



Investigating Miniaturization in GPC/SEC

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## What are the advantages of Minaturiztion?

Shorter run times for higher sample throughput

Improvements in resolution

Reduce cost by adjusting flow rates and cutting the amount of solvent required

Improve peak shape and reproducibility



# How to Achieve Higher Resolution

- High efficiency GPC/SEC columns packed with <u>small</u> <u>diameter particles</u>
- High pore volume GPC/SEC columns packed with "<u>multipore</u>" particles, with a near-linear molecular weight range appropriate for your samples, that will increase resolution
- <u>Shorter lengths and wider diameters</u> that allow high linear velocity to be used in order to greatly reduce run time and still maintain acceptable resolution over a wide molecular weight range



PLgel 3um / 5um PL aquagel-OH 5um

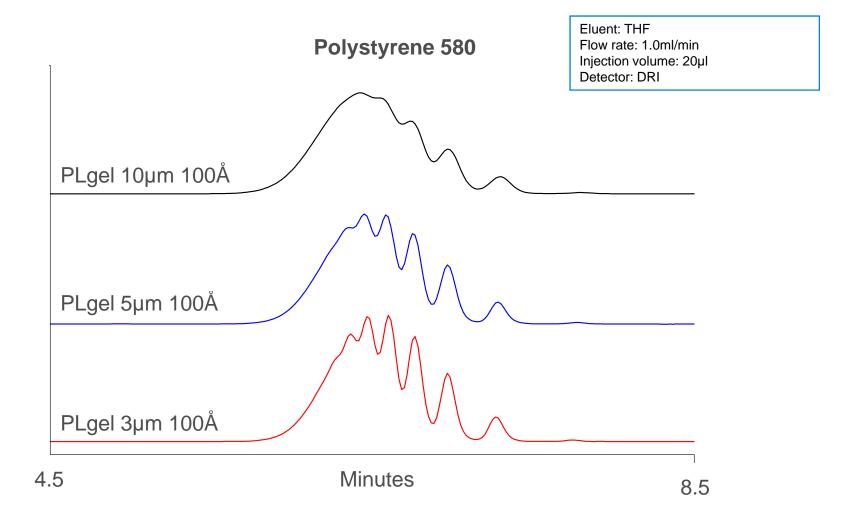
PlusPore

**RL** Rapide

PL Rapide Aqua

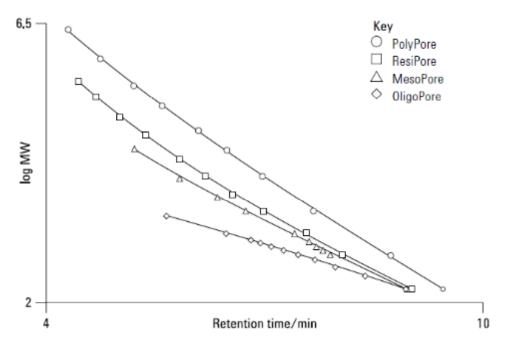


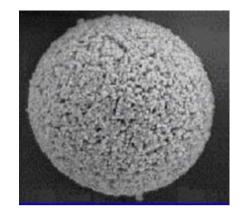
# High Resolution with Smaller Particles





#### PlusPore – High Performance GPC Columns





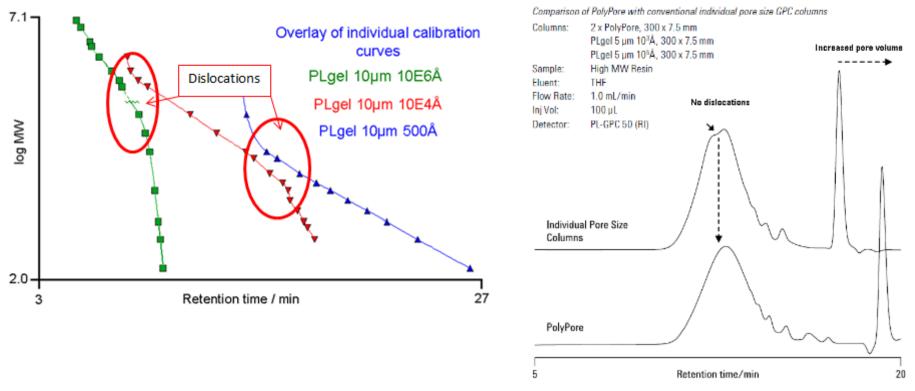
PlusPore calibration curves

#### **PlusPore selection guide**

Column	MW range (g/mol) (PS)	Nominal particle size (µm)	Typical efficiency (p/m)	Recommended calibrants	Frit porosity (µm)
PolyPore	200 to 2,000,000	5	>60,000	EasiCal PS-1or EasiVial PS-H	2
ResiPore	up to 500,000	3	>80,000	EasiCal PS-2 or EasiVial PS-M	2
MesoPore	up to 25,000	3	>80,000	Polystyrene S-L-10 Kit,	2
OligoPore	up to 3,300	6	>55,000	Polystyrene S-L2-10 Kit	2



