REDUCE ANALYSIS TIME AND MAINTAIN RESULTS INTEGRITY

With Agilent Fast GPC Columns

The Measure of Confidence

Agilent PlusPore and PL Rapide columns

GPC is an important technique for determining the molecular weight distribution of a polymer and for comparing batch-to-batch polymer quality.

In common with other liquid chromatographers, GPC users are making more demands on the technique both in terms of speed of analysis and quality of results. These additional demands have to be balanced with the need to control costs and maintain flexibility in the laboratory.

The combined requirements for faster size-based separations with cost savings and flexibility can be achieved using novel GPC column technology.

Agilent PlusPore and PL Rapide columns are based on industry-standard, highly cross-linked polystyrene/divinylbenzene (PS/DVB) that have been specially engineered with very high pore volumes.

In GPC high pore volume has numerous benefits, including the speeding up of separations.

Agilent PlusPore and PL Rapide columns for fast GPC

- **Increased throughput:** Shorter analysis times
- **Confidence in analysis:** Results consistent with conventional GPC
- **Lower costs:** Reduced solvent usage
- **Flexibility:** Fully compatible with all GPC systems

Verified for Agilent
1260 Infinity
GPC/SEC System

Agilent Technologies

Increased Throughput

GPC columns packed with high efficiency particles that have a very high pore volume deliver significantly increased resolution compared to a conventional GPC/SEC column set.

With such columns, analysis times can be decreased by employing shorter column lengths and higher linear velocities, whilst maintaining the desired separation quality.

High resolution in less than five minutes

For fast, high resolution polymer analysis, the PolyPore, ResiPore, MesoPore, and OligoPore columns of the Agilent PlusPore range offer excellent performance. The high efficiency particles used in the columns can be operated at higher linear velocities without sacrificing resolution.

Conditions

Column:	2 x MesoPore, 4.6 x 250 mm (PL1513-5325)
Sample:	Epoxy resin
Eluent:	THF
Flow rate:	0.35 and 1.2 mL/min
Inj vol:	4 µL
System:	1260 Infinity GPC/SEC System, UV, 254 nm

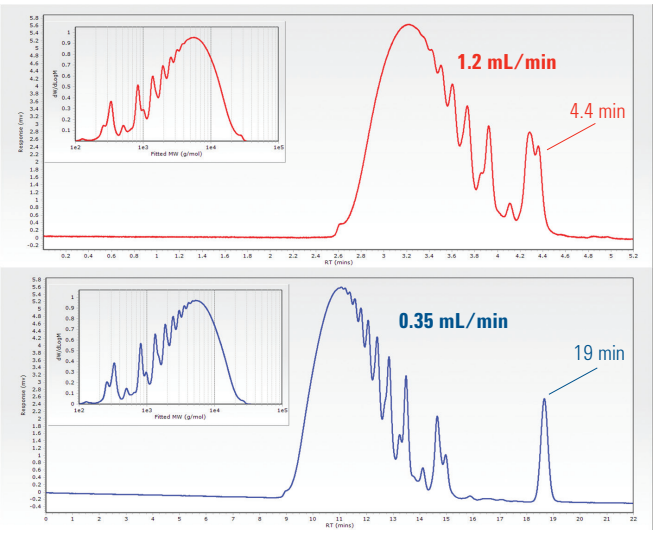


Figure 1. *Method transfer from standard GPC (bottom) to rapid GPC (top) on Agilent MesoPore 4.6 x 250 mm GPC columns. Note that analysis time is reduced to just five minutes, with minimal loss in resolution.*

Fastest ever analysis in less than two minutes

For the fastest ever GPC, when speed is of utmost concern and resolution is secondary, the Agilent PL Rapide column allows analysis in less than two minutes. These columns use reduced column length and high flow rates to deliver significantly increased sample throughput compared to a conventional GPC/SEC column set.

Conditions

Column:	PL Rapide L, 10 x 100 mm (PL1013-2300)
Sample:	Epoxy resin
Eluent:	THF
Flow rate:	1.0, 2.0 and 3.0 mL/min
System:	1260 Infinity GPC/SEC System, UV, 254 nm

