Using Guard Columns and Retention Gaps in GC (Part 1)

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increased the sample components will start to move (there is very little retention …that's why it's called a retention "gap"). When reaching the analytical column, the components will focus in the stationary phase resulting in a narrowing of injection band width (Figure 1). As these retention gaps are mainly used for on-column injection, the inside diameter is usually 0.32mm up to 0.53mm since the needle of an on-column syringe must be able to enter the retention gap. For coupling the retention gaps to the analytical column, we need generally coupling devices that can deal with different diameter capillary tubing.

Retention gaps and splitless injection

While on-column injection minimizes discrimination and provides the best quantitative data, especially for thermolabile components, it can be challenging to perform. Many laboratories will choose a splitless method for ease of use. For splitless injection we generally do not require a retention gap. The sample is injected in a hot injection port, evaporated, and transported with a carrier gas flow of approximately 1mL/min. into the capillary. The amount of solvent vapor that enters the column per unit time is much smaller than with on-column injection. Although with splitless injection the oven temperature is also 10-15°C below the boiling point of the solvent, there is little chance of the solvent condensing. The high concentration of solvent entering the capillary column will cause a strong focusing effect for the components, generating a narrow injection band. If, in splitless injection, a method is used where the initial (injection) oven temperature is much lower than the boiling point of the solvent, the risk of solvent condensation (forming a liquid plug) will increase. This can cause unwanted broadening of the injection band. Coupling a retention gap will also fix this problem.

Wettability of the retention gap

An important factor for good performance is the wettability of the retention gap surface. It is critical that the solvent spread evenly over the surface. This means that nonpolar solvents (hexane, methylene chloride, isooctane, benzene) require non/intermediate deactivated retention gaps and more polar solvents (methanol) will require polar deactivated retention gaps. If the polarity of the retention gap and solvent do not match, the solvent will form droplets inside the capillary. The carrier gas will "push" this droplet along the retention gap into the analytical column. The result is a broadened injection and possibly even peak splitting.

Retention gaps for large volume injection

Instead of injection of 1µl on a 1-2m retention gap, one can also inject much larger amounts on much longer retention gaps. Here we talk about large volume injection technique where retention gaps of 8-10m are used. Such retention gaps can be loaded with 100-200µl of sample. Injection must be slow to allow the solvent to evaporate while passing through the retention gap. With large volume injection, detection limits can be reduced by a factor of 100. The technique requires some skill to optimize all the injection parameters. Additionally, the large volume retention gaps do pollute relatively quickly due to the large amounts of sample introduced.

Guard columns and retention gaps are useful tools to the practicing chemist and it is important to understand the difference between them. In Part 2 of this article, we will review guard columns and discuss a new segment coating technology that allows retention gaps and guard columns to be built directly into the analytical column tubing. This new technology eliminates column coupling, substantially reducing analytical problems related to leaks and dead volume.

1 Grob, K., Journal of Chromatography 237:15 (1982). 2 Hinshaw J., LC•GC Europe 17(9): 460–466 (2004).

more to come!

See the next issue of *the Restek Advantage* for Part 2 of this article.

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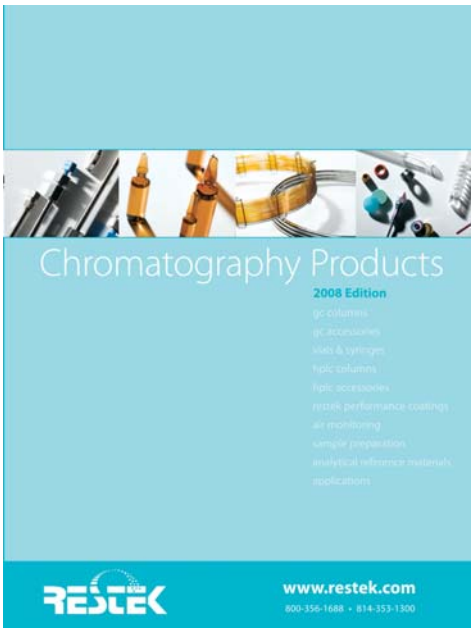

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