher resolution at a much shorter analysis time than multiple columns linked together, as shown in Figure 5.

FIGURE 23



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

FIGURE 24



COMPARING SEMI-MICRO AND CONVENTIONAL GPC COLUMNS
Columns: A. Conventional columns, 7.8 mm ID x 30 cm x 4; B. TSKgel SuperMultiporeHZ-N, 4.6 mm ID x 15 cm x 4; Sample: poly(teramethylene ether glycol); (PTMEG 650), 10 µg/µL; Mobile phase: THF; Flow rate: A. 1.0 mL/min B. 0.35 mL/min; Detection: RI; Temperature: 40°C; Injection vol.: A. 50 µL B. 10 µL;

TSKgel COLUMNS AND STANDARDS

TABLE 8

ORDERING INFORMATION

Part #	Description	Particle size (µm)	Exclusion limit (polystyrene)	Theoretical plates/15 cm	Column dimensions
21815	TSKgel SuperMultiporeHZ-N	3	1.2 x 10^5 Da	>20,000	4.6 mm ID x 15 cm
21488	TSKgel SuperMultiporeHZ-M	4	2 x 10^4 Da	>16,000	4.6 mm ID x 15 cm
21885	TSKgel SuperMultiporeHZ-H	6	4 x 10^3 Da	>11,000	4.6 mm ID x 15 cm
21886	TSKgel SuperMultiporeHZ-H Guardcolumn	6	N/A	N/A	4.6 mm ID x 2 cm
21489	TSKgel SuperMultiporeHZ-M Guardcolumn	4	N/A	N/A	4.6 mm ID x 2 cm
21816	TSKgel SuperMultiporeHZ-N Guardcolumn	3	N/A	N/A	4.6 mm ID x 2 cm

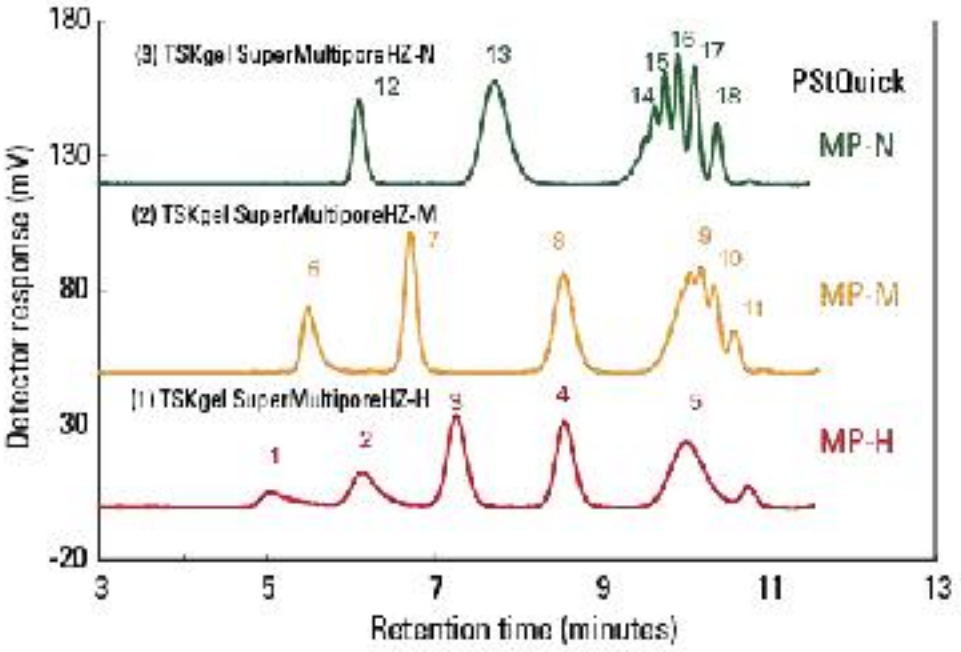
TSKgel COLUMNS AND STANDARDS

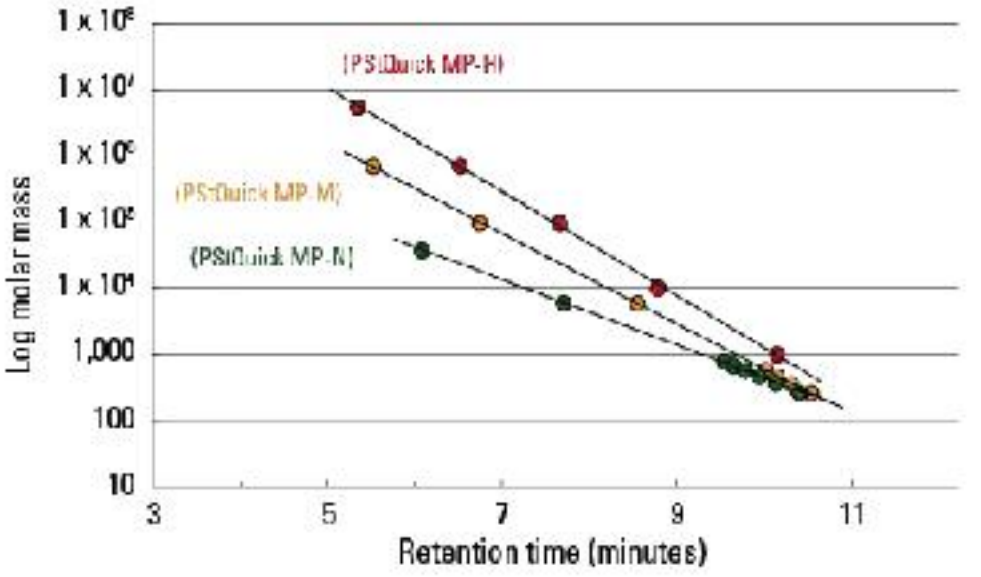
PStQuick GPC POLYSTYRENE CALIBRATION STANDARDS