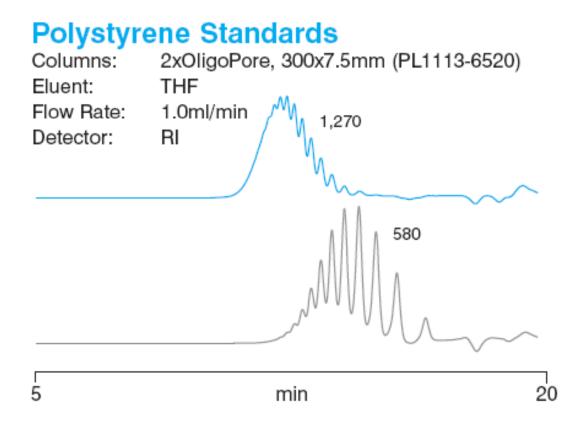
# Avoid Dislocations



- Individual pore size columns can exhibit dislocations where the Mw resolution ranges overlap
- Mixed bed columns have a wide linear range that prevent dislocations



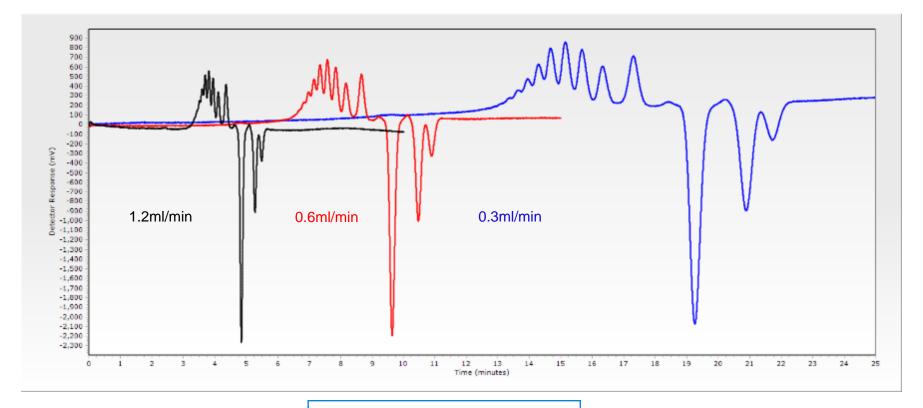
# High Resolution OligoPore Separations



- The OligoPore column is filled with 6um packing material, but due to a very large pore volume it gives increased resolution compared to a 3um Plgel column
- As a result of the larger particle size, OligoPore is very resilient to extra-column dispersion



#### Polystyrene Mw 580 – Oligopore 250x4.6mm



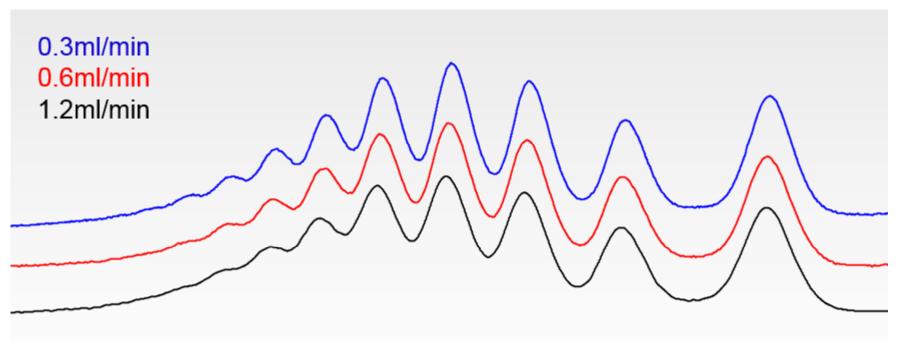
MW Range: up to 3,300 (g/mol)

