The Impact of Using Guard Columns in Gas Chromatography

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Gas chromatography is a technique which can be used for a wide class of sample types. If the components to analyse can be transferred in the gas-phase by increasing the temperature, then there is a high probability that they can be analysed by gas chromatography.

Once in the gas phase, analytes will generally not do much damage to the GC column. The challenge often experienced, is that the sample matrix contains all kinds of materials that are also introduced into the system. Extensive sample preparation can be done to clean up the samples. Practically, however there is a compromise with the number of analyses that can be perform with minimal system maintenance and the intensity of sample preparation efforts.

Figure 1

Cyclo splitter

Straight liner

Figure 1: Liners for optimal injection quality: Use deactivated glasswool if no interactions with compounds are expected; If interactions are expected or observed, use the cyclosplitter.

The analytical separation column will contaminate and the impact will be seen by lower response, broader peaks, tailing peaks, retention time shifts and increased background signal. Even ghost peaks and decomposition can occur. Usually the degradation of column performance is a slow process but it will happen. There are several ways to make sure we protect the analytical column for contamination:

- Reduce the amount injected
- Use of liners with glass wool
- Use of guard columns or precolumns

Reduce the amount injected

The degree of contamination is linear with the amount of sample that is introduced. If you can measure your (trace) components with sufficient high signal/noise ratio, you may consider injecting a smaller sample volume or dilute the sample 2, 3 or more times.

It is better to inject less or inject a diluted sample, than to use higher split-flows. The challenge is always to minimize the contamination of inlet section. Using a higher split flow will dilute the volatiles, but the matrix may build up activity in the liner.

Use of liners with glass wool

Glass wool has been used for a long time in liners since it was discovered that glass wool filled liners performed better then empty liners. The main benefit is the reproducibility of injection which is typically within 1%. With glass wool in place there is sufficient heat capacity available to homogeneously evaporate the solvent and analytes.

A second important benefit is that the glass wool traps a large part of the matrix. Frequently replacing the glass wool plug or the whole liner is already a common practice when running challenging samples.

A recent study [1] demonstrated that good data for splitted injection is obtained using liner configurations as shown in Figure 1. For analysing samples that have no issues with the glass wool a straight liner with glass wool works fine. If the matrix

or components show interactions with glass wool (e.g., water, amine, pesticides), then good results can be obtained using the cyclo splitter type liner.

Use of guard columns

Most of the time impurities accumulate in the first meter(s) of the column. As the separation process in the capillary starts here, the unwanted interactions that take place will immediatey impact the initial band width. By cutting off the contaminated section, the sample will be introduced in a "clean" section and we obtain similar separation as before.

Many users, choose therefore to connect guard columns in front of the analytical column.

These protect the analytical column from contamination. Guard columns are made of a piece of deactivated capillary, coated or uncoated, that is connected in front of the analytical column.

Such a guard column is deactivated and can be trimmed when polluted and eventually replaced. The type of guard column can be chosen based on length, internal diameter, type of deactivation, or stationary phase coating.

Depending on the application, guard columns have a lifetime of 1 week up to 6 months. Deactivated fused silica tubing can be purchased per meter of which a defined length is 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 of the guard column is that the column becomes shorter resulting in changed retention times; however this is quite predictable.

A bigger challenge is to make the coupling. The best generic results are obtained using a universal PressTight® connector. Make a 90-degree cut, wet the column end with some methanol and push it into the PressTight . By pushing it, an optical "ring" is observed (see Figure 2), which is the actual seal made by the polyimide and the surface. An alternative solution that eliminates column coupling is to use integrated guard columns.

