Hints for the Capillary Chromatographer

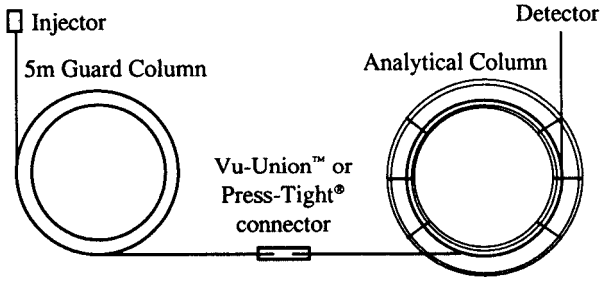
The Benefits of Guard Columns for Capillary Gas Chromatography

The use of guard columns has been commonplace in high performance liquid chromatography for many years. Their use as safeguards to protect the analytical column from highly retentive compounds and particles is well understood. It has only been in the past few years that the benefits of guard columns have been associated with capillary gas chromatography. Although guard columns prolong the life of capillary columns and protect them from sample contamination, they are not widely used. Understanding the basics of guard columns helps to dispel confusion and apprehension about their use.

What is a guard column?

A guard column for capillary chromatography is a short length of deactivated, uncoated fused silica tubing that is placed between the injection port and the analytical column (Figure 1).

Figure 1 ' A Guard Column Connected to an Analytical Column

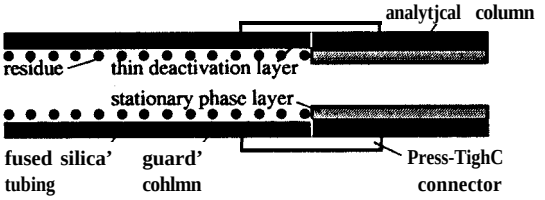


What advantages do guard columns offer?

Prolong column lifetime

A guard column protects and prolongs the lifetime of an analytical column in several ways. It traps non-volatile residue and prevents it from collecting at the head of the analytical column. This non-volatile residue may be very high molecular weight organic compounds, inorganic salts, or particulate materials. If these contaminants enter the analytical column, they can cause adsorption of active compounds and loss of resolution. When this contamination begins to affect sample analysis, a small section of the analytical column must be removed to restore proper performance. However, each time a section of the analytical column is removed, retention times change, some resolution is lost, and column length is decreased, eventually resulting in a useless analytical column. By trapping this contamination in the guard column, the analytical column remains the same length and stays cleaner longer.

Figure 2 - With a guard column installed, the interaction time is minimal between the sample & residue. Thus, decreasing maintenance requirements.



Sample residue deposits approximately l-meter into the column inlet. Without a guard column, dirt is deposited onto a stationary phase, causing the sample to partition in and out of this dirty region. Adsorptive effects are more likely to occur. Guard columns stop dirt, but do not retain the sample since there is no stationary phase. The interaction behveen the residue and subsequent sample injections is minimal, and more injections can be performed before maintenance is required.

Decrease maintenance requirements

Since there is no stationary phase on the guard column, the amount of time the sample spends in it is minimized. This reduces the interaction between sample components and the contamination from non-volatile residue (Figure 2). Therefore, guard columns allow more injections to be made before residue interferes with analytical results.

Improve resolution

Many analysts are reluctant to use guard columns because they believe that they will lose resolution. In fact, guard columns actually increase separation efficiency. The guard column acts as a retention gap to help focus the sample at the head of the column. When a sample is injected, it first exists as vapor and aerosol. Without a guard column, the vapor begins to partition in and out of the column's stationary phase. The aerosol portion of the sample does not partition in the phase and moves out ahead of the vaporized sample. This results in broader, less efficient peaks and, in extreme cases, can cause split peaks. Since a guard column is not coated with stationary phase, there is no interaction with the vaporized sample or the aerosol. They move along together in a tighter band. The aerosol vaporizes in the guard column so that when the sample reaches the coated column it is completely vaporized. This produces sharper, more efficient peaks, as shown in Figure 3. Table 1 shows the results of analyzing 2,6-dimethyl-phenol on a 30m, 0.53rnm ID, l.0um Stabilwax column with and without a guard column. The efficiency of the 2,6-dimethylphenol peak was measured in each case and the results show a 3.1% increase in efficiency with the guard column.

Figure 3 - Guard columns increase separation efficiency because the aerosol completely vaporizes before it reaches the coated column.