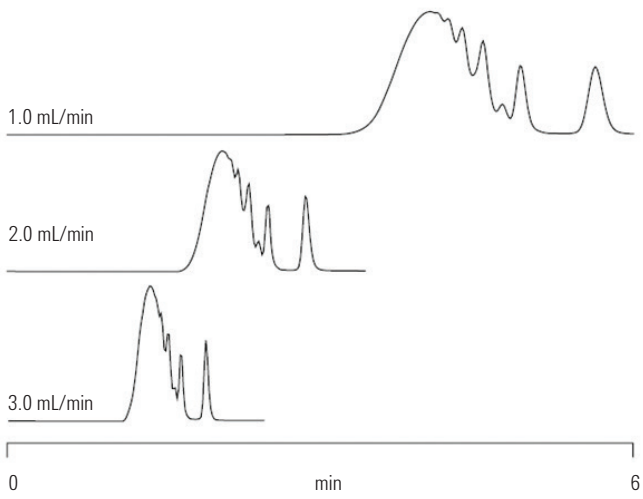


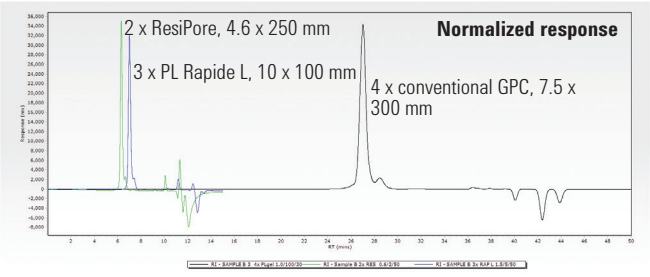
Figure 2. *Agilent PL Rapide GPC columns reduce analysis times while maintaining the excellent solvent compatibility and mechanical stability you expect from Agilent GPC columns.*

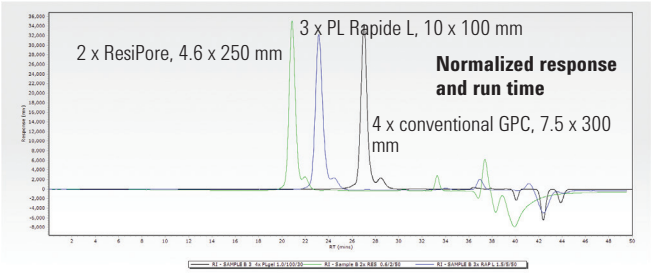
Without Sacrificing Separation Quality

The example below shows how a separation with conventional GPC columns can be transferred to Agilent PlusPore and PL Rapide with a reduction in run times and increase in throughput.

Conditions

	Conventional GPC	PL Rapide	PlusPore
Columns:	Agilent PLgel 10 µm 10^6 7.5 x 300 mm (p/n PL1110-6160), PLgel 5 µm 10^5 7.5 x 300 mm (p/n PL1110-6550), PLgel 5 µm 10^4 7.5 x 300 mm (p/n PL1110-6540), PLgel 5 µm 10^3 7.5 x 300 mm (p/n PL1110-6530)	3 x Agilent PL Rapide L, 10 x 100 mm, 3 µm (p/n PL1013-2300)	2 x Agilent ResiPore, 4.6 x 250 mm, 3 µm (p/n PL1513-5300)
Standards:	Agilent EasiVial PS-H, (MW 162 to 6,000,000) p/n PL2010-0201	Agilent EasiVial PS-M (MW 162 to 400,000) p/n PL2010-0301	
Mobile phase:	THF		
Flow rate:	1.0 mL/min	1.5 mL/min	0.6 mL/min
Sample:	Kraton, expected molecular weight < 400,000 g/mol		
Sample conc:	1.5 mg/mL		
Inj vol:	100 µL	5 µL	2 µL
Column temp:	30 °C	50 °C	
System:	Agilent 1260 Infinity GPC/SEC system with RI detector and Agilent GPC/SEC software		





Throughput is increased by more than 3x

Columns	Peak 2 retention time (min)	Run time (min)
4 x conventional 7.5 x 300 mm	28.46	50
3 x PL Rapide L 10 x 100 mm	7.41	15
2 x ResiPore 4.6 x 250 mm	6.66	15

Without sacrificing separation quality

Columns	Resolution (Rs)	Selectivity (α)	Area %	Height %
4 x conventional 7.5 x 300 mm	1.2	1.05	8	7
3 x PL Rapide L 10 x 100 mm	1.1	1.06	7	7
2 x ResiPore 4.6 x 250 mm	1.1	1.05	8	8

Figure 3. *Comparison of run times and separation quality between conventional GPC and Agilent ResiPore and PL Rapide L columns. Analysis times are decreaseed by a factor of more than 3 and the quality of separation is maintained.*

Further details of this application are available in the Agilent poster *Achieving Faster GPC Separations using Novel High Pore Volume Columns and Conventional Instruments* (5991-5207EN)

Confidence in Analysis

It is critical that when a method is changed the integrity of the results is maintained.

If the move from conventional GPC to fast GPC resulted in a significant change in the calculated results, this could undermine confidence in the analysis.