Figure 2

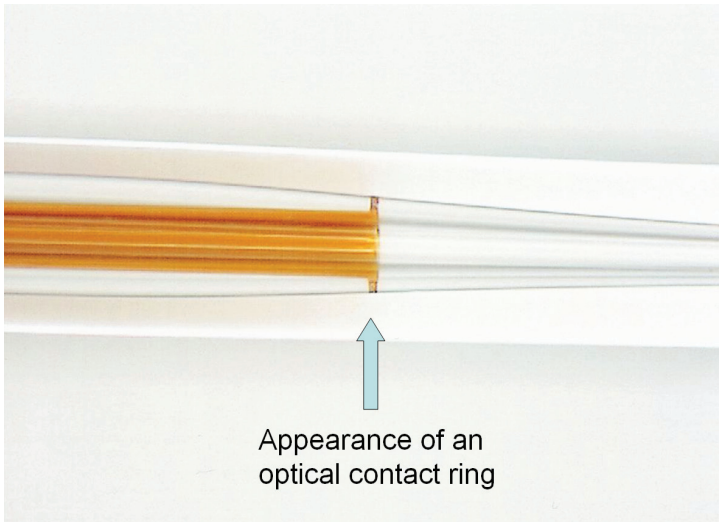

Figure 2: Enlarged view of connection of a PressTight. The optical ring is the actual seal with the inside of the PressTight.

Figure 3

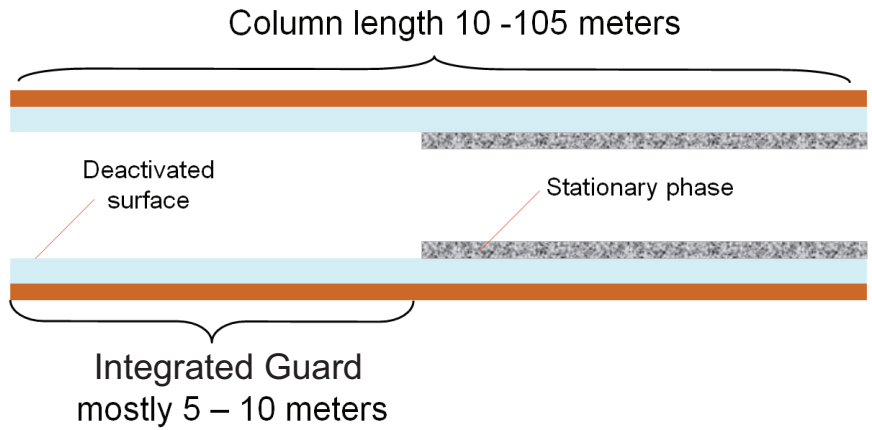

Figure 3: Schematic of an integrated guard column.

Integrated guard columns

Integrated guard columns were first introduced around 1990 and have been widely used since. Integrated guard columns are prepared by coating only the last section of the column. This technique is called "segment" coating and can be read about in more detail in reference [2].

As there is no coupling these guard colums offer several advantages:

- No connection to make, saves time
- No leaks, improved stability and more accurate data
- No dead volumes/activity or thermal mass
- Easy in maintenance; integrated solution

Such integrated guard columns are typically 5–10 m in length and can be made from most standard stationary phases and all column diameters (Figure 3). It is important is

