

Preventing Waste with Overlapping Injection

Introduction

Before a sample injection, the interstitial volume of a GPC/SEC column is typically filled with pure solvent. Normally, this solvent must clear the column before the exclusion limit, after which the first sample components elute. Once the late-eluting compounds appear and total permeation is achieved, the interstitial column volume is refilled with pure solvent (Figure 1). This process creates redundant periods where no sample is eluted, unnecessarily lengthening analysis time and increasing solvent consumption.

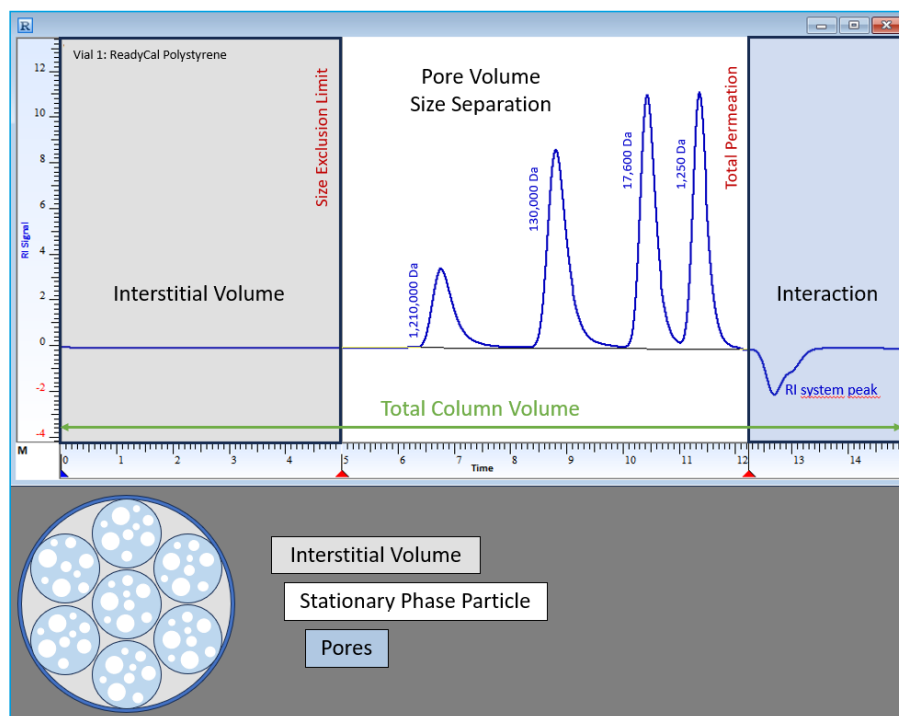


Figure 1. Graphical representation of total column volume, the interstitial volume, and the pore volume of a typical analytical GPC/SEC column with a length of 300 mm and internal diameter of 8 mm

One option to prevent this waste is to use the Overlapping Injection function in Agilent WinGPC. This feature makes it possible to inject a sample before the previous sample has completely eluted, saving solvent and analysis time.

Experimental

GPC/SEC columns with polymer gel stationary phases typically have interstitial volumes of around 30%. This value would correspond to approximately 5 mL in an analytical column with a length of 300 mm and an internal diameter of 8 mm. If a flow rate of 1 mL/min is applied to this column, no sample components would elute within roughly the first 5 minutes after an injection. As a result, it is possible to inject a sample a few minutes before a previous run has finished without risking loss of sample or resolution.

Figure 2 shows the WinGPC raw data window highlighting two different injections, one after the other (with a third, later injection that is not highlighted here). Each injection is indicated by an injection symbol (a blue triangle at the bottom of the window) and the sample name at the top.

Before the system peaks of "Vial 5: Sample 3" have eluted, the next sample, "Vial 6: Sample 4", is already injected at approximately 23 mL elution volume. Data evaluation for sample "Vial 5: Sample 3" is unaffected by the second injection. Baseline limits (compare the two red triangles) and integration limits can still be set as required by national and international GPC standards (e.g., ISO 13885.7).

The green and red areas in the elugram show the total required volume for "Vial 5: Sample 3" and "Vial 6 Sample 4", respectively. Around 8 mL mobile phase is saved per injection in this example. A further reduction in solvent is possible, as there is more than sufficient baseline area to set the baseline limits properly.

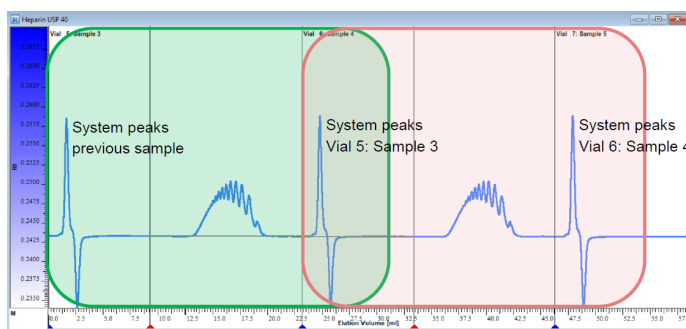


Figure 2. Overlapping Injection: "Vial 6: Sample 4" (red box) is injected before "Vial 5: Sample 3" (green box) is completely eluted. The displayed elution volume is valid for "Vial 5: Sample 3"

Conclusion

In the example described in this technical overview, approximately 30% solvent can be saved using the Agilent WinGPC Overlapping Injection software feature. Overlapping Injection is a feature that can be applied with all types of columns, independent of length and diameter; all that is required is a shortening of the injection interval. Resolution and analytical conditions such as flow rate or column loading are unaffected by the technique.

Reference

1. Application #27: Overlaid Injection. PSS WinGPC Newsletter 01, PSS publication number 30102, **2005**.