



GPC/SEC Troubleshooting

#1 Pressure Problems

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Q: My GPC/SEC system runs at a pressure of 80 bar/1160 psi. Now the system pressure has been increased and the pump has shut down to protect the columns. What should I do?

A: This is one of the most frequently occurring problems in GPC/SEC. Therefore, it is always good to have reference points for the pressure. For example, for your system with your solvents and at your typical flow-rates, you should know:

- the pressure of the system with detectors, but **WITHOUT** the columns
- the pressure of the system with detectors and all columns if your column certificates show the pressure for the column, it is not required to know the pressure for each single installed column.

The pressure of every single column roughly adds up to the system pressure.

A pressure increase can be caused by the column(s) or by the system. To decide if it is the column or the system disconnect the columns and measure the pressure without the columns:

If the pressure of the system is too high:

- Check, clean and replace any filters or frits in the system and see if that solves the problem.
- If not, disconnect single components of the systems, starting at the end. In other words, disconnect the (last) detector, then the tubing, etc. until you identify the part responsible for the pressure increase.
- If it is a tubing then replace it, keeping in mind that if the length changes it might be necessary to re-calibrate or to determine a new inter-detector delay.
- If it is a detector cell then have a look at the user documentation to see if there are instructions or recommendations. In many cases it helps to flush the cell with a good solvent for the last samples to have been analysed.

If the system pressure is as expected:

- Connect the columns separately to the system and check the pressure; that is, add the precolumn and read the pressure, add the first separation column and read the pressure, etc. Continue until you have identified the source of the pressure increase.
- If it is the precolumn, replace it and calibrate your system or verify that the calibration is still valid.
- If it is a separation column, read the user documentation to see if there are procedures to clean the column. If you do have spare frits for the columns and repair gel, it is sometimes possible to repair the column, even if it is a polymer column. However, be warned - if you do not have the frit and repair gel, do not open the column.
- If the problem is not solved by replacing the column frit, adsorption on the column surface might be to blame. In this case, review all samples you have measured lately and try to find out what might have caused the problem. Check with the documentation or correspond with the column manufacturer to find out if there is a cleaning procedure available.

How can a pressure increase be avoided?

Make sure that you prepare all sample solutions and solvents for GPC/SEC cautiously:

- Let polymers of $M_w < 200\,000$ Da, sit for 3-4 hours. Higher molar masses need longer. Ultra high molecular weight standards and samples $> 2\,000\,000$ Da may take from 1-3 days to dissolve completely.
- Filter your samples solutions prior to use. This is especially important if not all parts of the sample are soluble. Be careful with ultra high molecular weight samples, they might be destroyed when filtered!
- Use a precolumn and change frits and filters in your system (not in the column!) regularly.
- If different solvent types are used for sample preparation and mobile phase (e.g., HFIP to solve the sample and a mixture of Chloroform/HFIP to run the samples), make sure that no part of the sample precipitates when injected.

Daniela Held studied chemistry at the University of Mainz. Her PhD work was on the characterization of star-branched polymers using GPC/SEC and hyphenated techniques. She joined PSS in 2000 and is responsible for customer training and support.