

please note

For superior inertness, try our Siltek® guard columns!

See **page 28** for details.

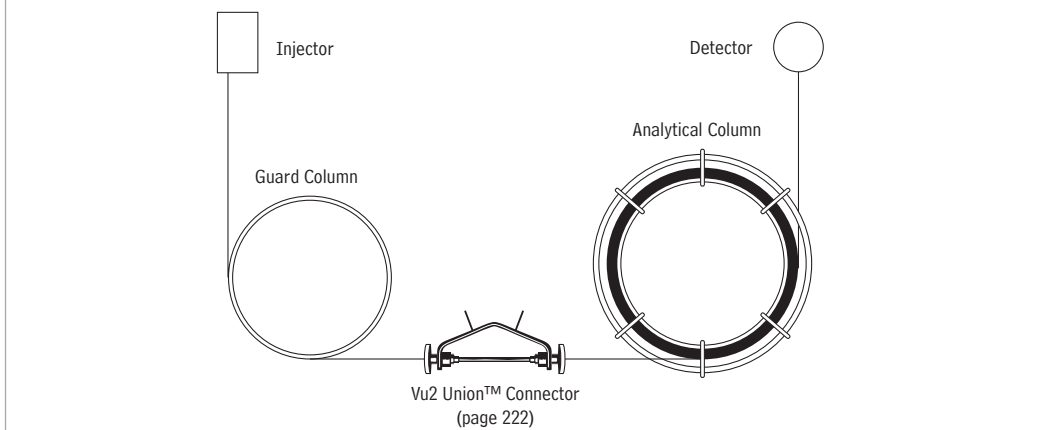
Having trouble making a leak-free connection? Try our "built in" Integra-Guard™ columns!

See **page 30** for details.

Guard Columns and Retention Gaps

Guard columns and retention gaps are widely used in gas chromatography. The concept of the guard column is to trap nonvolatile material at the head of the column, not allowing the material to reach the analytical column. The concept of the retention gap is to help focus the compounds transferred from the inlet to a small band at the head of the analytical column in order to reduce chromatographic peak broadening. Both concepts (trapping nonvolatile material and refocusing the target analytes) take place when a piece of deactivated tubing is connected to an analytical column as in Figure 1.

Figure 1 A guard column connected to an analytical column



Analyte Focusing

There are two injection techniques where the retention gap is used to help focus target analytes at the beginning of the analytical column, cool on-column injection and splitless injection.

For cool on-column injection, the use of a retention gap is to help focus the sample components when introducing a liquid sample directly into the retention gap. The cool on-column injection is performed by inserting the syringe needle into the retention gap (this can be accomplished with a 0.53mm ID retention gap and a 26s gauge syringe) and transferring the liquid sample directly into the retention gap. The injection is made with the injector and column oven set below the boiling point of the solvent. As the solvent is evaporated, the volatile target analytes migrate in the solvent towards the analytical column, and the heavier analytes will be distributed over the retention gap. As the oven temperature increases, the target analytes vaporize and move unretained down the retention gap column until the compounds reach the liquid stationary phase of the analytical column. At this juncture, the target analytes are trapped/focused by the liquid phase forming a narrow injection band.

did you know?

We test our guard columns/transfer lines with the Grob test mix to ensure high inertness.

The retention gap may also be useful in hot vaporization injections when the transfer of the compounds from the inlet to the column does not form a focused band. Typical applications include water injections or injections using small ID columns, where split or tailing peaks would indicate an unfocused band. In these applications, the target analytes are trapped in a nonuniform or longitudinally diffuse band at the head of the retention gap (Figure 2a). As the oven temperature is increased, the solvent and target compounds are vaporized and move unretained through the retention gap (Figure 2b). When the target compounds come in contact with the stationary phase, they are refocused in a narrow band (Figure 2c), improving the chromatography.

Protecting the Analytical Column

The concept of a guard column is to protect the analytical column from becoming contaminated with nonvolatile compounds. The guard column is used to retain nonvolatile material, usually in the first 10-20cm, not allowing this material to elute onto the liquid phase of the analytical column. As the oven temperature increases, the more volatile target compounds vaporize, elute down the guard column, and refocus at the head of the analytical column without interference from the nonvolatile material.