UGPC? - New Dimensions for GPC/SEC

Application Note

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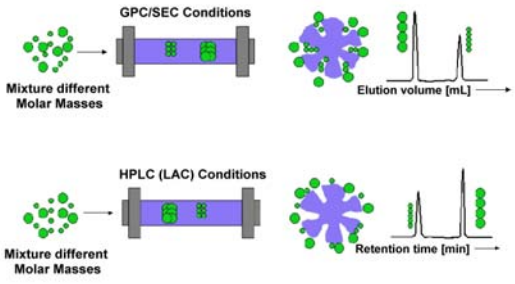
Introduction
Ultra-HPLC (UHPLC) provides a new potential for method development and analysis. It is achieved by using sub-2 µm particle size column packing at increased linear velocities. The advantage of UHPLC over conventional HPLC is the reduced analysis time without sacrificing efficiency. In comparison to traditional HPLC analysis time can be decreased up to a factor of 7-10.

So the question for GPC/SEC users is:
Can this approach be easily transferred to speed up GPC/SEC measurements.

Unfortunately the answer is: No - you need other solutions as shown below!

Why new solutions?
Although the general instrumentation required for HPLC and GPC/SEC is quite similar (pump, injection system, column, detector), the separation mechanism is totally different. While HPLC separation is dominated by interaction with the stationary phase surface, GPC/SEC separation requires interaction-free size exclusion in the stationary phase pores. The Figure illustrates these differences for a mixture of PMMA with different molar masses.



HPLC separation can be influenced by the choice of the stationary phase material, the gradient and the temperature. GPC/SEC separation can only be influenced by the pore volume of the accessible pores. This means that the resolution can only be increased by, e.g. adding another separation column. Unfortunately this means that the analysis time and the amount of solvent needed also increase.

In addition, it is not possible to reduce the particle size in GPC/SEC as in UHPLC for two reasons:
- If the particles are too small and too many wide pores are present the particle will be very unstable.
- If the particle is too small the shear rate for (high molar mass) macromolecules can be so strong, that they are degraded.

An exception is only for low molar mass macromolecules. Latest improvements here are e.g. PSS SUPREMA columns with reduced particle size and highest resolution for oligomers and proteins in solutions with pH values > 7.

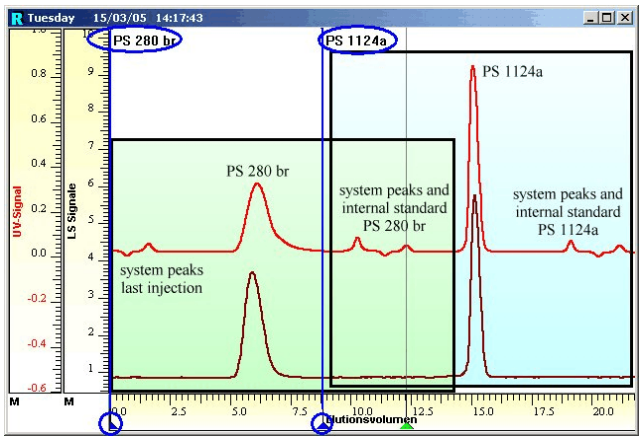
This means for UGPC totally new concepts are required and have been developed.

GPC/SEC UGPC Solutions Saving Time and Solvent

1) Overlaid Injection

This approach, when compared with standard injection, saves 30-45% of solvent and time for every sample - without any loss in resolution.

Every GPC/SEC column has an interstitial volume, where only solvent can elute while the sample is still moving through the column.
For analytical columns with a length of 300 mm and a diameter of 8 mm this is approximately 5 mL. For separations at a flow rate of 1 mL/min this means that during the first 5 minutes, sample can not be eluted. So it is not necessary to wait for complete elution of one sample before injecting the next sample. The figure shows an example of overlaid injection (injection mark = blue triangle at the bottom). Before the system peaks and the internal standard (light green triangle) of “PS 280 br” are eluted, the next sample “PS 1124 a” is already injected. If the software used supports overlaid injection the evaluation of the sample is NOT affected.



2) HighSpeed GPC/SEC Columns

This approach reduces the analysis time required by 67 - 85% for every sample - a loss of resolution is only observed in the low molar mass region below 2 000 g/mol. This approach requires the same amount of eluent as conventional analysis with analytical GPC/SEC columns.

HighSpeed columns are run at a flow-rate of 3 to 6.25 mL/min (depending on the solvent viscosity) and can replace analytical columns easily. They are successfully used in many HT applications, in production control, and in on-line 2-dimensional chromatography. An additional benefit is the low back pressure generated when using these columns. Therefore they are ideal for high molar mass samples.

3) Micro GPC/SEC Columns

This approach allows the savings of 66% eluent for every sample. However to avoid a loss in resolution optimized columns and instrument hardware are required. The best results for micro columns are obtained when using semi-micro instrumentation with optimized flow paths, low internal dead volume and small detector cells (e.g. EcoSEC semi-micro GPC system).

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The key to techniques 2 and 3 above is in both cases to use new dimensions in GPC/SEC[1-4]:

Optimized dimensions, as shown in the figure, optimized column materials, and matching flow-rates allow the user to save either time or eluent without sacrificing resolution.

Overlaid injection is an approach that is independent of the columns used and is applicable to analytical, HighSpeed, and micro columns.

Summary System Requirements, Parameter & Savings

Requirements	***Overlaid Injection***	***HighSpeed GPC/SEC***	***Micro GPC/SEC***
Hardware	works with all instruments and columns from all vendors	works with all instruments from all vendors	requires optimized hardware, e.g EcoSEC
Software	PSS WinGPC Unity	any software (PSS WinGPC Unity)	any software (PSS WinGPC Unity)
Column details	any column	PSS HighSpeed ID: 20 mm length: 50 mm	PSS Micro ID: 4.6 mm length: 250 mm
Flow-rate	any	3 - 6.25 mL/min	0.33 mL /min
Eluent needed /sample and column	depends on column type used	12 mL (the same as a comparable analytical column)	3.5 mL
Time needed /sample and column	depends on column type used	2 - 4 minutes	10 minutes (comparable with an analytical column)
Savings	30-45% time and eluent per sample	66-85% time	66% eluent

Literature

[1] T. Hofe, D. Held, G. Reinhold, C. Trimmer, Streamlined GPC/SEC analysis with optimum column design, American Laboratory 3 (2003), page 12

[2] H. Pasch, P. Kilz, Fast Liquid Chromatography for High-Throughput Screening of Polymers, Macromolecular Rapid Communication 24 (2003), page 104

[3] P. Kilz, Methods and columns for High-Speed size exclusion chromatography separations, Handbook of size exclusion chromatography and related techniques (2004), page 561

[4] PSS Streamliner 1/2009: Focus: Green GPC/SEC, Company Magazine Issue 1 (2009)

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