Figure 4

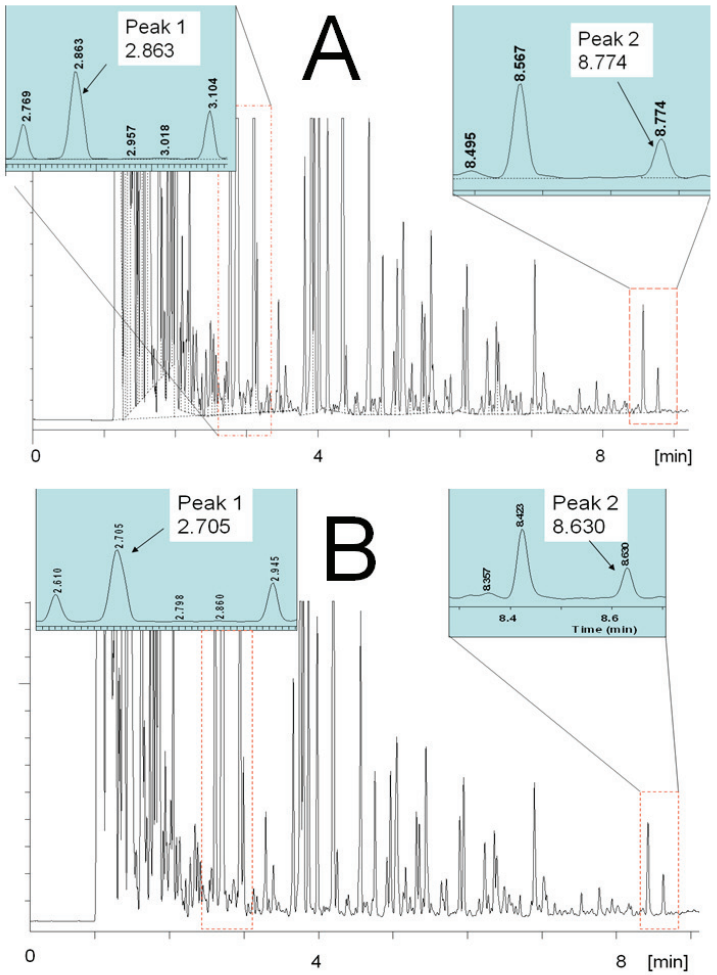

Figure 4: Impact of cutting a guard column. Sample: gasoline; Oven: 60 °C, 1min. ⇨140 °C, 10°C/min ⇨ 220°C, 50°C/min., 5 min; Carrier: He, 1.9 mL/min, constant flow; A: retention on 35 m (5 m integrated guard); B: retention after cutting 3 coils; 33.2 m (3.2 m integrated guard)

to make a clear mark on the column where the guard column starts. Not every stationary phase can be used as a integrated guard solution. The column needs to be deactivated first, followed by a second deposition of the stationary phase layer. There are different phases that can be manufactured with an integrated guard. Working with integrated guard columns allows the user to cut a piece periodically from the inlet, without the trouble of making a coupling. Such integrated guard columns can be made in different length.

Table 2

Column	Rt peak 1	ΔTr	Rt peak 2	ΔTr
30 m + 5.0 m guard	2.863		8.774	
30 m + 4.4 m guard	2.809	0.054	8.726	0.048
30 m + 3.8 m guard	2.759	0.050	8.678	0.048
30 m + 3.2 m guard	2.705	0.046	8.630	0.048

Table 2: Impact of cutting guard on retention time in temp. progr. analysis

Impact on retention times

When a guard column is used, and completely replaced with a new guard of the same length, the retention times will be exactly the same as before. When a piece of the guard column is cut, the total column length changes and one can expect a reduction in retention time. Because the guard column has no stationary phase, it will not add a lot to the retention. As it gets shorter, the components will travel faster through this part resulting in a shorter retention time.

To demonstrate the impact on retention time, a 30 m x 0.25 mm column, film of 0.25 µm with a 5 m integrated guard column (total length = 35 m) was used, of which systematically similar sections were cut off. The column was operated with helium under a constant flow of 1.9 mL/min. A gasoline mixture was analysed using a temperature program (see Figure 4). After analysis, one coil (60 cm) was cut from the inlet, while keeping the helium flow rate the same and the gasoline was analysed again. This was repeated twice more. The retention times of an early eluting component (peak 1) and a late eluting component (peak 2) were measured and listed in Table 2.

Using constant flow, a systematical decrease of retention times was observed. For the late as well as for the early eluting components the components eluted systematically three seconds earlier. This difference was the same when another coil was taken from the guard column. Practically, this means one can easily estimate retention times when guard columns are shortened. To make it predictable one must always cut the same length when maintenance is performed. The absence of a coupling device simplifies the maintenance in routine analysis. Also in the case of

MS-applications many can benefit from elimination of the column coupling. The same experiment was also performed without a guard column. The reduction of retention time by cutting coils of the inlet was approximately 2.5 times larger as observed with the guard columns. This was to be expected as the guard section cannot add as much to retention as a coated section will.

References

[1] J. Cochran, in preparation
[2] J. de Zeeuw, *Chromatography Techniques*, jan/feb. 2008.

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