



GPC/SEC Troubleshooting

Additional Ghost or System Peaks

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Q: I see a lot of peaks, especially in the low molar mass region of the chromatogram. Is that normal?

Comment

The most common detector in GPC/SEC is the refractive index detector, RI. In many cases this detector shows numerous positive and negative signals at the total penetration limit of the column, even when pure solvent is injected. These peaks are not part of the sample, but of the overall system. System peaks can also appear with other detectors (e.g., UV/DAD).

A: Several additional positive and negative signals in a GPC/SEC system are normal, especially when RI detection is used. If these signals are kept to a minimum and if data evaluation is done properly, they do not influence the functionality of the system.


Identifying ghost/system peaks is easy:

For this a blank sample, which is the eluent used for sample preparation, is injected. It is best to use the same procedure for preparing the blank sample and the samples. If the sample is, for example, filtered, the blank sample should be filtered also. All peaks in the blank sample are system or ghost peaks. An overlay of the blank sample with the sample helps to identify the true sample peaks.

Sample peaks in the low molar mass area may be the result of residual solvent, initiator,

monomer, or other educts from the polymerization process. If the polymerization recipe is known, injection of the solvent, initiator or monomer, each as a single sample dissolved in the mobile phase, helps to assign these peaks. Identification of unknowns is possible by, for example, on-line collection on a Germanium disc followed by IR analysis or by collecting the peaks with a fraction collector.

Sample peaks eluting after the negative signal(s) of the RI detector are highly suspicious in GPC/SEC. This is an indication for a mixed separation mode or for separation dominated by adsorption. As true GPC/SEC must be interaction free, these peaks should not be analysed. Modification of the method (mobile phase, additives, modifiers) or changing to a different stationary phase (other column material) is needed to get a true size separation



and to be able to analyse these samples.

Tip: How do I reduce system or ghost peaks to a minimum?

1. Use only high-quality solvent and exchange it regularly. Degas your solvent and check the mobile phase for impurities.
2. If the recycle mode is used make sure that the solvent is not too contaminated. After solvent exchange wait at least 3-5 column volumes before starting the analysis.
3. Use solvent taken from the solvent bottle to prepare the samples and the blank sample.
4. Use solvent taken from the solvent bottle to clean the autosampler needle. Some autosamplers do that automatically, for manual injection or for other autosamplers it is your responsibility.
5. Maintain your system: exchange frits, autosampler needles and seals regularly or when additional peaks appear.

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.