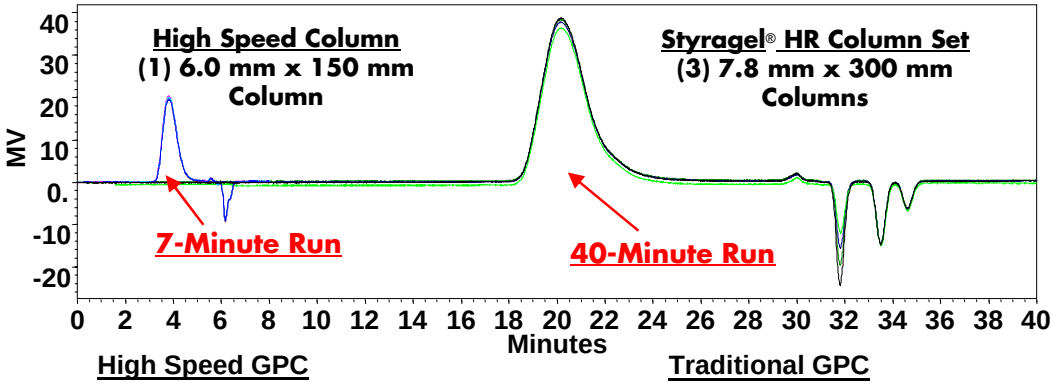
Performance PerSPECtives

Waters® Alliance System: High Speed GPC Fast, Accurate MW & MWD Results (Part 2 of 2)

High Speed GPC columns can be effectively used to generate rapid and reproducible weight average molecular weights (MW) of polymer samples. (See Waters Performance PerSPECtive 720000411EN: Waters Alliance® System: High Speed GPC - Fast, Reproducible MW Results (Part 1 of 2)) Figure 1 demonstrates how a traditional 40-minute GPC analysis of a polystyrene sample can be reduced to 7 minutes using a High Speed GPC column. Using narrow standard calibration, this technique can provide accurate results for Mw. Due to the increased effects of column and system band spreading on High Speed GPC results, the number average molecular weight (Mn), Z-average molecular weight (Mz), and molecular weight distribution (MWD) will exhibit significant deviations compared to data obtained using a more traditional GPC approach with multiple columns.

Figure 1: Overlay of Four Injections of a Broad Polystyrene Sample



High Speed GPC

System: Alliance (2695 Separations Module)
Detector: 2414 RI at 5 pts/sec (35° C)
Columns: (1) 6.0 X 150 mm High Speed Column (35° C)
Sample: Broad Polystyrene Sample
Eluent: THF(Stabilized) at 0.6 mL/min
Inj. Vol.: 5 µL
Data: Millennium®32 Chromatography Manager

Traditional GPC

System: Alliance (2695 Separations Module)
Detector: 2414 RI at 5 pts/sec (35° C)
Columns: (2) Styragel® HR5E, (1) HR2 columns 7.8 X 300 mm (35° C)
Sample: Broad Polystyrene Sample
Eluent: THF(Stabilized) at 1 mL/min
Inj. Vol.: 50 µL
Data: Millennium®32 Chromatography Manager

Broad standard cumulative matching calibration corrects for observed differences in total system band spreading between results generated using High Speed versus Traditional GPC methods. Accurate determinations of Mw, Mn, Mz, and MWD are therefore possible. This simple calibration technique is performed with Waters Millennium® 32 Chromatography Manager as follows:

Step 1: Determine molecular weight distribution for broad sample using traditional multi-column set with narrow standard calibration. Save resulting MWD data as “Named Distribution” (% Peak Area vs LogMW).

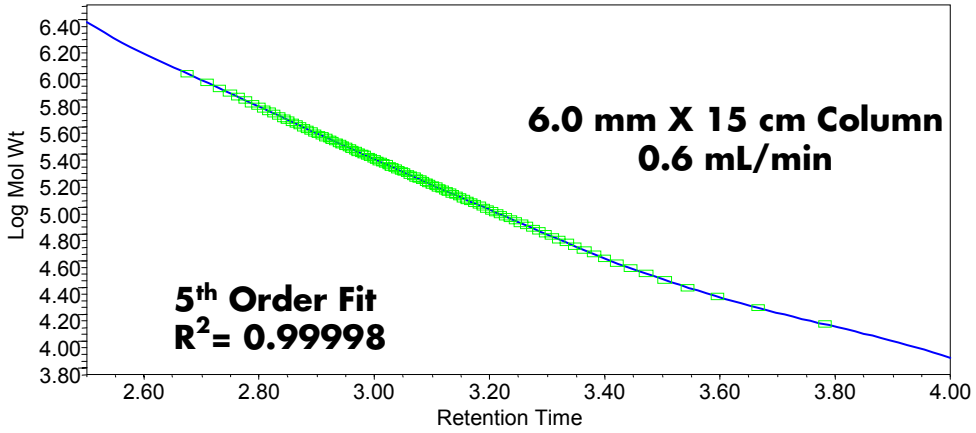
Step 2: Run broad sample on high speed column as a broad standard. Integrate and assign previously saved MWD results to broad standard peak. A cumulative matching calibration curve containing 100 data points (1% peak area increments) is generated.

Step 3: Run unknown samples on high speed column and process using the cumulative matching calibration curve.

Waters Corporation 34 Maple Street Milford, MA 01757 508 478 2000

Using this method, a cumulative matching calibration curve was obtained for a broad polystyrene standard (Figure 2). The 100 calibration points cover the molecular weight range of the broad polystyrene standard used for the calibration. If desired, additional narrow or broad standards can be added to extend the MW range of the calibration curve.

Figure 2: Broad Standard Cumulative Matching Calibration



Using the broad standard cumulative matching calibration curve, quantitation of a broad polystyrene sample using a High Speed GPC column was compared to results obtained using traditional, multi-column GPC techniques. As indicated in Table 1, calculated results were essentially the same for both GPC methods .

Table 1: High Speed vs Traditional, Multi-Column GPC Methods (N=6)

		Mn	Mw	Mz	Polydispersity
Traditional GPC	**Mean -**	105,000	233,600	396,600	2.23
	%RSD -	0.8%	0.2%	0.4%	0.6%
High Speed GPC With Cumulative Matching Calib.	**Mean -**	103,600	232,300	390,600	2.24
	%RSD -	0.8%	0.3%	0.5%	0.9%
% Variance		1.33%	0.56%	1.51%	0.45%

Summary:

- The use of High Speed GPC columns can reduce analysis times of polymer samples from 40 minutes to less than 7 minutes. This allows scientists to get precise molecular weight information very quickly.
- Accurate and reproducible Mw values can be obtained for polymer samples using narrow standard calibration data processing.
- Broad standard calibration routines, such as cumulative matching calibration, can be used for the accurate determination of Mn, Mw, Mz, and molecular weight distribution information.