Agilent PlusPore and PL Rapide columns are based on industry-standard, highly cross-linked polystyrene/divinylbenzene (PS/DVB) particles for which the mechanisms of size based separations are well characterized. As a consequence the results obtained with them are consistent with conventional GPC.

The table below shows the average and relative standard deviation for the average molecular weights from the polymer analysis on the previous page determined using conventional GPC and fast GPC using Agilent ResiPore and PL Rapide L columns.

Table 1. *Average molecular weights and precision of values obtained with conventional GPC, Agilent ResiPore and PL Rapide L columns. Low % RSD shows that the fast GPC results are consistent with conventional GPC.*

Sample B			
	Mp	**Mn**	**Mw**
Peak 1 Mean	110262	107964	111181
Peak 1 % RSD	3	1	1

Avoid dislocations

The use of individual pore size columns in GPC is often problematic because spurious peak shapes can be created and precision lost in the calculated molecular weight averages due to "mismatched" calibration curves.

Agilent PlusPore and PL Rapide columns overcome this issue through the use of MIXED bed technology. Every column contains a mixture of individual pore size materials, accurately blended to cover a specified broad range of molecular weight with a linear calibration that eliminates dislocations.

Figure 4. *Spurious peak shape causes by dislocation (mismatched calibration curves)*

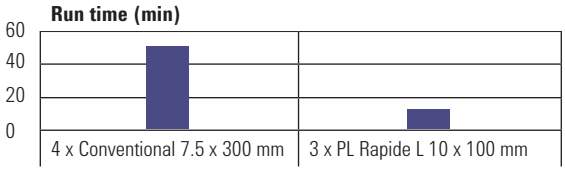
Lower Costs

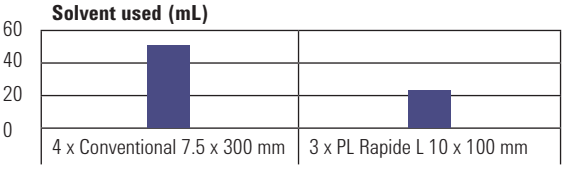
Cost per analysis is an important metric for many laboratories. Reducing the time taken to perform an analysis and the amount of any materials used during the analysis will reduce this cost.

For GPC analysis, solvent is used during each run. There are costs associated with both the purchase and disposal of solvents.

The table below shows the impact of converting the analysis shown earlier in this literature from conventional GPC to fast GPC using Agilent PL Rapide columns.

70% saving in analysis time and 55% saving in solvent usage

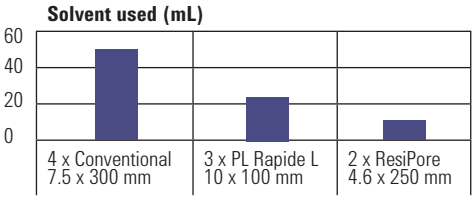




Columns	Run time (min)	Flow rate (mL/min)	Solvent used (mL)
4 x Conventional 7.5 x 300 mm	50	1.0	50
3 x PL Rapide L 10 x 100 mm	15	1.5	22.5

Figure 5. *Run time and solvent used for conventional GPC columns compared to PL Rapide L, demonstrating a 70% saving in analysis time and a 55% saving in solvent usage.*

Use a narrower id column for an 82% saving on solvent usage



Columns	Run time (min)	Flow rate (mL/min)	Solvent used (mL)
4 x Conventional 7.5 x 300 mm	50	1.0	50
2 x ResiPore 4.6 x 250 mm	15	0.6	9

Figure 6. *Run time and solvent used for conventional GPC columns compared to ResiPore, demonstrating an 82% saving in solvent usage.*

Flexibility

In liquid chromatography as particle size decreases the pressure required to pump solvent through a column increases. If very small particles are used the pressures become so high that specialized instruments are required.

The need for a specialized instrument for a particular analysis increases costs and reduces flexibility in the laboratory.

Because Agilent PlusPore and PL Rapide columns are based on PS/DVB particles with diameters of 3 µm and larger they maintain compatibility with conventional GPC instruments.

This means that fast GPC using Agilent columns can be performed on a variety of instruments including the Agilent 1260 Infinity GPC/SEC systems.

Agilent 1260 Infinity GPC/SEC System

The 1260 Infinity GPC/SEC System is designed for cost-effective, routine polymer characterization with refractive index, UV-visible or evaporative light scattering detectors. It is based on the reliable Agilent 1200 Infinity Series LC modules. Instrument control, data acquisition and analysis can be performed with the easy-to-use Agilent GPC/SEC software.