Nominal Particle Size: 6 µm

Typical Efficiency: >55,000 p/m



# Resolution maintained as speed is increased

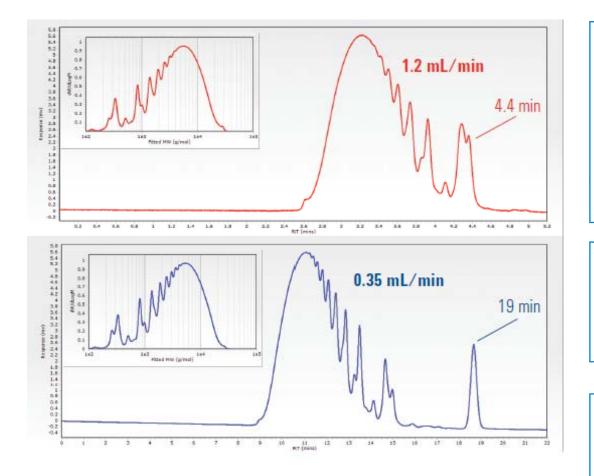


Polystyrene Mw 580 resolved using Oligopore 4.6x250mm

Different flow rates overlaid to show that faster doesn't sacrifice resolution. The chromatograms have been normalised to better illustrate the differences



#### High Speed MesoPore Columns



5
2 x MesoPore, 4.6 x 250 mm (PL1513-5325)
Epoxy resin
THF
0.35 and 1.2 mL/min
4 µL
1260 Infinity GPC/SEC System, UV, 254 nm

Easy Method Transfer from Standard to rapid GPC on MesoPore 250x4.6mm GPC columns

*MW Range*: up to 25,000 (g/mol) Nominal Particle Size: 3 μm Typical Efficiency: >80,000 p/m



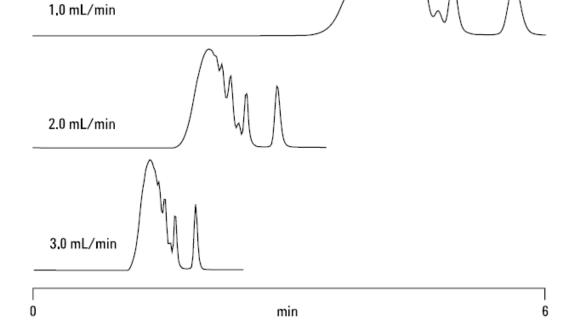
#### Very High Speed PL-Rapide Separations

PL Rapide columns reduce analysis times while maintaining the excellent solvent compatibility and mechanical stability of all GPC columns from Agilent

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Column:	PL Rapide L, 100 x 10 mm
Sample:	Epoxy resin
Eluent:	THF
Flow Rate:	1.0 , 2.0 and 3.0 mL/min
Detector:	UV, 254 nm

Description	MW range (g/mol)
PL Rapide H, 150 x 7.5 mm	500 to 10,000,000
PL Rapide H, 100 x 10mm	500 to 10,000,000
PL Rapide M, 150 x 7.5 mm	200 to 2,000,000
PL Rapide M, 100 x 10 mm	200 to 2,000,000
PL Rapide L, 150 x 7.5 mm	200 to 500,000
PL Rapide L, 100 x 10 mm	200 to 500,000
PL Rapide F, 150 x 7.5 mm	up to 3,300
PL Rapide F, 100 x 10 mm	up to 3,300

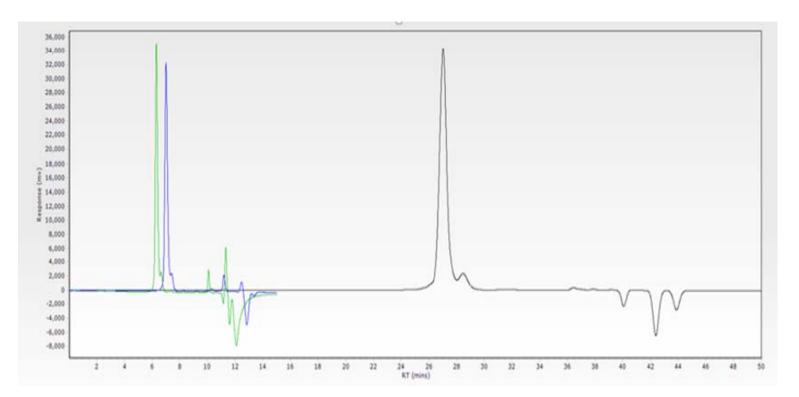




# Reproducible Results

Samples	Columns
	Agilent PLgel 10 µm 106 7.5 x 300 mm
	Agilent PLgel 5 µm 105 7.5 x 300 mm
Agilent EasiVial	Agilent PLgel 5 µm 10 <sup>4</sup> 7.5 x 300 mm
PS-H and PS-M	Agilent PLgel 5 µm 103 7.5 x 300 mm
	3 x Agilent PL Rapide L, 10 x 100 mm
Kraton	Linear MW range up to 500,000 g/mol
	2 x Agilent ResiPore, 4.6 x 250 mm
	Linear MW range up to 500,000 g/mol

