



# Tips & Tricks: GPC/SEC

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## Q: How do I find the optimum sample concentration and injection volume for my GPC/SEC measurements?

Sample concentration is an essential part of the analysis in gel permeation chromatography/size exclusion chromatography (GPC/SEC). Larger signals and improved signal-to-noise (S/N) ratios can be achieved by increasing the sample concentration or the injection volume. However, a large signal is not always the best answer and does not guarantee accurate molar mass determination (Figure 1).

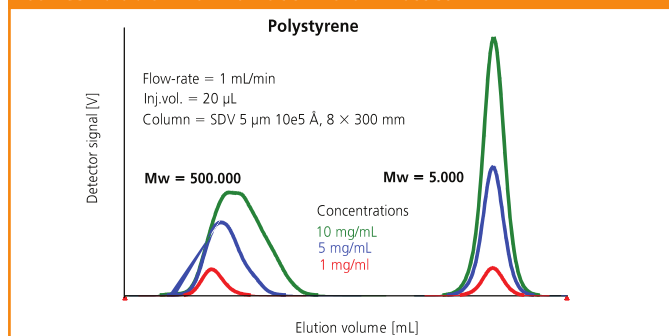
The optimum concentration and injection volume depends on the sample itself. The first thing to find out is if the samples are generally high- or low-molecular weight. Because high molar masses lead to high-solution viscosities, a high concentration produces a very viscous injection band, so that the diffusion process on the column can be hindered. When this happens, higher elution volumes are measured, yielding lower molar masses when a conventional calibration curve is used. Even when absolute methods are used (e.g., GPC/SEC light scattering) the distribution information gets lost. This problem is less pronounced for low molecular weights.

Usually the recommended GPC/SEC sample concentration for broad technical samples lies within the range of 0.1–10 g/L, with injection volumes from 2–100  $\mu\text{L}$ . In practice, 2–3 g/L sample concentration is sufficient and a good value to start with for widely distributed samples (PDI > 1.5). For narrowly distributed samples such as polymer reference materials (PDI < 1.15), 1 g/L is recommended.

The injected mass (injection volume  $\times$  concentration) affects both the peak position (retention volume) and the peak shape. For a given injection volume, lower the sample concentration until the peak position and shape stay constant. Only the peak area should change as a function of the injected mass or concentration (Figure 1).

If the detector signal becomes too small because of the low concentration, increase the injection volume. This guarantees, that the S/N ratio is large enough. In situations of extremely

Figure 1: Elution volume as a function of the sample concentration for various molar masses.



high molar masses (e.g., several millions) inject up to 250  $\mu\text{L}$  of sample solution, at a concentration of 0.1 g/L.

With broad samples or small molecules the concentration range can be increased up to 10 g/L without running into the peak shape or peak position trouble. For smaller molecules the viscosity effect does not play a substantial role.

### Conclusion

- The best way to achieve optimum sample concentration and injection volume with large molecules is to use a smaller sample concentration and increase the injection volume.
- For small molecules it is best to use high concentrations and small injection volumes.
- Choose a large porous column at the first column of a column combination. This helps to reduce the viscosity of the injection band at the beginning of the separation.

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