The Agilent 1260 Infinity Multi-Detector GPC/SEC System is a high-end multiple detector platform, which provides not only accurate molecular weight data independent of the chemistry of the standards, but also gives insight into the behavior of the polymer in solution.

Agilent 1260 Infinity Multi-Detector GPC/SEC System

Ordering Information

Columns for use with organic solvents

Description	MW range (g/mol PS)	Particle size (µm)	Guaranteed efficiency (p/m)	Part number	Recommended calibrants
PL Rapide H, 10 x 100 mm	500 to 10,000,000	10	>35,000	PL1013-2100	EasiCal PS-1 (p/n PL2010-0501) or EasiVial PS-H (p/n PL2010-0201)
PL Rapide H, 7.5 x 150 mm				PL1113-3100	
PolyPore 2.1 x 250mm	200 to 2,000,000	5	>60,000	PL1913-5500	EasiCal PS-1 (p/n PL2010-0501) or EasiVial PS-H (p/n PL2010-0201)
PolyPore, 4.6 x 250 mm				PL1513-5500	
PolyPore, 7.5 x 300 mm				PL1113-6500	
PL Rapide M, 10 x 100 mm				PL1013-2500	
PL Rapide M, 7.5 x 150 mm				PL1113-3500	
ResiPore 2.1 x 250 mm	200 to 400,000	3	>80,000	PL1913-5300	EasiCal PS-2 (p/n PL2010-0601) or EasiVial PS-M (p/n PL2010-0301)
ResiPore, 4.6 x 250 mm				PL1513-5300	
ResiPore, 7.5 x 300 mm				PL1113-6300	
PL Rapide L, 10 x 100 mm				PL1013-2300	
PL Rapide L, 7.5 x 150 mm				PL1113-3300	
MesoPore, 2.1 x 250 mm	up to 25,000	3	>80,000	PL1913-5325	Polystyrene S-L-10 Kit (p/n PL2010-0101)
MesoPore 4.6 x 250 mm				PL1513-5325	
MesoPore, 7.5 x 300 mm				PL1113-6325	
OligoPore, 2.1 x 250 mm	up to 4,500	6	>80,000	PL1913-5520	Polystyrene S-L2-10 Kit (p/n PL2010-0105)
OligoPore 4.6 x 250 mm				PL1513-5520	
OligoPore, 7.5 x 300 mm				PL1113-6520	
PL Rapide F, 10 x 100 mm				PL1013-2120	
PL Rapide F, 7.5 x 150 mm				PL1113-3120	

Columns for use with aqueous solvents

Description	MW range (g/mol PEG/PEO)	Particle size (µm)	Guaranteed efficiency (p/m)	Part number	Recommended calibrants
PL Rapide Aqua H, 10 x 100 mm	100 to 10,000,000	8	>35,000	PL1049-2800	EasiVial PEG/PEO (p/n PL2080-0201)
PL Rapide Aqua H, 7.5 x 150 mm				PL1149-3800	
PL Rapide Aqua L, 10 x 100 mm	100 to 30,000			PL1020-2830	EasiVial PEG (p/n PL2070-0201)
PL Rapide Aqua L, 7.5 x 150 mm				PL1120-3830	

Preparative columns

Description	Part number
OligoPore Prep, 25 x 300 mm	PL1213-6520

Guard columns

Description	Part number
PolyPore Guard, 4.6 x 50 mm	PL1513-1500
PolyPore Guard, 7.5 x 50 mm	PL1113-1500
ResiPore Guard, 4.6 x 50 mm	PL1513-1300
ResiPore Guard, 7.5 x 50 mm	PL1113-1300
MesoPore Guard, 7.5 x 50 mm	PL1113-1325
OligoPore Guard, 4.6 x 50 mm	PL1513-1320
OligoPore Guard, 7.5 x 50 mm	PL1113-1320

Agilent GPC columns and calibration standards are renowed for reproducibility, column-to-column, batch-to-batch, year after year. They cover all molecular weight ranges, across organic, aqueous, and polar solvents.

You can rely on the Agilent GPC/SEC portfolio to give you the information you need for complete polymer characterization, from the company with the GPC pedigree.

To get the most from your analysis, use Agilent calibration standards. To learn more about calibrating your GPC columns, refer to the primer ***Calibrating GPC Columns - A Guide to Best Practice*** (5991-2720EN).

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Providing you access to a full-circle partner you can trust, Agilent delivers:

- Over 35 years of industry-leading solutions for characterizing and separating polymers by GPC/SEC
- A comprehensive portfolio of industry-leading columns and calibrants
- A full lineup of instruments and software, for accurate polymer analysis
- 24/7 worldwide technical support
- Unsurpassed global logistics for on-time delivery of critical supplies

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