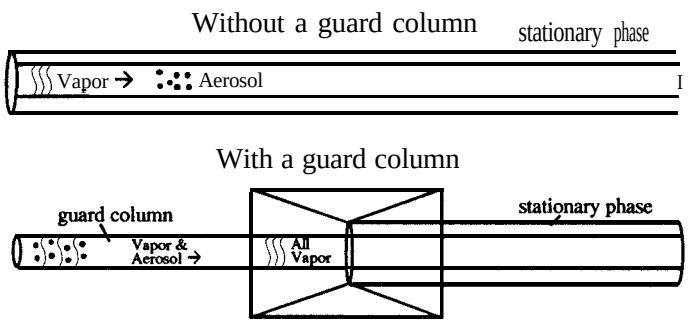


Table I - Column Efficiency Data
(1µl split injection of 2,6-dimethylphenol)

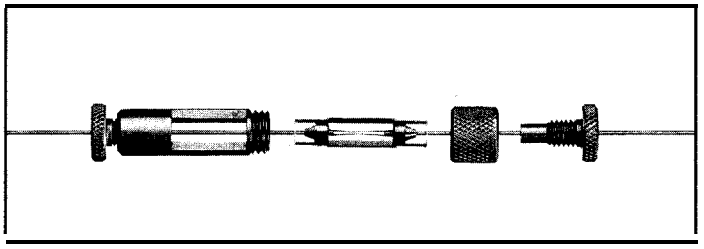
Without Guard Column	*With Guard Column*
Total plates = 51500	**Total plates = 53100**
Plates/meter = 1716	**Plates/meter = 1770**

3.1% increase in plates

How is a Guard Column Connected to an Analytical Column?

A Press-Tight Connector is commonly used to connect a guard column to an analytical column. However, the higher temperature polyimides used by most capillary manufacturers today do not seal well with Press-Tight connectors. They have been known to disconnect unexpectedly when used at temperatures exceeding 300°C. A new type of connector has become available that has both the benefits of Press-Tight connectors and the dependability of fermle seals. The connector, called a Vu-Union, incorporates ferrules that seal inside a glass union to provide a dead volume free, inert sample pathway. Figure 4 shows the primary Press-Tight sealing mechanism and the secondary ferrule fail safe sealing mechanism.

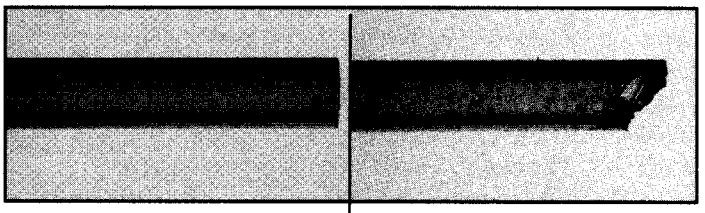
Figure 4 - A disassembled Vu-Union shows the primary and secondary sealing mechanisms.



The metal housing holds the tapered glass insert securely. Two thumb screws are positioned at the end of each union to tightly compress the ferrule in the insert. The primary, low dead volume seal occurs as the column ena are compressed in the press-tight taper region.

Whether you are using a Press-Tight connector or a Vu-Union, it is important to cut the column end squarely with a ceramic scoring wafer or sapphire blade. Pointed cutting devices are not recommended since they create a crevice on the side of the tubing and result in leakage. Once a square cut has been made, insert the fused silica tubing until you see a brown sealing ring (Figure 5). The presence of a uniform brown ring around the connector is a good indication that the connection will be dead volume free and not contribute to band broadening.

Figure 5 - A square cut is necessary to get a good column seal.



Square cuts made by sapphire scribes or ceramic wafers show the presence of complete sealing rings.

Cuts made by pointed devices allow carrier gas to leak around the column end.

When should a guard column be replaced?

As the guard column becomes contaminated with non-volatile residue, the performance of the chromatographic system will begin to deteriorate. This is normally exhibited as a drastic decrease in the response of active compounds or peak tailing.

How often must a guard column be replaced?

The life expectancy of a guard column depends on the length of the guard column, the amount of non-volatile residue in the samples, and the number of samples run. When analyzing dirty samples, the guard column becomes contaminated quickly. Normally, contamination deposits in the first .5 to .8 meters of the guard column. If a short guard column (l-meter or less) is used, it must be completely replaced when it becomes overly contaminated. If a longer guard column (5-meters) is used, the contaminated section can be removed without reconnecting the analytical column.

How long should a guard column be?

A guard column should be long enough to keep non-volatile residue from entering the column, but short enough so that the analysis time is not significantly increased. Five meter guard columns are more cost effective, reduce the frustrations of making the connection between the guard column and analytical column, and are preferred by most analysts. If a very long guard column 10-meters) is used, the residence time of the sample components increases, resulting in longer analysis times and skewed peak shapes:

Guard columns help prolong the life expectancy of capillary columns and are an excellent, economical alternative to column replacement. Analysts working with dirty samples find that the use of guard columns significantly reduces column replacement costs and time lost in troubleshooting column contamination problems.

For more information, call technical service at 1-800-356-1688, ext. 4, and request your free copy of *A Guide When Injecting Dirty Samples.* ■