Columns:	4 x PL gel	3 x PL <u>Rapide</u> L	2 x ResiPore		
Mobile phase:	THF				
Flow rate:	1.0 mL/min	1.5 mL/min	0.6 mL/min		
Sample conc.:		1.5 mg/mL	-		
Inj. volume:	100 µL	5 µL	2 µL		
Temperature:	30 °C	50 °C	50 °C		
Run time:	50 minutes	15 minutes	15 minutes		





#### **Consistent Results**

methods can be transferred with confidence without the risk of absorbance effects or other interactions between the analytes and stationary phase.

Chromatography and MW results run on PL gel, PL Rapide L and Resipore columns

Columns	Tr (Peak 2)	Rs	N∕m (Peak 2)	а	Area (%)	Height (%)
4 x PLgel	28.46	1.22	5653	1.05	8	7
3 x PL Rapide L	7.41	1.13	23727	1.06	7	7
2 x ResiPore	6.66	1.10	14510	1.05	8	8

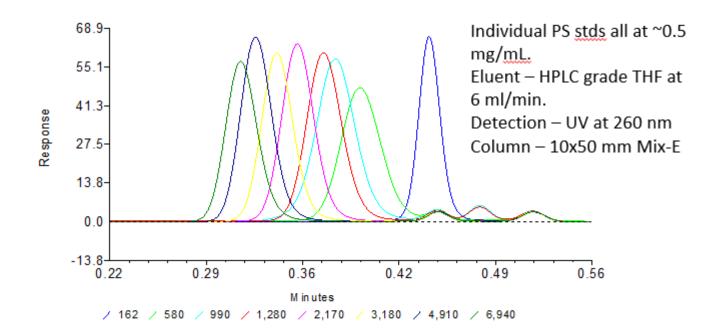
Average and precision of molecular weight results run on PL gel, PL Rapide L and Resipore columns

	Мр	Mn	Mw	PD	Мр	Mn	Mw	PD
	Peak 1				Peak 2			
Mean	110262	107964	111181	1.03	56041	51015	53208	1.05
%RSD	3	1	1	0	5	8	5	4



#### Potential for 2DLC/GPC

PS standards UV Chromatograms - 10x50 Mix-E at 6ml/min

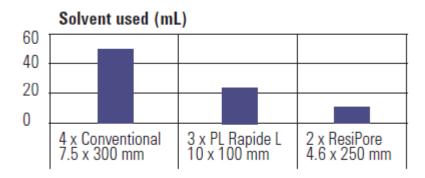


Comparison of molecular weight distributions derived from 10 x 50 mm and 7.5 x 600 mm versions of the Agilent PLgel MIXED-E column.

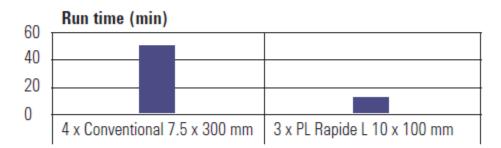
Agilent PLgel dimensions	Mn (g/mole)	Mw (g/mole)	Mz (g/mole)	Mz+1 (g/mole)
10 x 50 mm	1.369	2.938	4.957	6.517
7.5 x 600 mm	1.509	2.912	4.562	5.732



### Solvent Usage – Cost Saving



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



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



# Summary

Shorter run times	High speed, high resolution separations can be achieved using Multiporous Polystyrene/Divinyl Benzene particles
Save cost with reduced solvent consumption	Smaller i.d. columns and faster flow rates reduces solvent consumption by as much as 80% compared to conventional columns.
No column dislocations or absorption	Linear column calibrations improve peak shape and accuracy of results
Increased performance on an analytical GPC system	Miniaturized high pore volume columns packed with PS/DVB particles operate at low pressure and elevated flow rates





