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# Using Guard Columns and Retention Gaps in GC (Part 2)

Jaap de Zeeuw, International GC Consumables Specialist, Restek Corporation



Guard columns and retention gaps are used widely in gas chromatography (GC). Many users have difficulty understanding the difference between these two products, even though there is a significant difference in application. In Part 1 of this article we reviewed retention gaps, which mainly are used for focusing the sample components when introducing a large (liquid) sample directly onto the column. In contrast, guard columns are used to protect the analytical column from contamination. Guard columns and retention gaps both must be coupled to the analytical column, and this connection introduces a potential point of risk. A new approach is to integrate the retention gap directly into the analytical column tubing. By applying a “segment” coating technology the stationary phase can be deposited only in a certain part of the column allowing a deactivated section at the beginning. Column coupling is not required and maintenance is greatly simplified. Here we will review guard columns and discuss the new segment coating technology.

## Use of guard columns

The purpose of using guard columns is to protect the analytical column from contamination since the sample that is introduced is not always pure. Although the best chromatography is obtained with “clean” samples, the practical situation is that sample clean-up procedures are minimized and relative “dirty” samples are introduced onto the column. Samples can contain particulates, heavy components, derivatization reagents, ionic residues, acids, bases... all these compounds can interfere with the stationary phase and they will influence the separation process. Usually the degradation of column performance is a slow process but it will happen.

Most of the time the impurities accumulate in the first meter(s) of the column and by cutting off this section adequate separation is restored. Many users choose to connect a guard column in front of the analytical column. Such a guard column is deactivated and can be trimmed when contaminated and eventually replaced. Depending on the application, guard columns have a lifetime of 1 week up to 6 months. One has different choices for guard columns; a guard column can consist only of deactivated capillary, or it can be a coated capillary.

*Deactivated capillary tubing:* Deactivated fused silica tubing can be purchased by the meter and then a defined length can be coupled in front of the analytical column. Upon contamination, a section of the guard column is removed. When the whole guard is “consumed” a new guard column can be coupled. The disadvantage of cutting parts off of the guard column is that the column becomes shorter and this may affect retention times. However, if a similar length is always cut from the guard column, the change in retention time becomes very predictable. A deactivated guard column will also result in band focusing. If the injection is not optimal, there will be a focusing effect similar to that of a retention gap.

*Coated capillary tubing:* As the guard column needs to prevent contamination of the analytical column, a coated guard column can help as it has both the surface deactivation and also the stationary phase layer. The easiest and most economical way of using coated guard columns (or precolumns) is to buy two analytical columns. One we will use as a separation column and the second one will be used to make coated guard columns. From this second column we will cut 2m sections and couple a section in front of the analytical separation column. We can run our samples until contamination affects peak shape/response and then we can replace the guard with a new 2m section.

The system we have created will produce reproducible retention times as we always will replace the entire 2m coated guard column. Since the stationary phase is the same on the guard as on the analytical column, there will be no surprises. The coated guard column also will allow more aggressive samples/more contamination before it will give up. Lastly, we are able to cut 15 coated guard columns from a full 30m analytical column...that’s also economical! However, if using a coated guard column, there will be no focusing effects.

Continued on page 31.

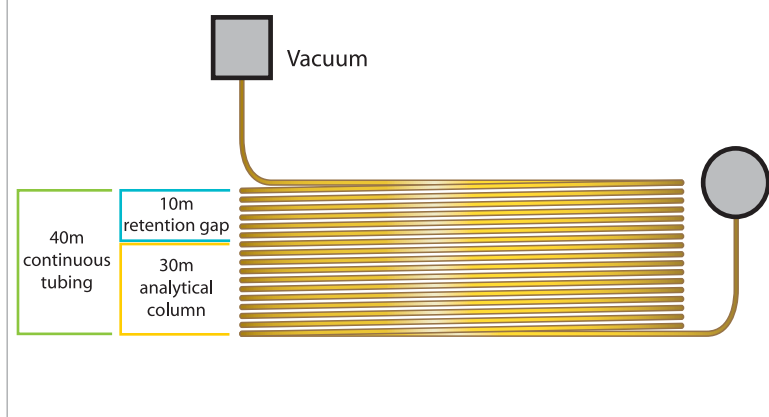
## Using Guard Columns and Retention Gaps in GC (Part 2) *Continued from page 2.*

### Segment coating technology eliminates problematic connections

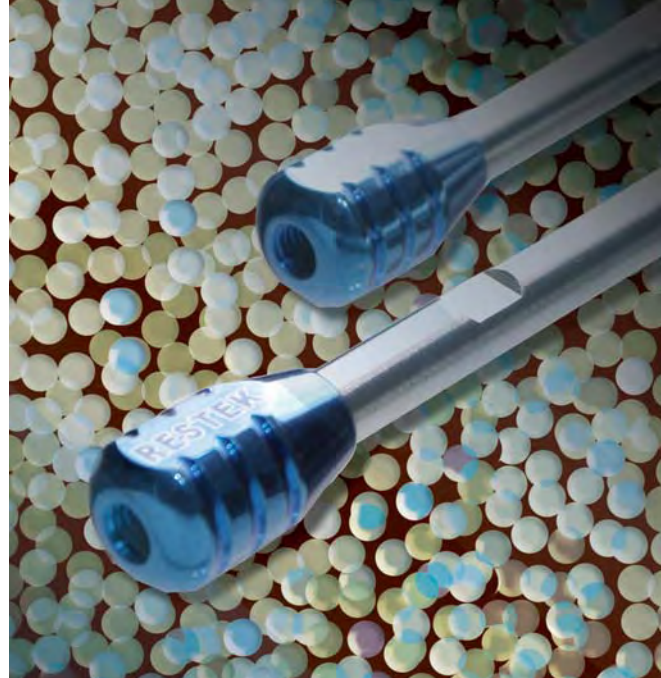
Both retention gaps and guard columns must be coupled to the analytical column. While there are several types of effective coupling devices, all can create dead volume and can be a potential source of leaks and reactivity. Segment coating technology allows the retention gap or guard column to be built directly in the same piece of tubing as the analytical column, eliminating the connector and associated risks. This technology, available from Restek, is termed Integra-Guard™ or Integra-Gap™ and is based on the static coating method. In this process the capillary column is filled with a coating solution of stationary phase in a volatile solvent. The column is sealed on one end and on the other side a vacuum is applied. The solvent is evaporated and the dissolved polymer is deposited on the inside deactivated wall of the fused silica column. The static coating method allows columns to be coated by segment. When filling, for example, a 40m capillary with the coating solution, only 30m are filled. The first 10m remain uncoated, having only the deactivation treatment (Figure 1). This method deposits the stationary phase only in a designated portion of the capillary, creating the Integra-Guard™ or the Integra-Gap™. The advantages of this technology are clear: eliminating the connector removes a potential source of leaks and reduces dead volume. Additionally, maintenance is faster and simpler since there is no manual connection to make.

Guard columns and retention gaps are useful tools to the practicing chemist, and it is important to understand the difference between them. While they help protect analytical columns and focus samples, respectively, they are also a source of potential problems, such as leaks. Segment coating technology offers a better solution—integrated columns containing both the guard or gap section and the analytical column together in a single piece of tubing. These Integra-Guard™ and Integra-Gap™ columns are a simple, effective solution; they eliminate the risks of a separate connection and provide stable, accurate data.

**Figure 1** Static coating allows Integra-Gap™ integrated retention gaps to be built directly into the analytical column tubing.



For more information on Integra-Gap™ technology, see “Selecting a GC Column for Glycerin in Biodiesel” on page 10.



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