

Tips & Tricks: GPC/SEC How to Expand The Lifetime of Columns

Günter Reinhold, PSS Polymer Standards Service GmbH, Mainz, Germany.

Unlike HPLC columns, GPC/SEC columns are made of crosslinked polymeric materials with a permanent porosity. Because of the permanent porosity GPC/SEC columns are pressure sensitive and need more care than other LC columns.

An important quality criterion for the lifetime of columns is the crosslinking density. A low crosslinking density leads to poor mechanical properties and low solvent compatibility. However, highly crosslinked material supports unwanted adsorption, so that a good compromise needs to be found.

Packing of GPC/SEC columns requires special knowledge and strict procedures to achieve highest resolution and optimum size separation. This makes GPC/SEC columns more expensive than HPLC ones.

What Lifetime Can be Expected?

Although GPC/SEC columns are expected to be, in general, less stable than HPLC columns, they can be used for several years if handled properly. For example, the PSS contract analysis

laboratory has several columns that have been in use for ten years or more. These columns are still working to the utmost satisfaction and show how stable high-quality columns can be.

However, wrong conditions, a non-matching solvent or high pressure can destroy the column. The first injection can come to be the last one if the wrong column type or solvent is used. Therefore, it is important to review the specifications of a column (pressure limit, solvent compatibility, recommended applications) before developing new methods. Figure 1 shows the PSS Magic Triangle that is a visual guide for selecting the appropriate column material with respect to the sample and the solvent.

Which Parameters Should be Monitored for the Column to Continuously Increase the Lifetime?

It is good practice to determine at least the **plate count** regularly (e.g., with every run or at given intervals). In addition the **system pressure** without columns **and**

with all columns connected should be known. Any plate count decrease or pressure increase with an injection or slowly over time is an indicator that a problem exists and this could influence the lifetime of the column.

How Can GPC/SEC Columns be Tested?

A good measure for the performance of columns is the plate count and the specific resolution. There are several standards (e.g., ISO 13885, DIN 55672, ASTM D 5296-05 and others) that provide criteria for plate count, asymmetry and resolution.

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These parameters should be known for the complete GPC/SEC system including the columns.

To measure these parameters a low molar mass substance (acetone, BHT etc.) will be injected. Detailed experimental conditions can be found in the corresponding standard or on the column quality certificate, for example.

The calculation of the theoretical plate count per metre, N_{th} [1/m], uses the peak position and the peak width at half peak height according to

$$N_{th} = \left(\frac{V_R}{\sigma}\right)^2 = \frac{554}{L} \cdot \left(\frac{V_R}{w_{1/2}}\right)^2 \quad [1]$$

where σ is the variance, which can be estimated by the half-height method, $w_{1/2}$ and L is the column length in cm.

The specific resolution R_{sp} specifies the quality of resolution of two peaks, R_s , whose molecular weight differs by one order in magnitude:

$$R_{sp} = \frac{R_s}{\lg \frac{M_1}{M_2}} = \frac{0.579}{\sigma \cdot D} \quad [2]$$

Figure 2 shows the results for a column set of three different analytical PSS SDV 5 μ columns and a precolumn. The green

numbers indicate that the specifications for the standard are met.

How Can the Column Lifetime be Extended?

A few tricks can help to increase the column lifetime.

Use a precolumn:

Precolumns are filled with the same material with respect to stationary phase and particle size as the analytical column. However, the surface area of the material is high so that they have a high adsorption potential. This helps to protect the main columns. Because precolumns are much shorter they are less expensive, so precolumns help to save money.

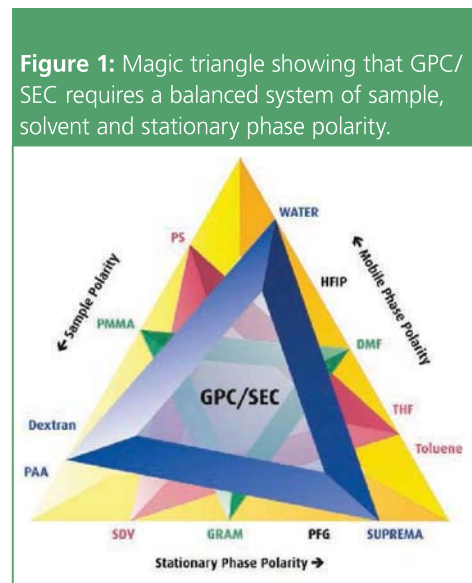


Figure 1: Magic triangle showing that GPC/SEC requires a balanced system of sample, solvent and stationary phase polarity.

