

Sample Viscosity Effect and Positive Displacement Autosamplers

One of the major advantages of using the Positive Displacement method of sampling is that the mechanism is independent of sample viscosity. On the other hand, the FilterCaps, by their restrictive nature, can slow the flow of sample into the Autosampler's needle and transfer tube. This restriction can possibly cause poor injection precision or make the instrument stall if highly viscous samples are used. Figure 1 shows the results of a comparison study showing the effect of sample viscosity on injection precision when using the standard cap versus the FilterCap. The sample was a 0.01 mg / mL solution of Butyl Paraben made up in methanol and in five different solutions of methanol and glycerin with solution viscosities ranging from 0.55 cp to 78.23 cp as shown in table below. Six injections were made with each sample, first from vials fitted with the standard cap and then from vials fitted with the FilterCap. The Relative Standard Deviations for these injections were plotted against the sample viscosity as shown in Figure 5. The last sample, dissolved in the 80% Glycerin in Methanol solution, caused the Autosampler to stall, resulting in no injections and are thus not plotted on the graph. The injection precision for each solution was well below the Autosampler's specification of $\pm 0.5\%$ up to a sample viscosity of around 37.5 cp. Above this viscosity, the precision becomes poorer because solution transfer becomes too slow to fill the loop in a consistent manner. Higher viscosities will cause the instrument to stall completely. Fortunately, the sample viscosity for most HPLC applications are well below 30 cp (typically less than 5 cp).

Experimental Conditions, Viscosity Study (Used to generate Figure 1)

Column:	Spherisorb ODS II, 5 μ m, 250 mm \times 4.6 mm ID
Mobile Phase:	70% Methanol in Water
Flow Rate:	1.5 mL / min.
Detection:	UV @ 250 nm
Injection Size:	10 μ L
Sample:	Butyl Paraben, 0.01 mg / mL

Sample Solvents:	Solvent	Viscosity (cp 24 °C)
	100% Methanol	0.55
	10% Glycerin in Methanol	4.43
	20% Glycerin in Methanol	15.12
	40% Glycerin in Methanol	36.12
	60% Glycerin in Methanol	37.49
	80% Glycerin in Methanol	78.23

FIGURE 1: PRECISION vs SAMPLE VISCOSITY
Standard Cap and FilterCap

