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Erratum

The heading of Figure 1 on page 8 of the 2007.03 issue of the Restek Advantage incorrectly describes the column internal diameter as 0.18mm. The correct internal diameter is 0.32mm.

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

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. Retention gaps mainly are used for focusing the sample components when introducing a large (liquid) sample directly onto the column. Guard columns are used to protect the analytical column from contamination.

When using a retention gap system, the retention gap will also act as a guard column, but its primary function is to create a focusing effect.

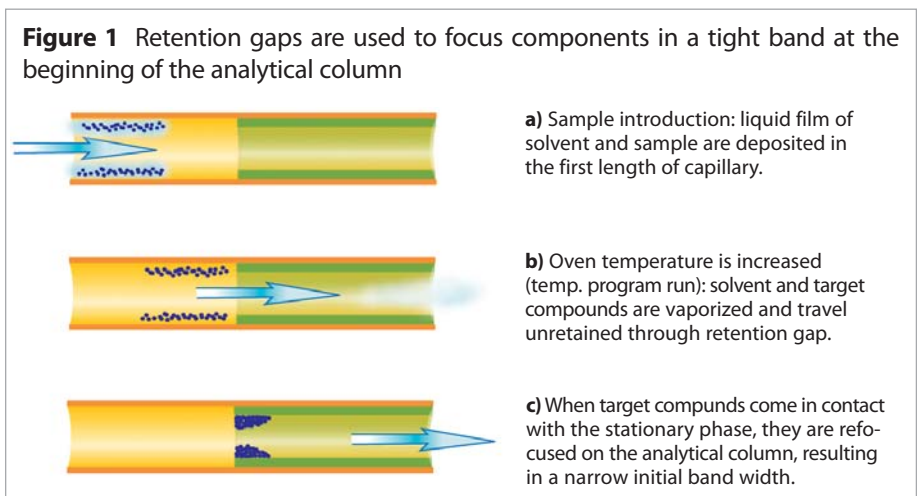
Guard columns and retention gaps both must be coupled to the analytical column, and this connection introduces a potential point of risk. A new approach is to integrate the retention gap directly into the analytical column. By applying a “segment” coating technology, the stationary phase can be deposited in a certain part of the column allowing a deactivated section at the beginning. Column coupling is not required, and maintenance is greatly simplified. In Part 1 of this article, we will explore retention gaps and build a foundation for a comparison to guard columns. In Part 2, we will review guard columns and discuss the new segment coating technology.

Use of retention gaps

In today’s laboratory, GC methods must be simple, fast, and low detection limits are required. Besides that, sufficient precision must also be obtained. It all starts by introducing the sample in the smallest possible injection band and making the band migrate through the capillary with minimal loss of the target components. With on-column injection, a liquid sample is directly introduced into the capillary column as a liquid while the capillary column is kept at a temperature 10-15°C below the boiling point of the solvent. During this process, the sample components are spread in an unreproducible way over the first 20-100cm of capillary while the solvent is evaporating. Parameters like injection speed, carrier gas flow, temperature of solvent and column, type of solvent and pressure all will affect the injection band width. Additionally, when nonbonded stationary phases are used, the direct contact with liquids will result in a distortion of the stationary phase film and very short column lifetime. The majority of today’s stationary phases, like the Rtx® and Rxi® phases, are immobilized by cross- and surface bonding techniques.

For proper application of the on-column injection technique, the use of retention gaps is essential.^{1,2} The retention gap consists of a 1-3m length of deactivated capillary that is positioned in front of the analytical column. All the processes described will still take place, but now the components are distributed over the retention gap. When the oven temperature is

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

Continued from page 2

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).

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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.

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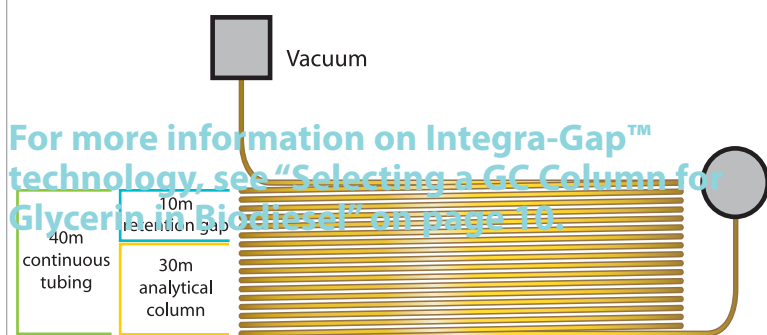
Using Guard Columns and Retention Gaps in GC (Part 2) *Continued from page 2.*

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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