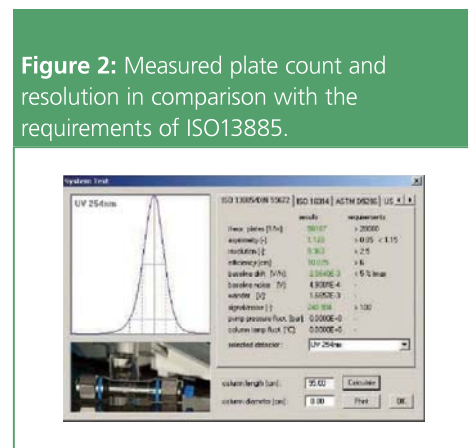


Figure 2: Measured plate count and resolution in comparison with the requirements of ISO13885.

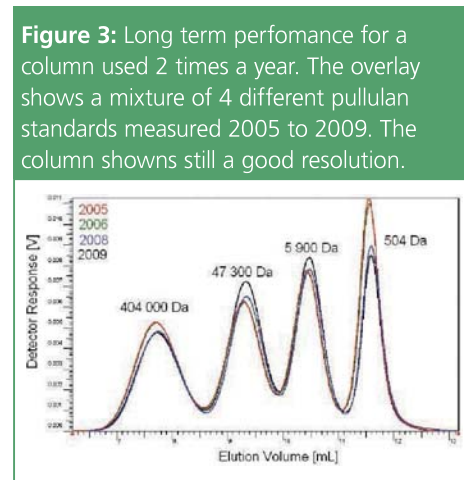


Figure 3: Long term performance for a column used 2 times a year. The overlay shows a mixture of 4 different pullulan standards measured 2005 to 2009. The column shows still a good resolution.

Prepare sample solutions cautiously:

It is imperative that a sample passing through a column be soluble in the mobile phase. When using a solvent different to the mobile phase for sample dissolution, the two solvents must be miscible. The formation of precipitates inside the columns must be prevented. This should be tested before the sample is injected.

Macromolecules need time to dissolve. Let polymers below 200000 Da sit for at least 3–4 hours. Increase the dissolution time with increasing molar mass. Dissolution for ultra high molar mass samples (>2000000 Da) may take several days. Micro gels in the range of 0.5 to 2 μ m in samples can cause problems with the system. Therefore, filtering of the samples before injection is recommended. Most of the time column frit blockage can be prevented using 0.45 to 1 μ m filters.

Additives in technical products (stabilizers, antioxidants and others) can cause damage if the chromatographic conditions are not ideal for these substances. For example, if the column shows lower plate-count, lower resolution or unexpected double peaks after injecting a new sample it is important to react fast. Check the column performance, try to identify additives (or new monomeric units in the macromolecule) and talk to the column

manufacturer. It's often possible to repair the column when using special column cleaning procedures. However, these protocols are not universal because every column manufacturer has its own recipe for the stationary phase synthesis.

Use compatible solvents:

A wrong solvent can destroy the column beyond repair. Refer to the column user manual for manufacturer recommendations. If in doubt contact the manufacturer or refer to the literature.

Store columns appropriate:

Never have salt solutions in the columns without flow. Always apply at least a low flow-rate, even when the system is not used. If the columns are not going to be used

for several weeks/months, it is good practice to keep columns with volatile or instable mobile phases in a refrigerator (4 °C) to prevent solvent evaporation or degradation. Remove all salt solutions with pure solvents (approx. 10 column volumes needed) and plug the columns tightly with the original end plugs. However, never let the column temperature fall below the freezing point of the storage solvent. This can destroy the stationary phase.

Occasionally solvent is lost during long-term storage or as a result of high storage temperatures. When the expected system pressure does not build up or the pump constantly re-regulates the flow, it is an indication of a partially evaporated column solvent. To re-wet a partially dry column use the procedures described in the column user documentation.

Dr Günter Reinhold studied chemistry in Merseburg (Germany) and is working in the PSS column department. He develops new column materials and is responsible for column applications.

E-mail: Greinhold@polymer.de
Website: www.polymer.de

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