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 25





(PStQuick MP-H)	(PStQuick MP-M)	(PStQuick MP-N)
1.Mw 5480000(Lot No.TS- 30)	6.Mw 708000(Lot No.TS-201)	12.Mw 37900(Lot No.TS-202)
2.Mw 706000(Lot No.TS-201)	7.Mw 96400(Lot No.TS-144)	13.Mw 5970(Lot No.TS-503)
3.Mw 96400(Lot No.TS-144)	8.Mw 5970(Lot No.TS-503)	14.Mw 682(Lot No.TS-505)
4.Mw 10200(Lot No.TS-508)	9.Mw 474(Lot No.TS-505)	15.Mw 578(Lot No.TS-505)
5.Mw 1010(Lot No.TS-507)	10.Mw 370(Lot No.TS-505)	16.Mw 474(Lot No.TS-505)
	11.Mw 266(Lot No.TS-505)	17.Mw 370(Lot No.TS-505)
		18.Mw 266(Lot No.TS-505)

CHROMATOGRAMS AND CALIBRATION CURVES OBTAINED USING THE PStQuickMP SERIES
Columns: SuperMultiporeHZ-H, 4.6 mm ID x 15 cm x 2; SuperMultiporeHZ-M, 4.6 mm ID x 15 cm L x 2; SuperMultiporeHZ-N, 4.6 mm ID x 15 cm x 2; Sample: PStQuick MPseries; Mobile phase: THF; Flow rate: 0.35 mL/min; Injection volume: 10 µL; Temperature: 25°C; Detection: UV@254nm (UV-8020 microcell)

TSKgel COLUMNS AND STANDARDS

TSKgel POLYSTYRENE CALIBRATION STANDARDS

TSKgel polystyrene bulk calibration standards are used to calibrate size exclusion columns for subsequent analysis of unknown samples. The standards range from 400 to 21,000,000 Da.

TABLE 10

ORDERING INFORMATION FOR TSKgel POLYSTYRENE CALIBRATION STANDARDS

Part #	Description	Weight
05202	A-300, 400 MW	10 g
05203	A-500, 530 MW	10 g
05204	A-1000, 950 MW	10 g
05205	A-2500, 2800 MW	5 g
05206	A-5000, 6200 MW	5 g
05207	F-1, 1.0 x 10^4 MW	5 g
05208	F-2, 1.7 x 10^4 MW	5 g
05209	F-4, 4.4 x 10^4 MW	5 g
05210	F-10, 1.0 x 10^5 MW	5 g
05211	F-20, 1.9 x 10^5 MW	5 g
05212	F-40, 4.2 x 10^5 MW	5 g
05213	F-80, 7.8 x 10^5 MW	5 g
05214	F-128, 1.3 x 10^6 MW	1 g
05215	F-288, 2.9 x 10^6 MW	1 g
05216	F-380, 3.8 x 10^6 MW	1 g
05217	F-450, 4.5 x 10^6 MW	1 g
05218	F-550, 5.5 x 10^6 MW	1 g
05219	F-700, 6.8 x 10^6 MW	1 g
05220	F-850, 8.4 x 10^6 MW	1 g
05221	F-2000, 2.1 x 10^7 MW	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

TSKgel COLUMNS AND STANDARDS

TABLE 9

ORDERING INFORMATION FOR PStQuick POLYSTYRENE CALIBRATION STANDARDS

To calibrate TSKgel SuperMultiporeHZ Columns

Part #	Description	Remarks	Calibration range	Contents	Vials
21912	PStQuick MP-N	For SuperMultiporeHZ-N	5.3×10^2 to 4.4×10^4	A-500, A-5000, F-4	60
21913	PStQuick MP-M	For SuperMultiporeHZ-M	5.3×10^2 to 8.0×10^5	A-500, A-5000, F-10, F-80	60
21914	PStQuick MP-H	For SuperMultiporeHZ-H	9.5×10^2 to 5.5×10^6	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	5.3×10^2 to 4.2×10^5	PStQuick E, F	40**
21916	PStQuick Kit-M	for H-type – M Grade	5.3×10^2 to 2.9×10^6	PStQuick C, D	40**
21917	PStQuick Kit-H	for H-type – H Grade	5.3×10^2 to 8.4×10^6	PStQuick A, B, C	60*

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

To calibrate TSKgel GPC Columns

Part #	Description	Remarks	Calibration range	Contents	Vials
21911	PStQuick A	for other GPC columns	2.8×10^3 to 8.4×10^6	A-2500, F-2, F-20, F-128, F-850	20
21910	PStQuick B	for other GPC columns	9.5×10^2 to 5.5×10^6	A-1000, F-1, F-10, F-80, F-550	20
21909	PStQuick C	for other GPC columns	5.3×10^2 to 2.9×10^6	A-500, A-5000, F-4, F-40, F-288	20
21908	PStQuick D	for other GPC columns	2.8×10^3 to 1.3×10^6	A-2500, F-2, F-20, F-128	20
21907	PStQuick E	for other GPC columns	9.5×10^2 to 4.2×10^5	A-1000, A-5000, F-4, F-40	20
21906	PStQuick F	for other GPC columns	5.3×10^2 to 1.9×10^5	A-500, A-2500, F-2, F-20	20


TOYOPEARL®
Butyl-650M
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TOSOH BIOSCIENCE

Zettachring 6, 70567 Stuttgart, Germany
Tel: +49 (0)711 13257 0 Fax: +49 (0)711 13257 89
info.tbg@tosoh.com www.tosohbioscience.com

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