Gas Chromatography Problem Solving and Troubleshooting

Question:

Sometimes I experience peak shape or size problems shortly after changing the glass wool in the injector liner. Other times, the problems do not occur for a long time. When should the glass wool be changed, and what is the best method to pack the liner?

Answer:

Most problems with a new liner packed with glass wool are related to the amount or location of the glass wool. There are two major benefits provided by glass wool. One is that it filters or traps the nonvolatile portions of the injected sample and prevents them from entering the column. Pieces of septa or other foreign particulates are also trapped by the glass wool. Its other benefit is that it remixes the injected sample after vaporization by the heated injector. This often improves reproducibility and increases peak size of the later eluting (i.e., high boiling) compounds. It is normal for a lower percentage of the high boiling sample compounds to enter the column than the lower boiling (more volatile) sample compounds. This is called injector discrimination. Injector discrimination is often reduced by placing glass wool in the injector liner. The most pronounced impact occurs when split and Megabore direct injectors are used. Glass wool is usually not recommended for use with splitless injectors unless the samples are particularly dirty. Using glass wool with splitless injectors often causes any peaks eluting near the solvent front to increase in width. Also, active compounds (those containing -OH or -NH groups and some aldehydes) may exhibit some peak tailing or loss of size.

Vaporized sample is transported by the carrier gas from the liner into the column. As the carrier gas travels through the glass wool, it is mixed due to the numerous flow paths through the mass of glass wool. This helps to mix the vaporized sample and makes it more uniform or homogeneous, which often improves the sensitivity (i.e., increases peak size) of the later eluting compounds. Split injector liners with flow disruption features are designed to mix the vaporized sample in the same manner as liners packed with glass wool. Split liners packed with glass wool often provide the same benefits as inverted cup, fitted, or laminar flow liners.

The location, amount, or packing density of the glass wool influences peak shape and injector discrimination. There is not complete agreement concerning the best location and amount of glass wool in a liner. The best position and amount is often dependent on the compounds being analyzed. Experimentation may be useful when trying to optimize peak shape and size. Most liners are designed to hold the glass wool in the middle to lower portion of the liner. This places the glass wool below the tip of the inserted syringe needle. Obviously, this is required for any type of sample filtering to occur. Many split liners have dimples or restrictions that hold the glass wool in the proper location. In most cases, the glass wool has to be moved by a large amount before any significant change in peak shape or size is observed.

A 5–10-mm-high plug of glass wool is satisfactory for most analyses. If the size of the glass wool plug is changed by a large amount, injector discrimination is changed, often noticeably. A tighter or more dense plug of glass wool also affects the amount of injector discrimination. Different amounts or densities of glass wool cause a different amount of sample mixing, which changes the amount of injector discrimination. For the most reproducible results, prepacked liners should be obtained from the same supplier. If the liners are packed in the laboratory, the same person should pack the liners. It is surprising how packing technique differences between individuals affects the amount of injector discrimination. If injector discrimination changes are not critical (they usually are not), small differences in packing technique are inconsequential.

The primary problems encountered when using glass wool are shifting or obstruction of the glass wool plug, dirtiness, or activity. If an excessive or tight (dense) plug of glass wool is used, the carrier gas may push the plug to the bottom of the liner. This frequently blocks the opening to the column or split line. Poor peak shapes, shifting retention times, changes in column head pressure, or erratic gas flow may result. Long term exposure to high injector temperatures may cause the glass wool to become brittle and fracture into very small pieces. The plug then becomes dense and blocks the flow of carrier gas. These small glass wool pieces may also plug the column or split line. If this occurs, high head pressure and little column flow usually occurs. When injecting dirty samples, the glass wool eventually becomes contaminated with the accumulated nonvolatile residues. These collected residues can interact with subsequently injected samples. Degradation of peak shape or size are the usual symptoms. Glass wool eventually becomes active with use and can interact with active compounds. This leads to peak tailing or loss of size for these compounds.

When packing your own liners, use only silylated glass wool or fused-silica wool. Using regular glass wool results in the loss of active compounds. Only the outer surface of glass wool is deactivated, thus minimize the number of exposed ends that are created during the packing process. Gently fold and compress the glass wool fibers so that the fibers remain as intact as possible. This also minimizes the number of small fibers that can be created as the glass wool degrades with age. When removing a glass wool plug from a liner, be careful not to scratch the surface of the liner. This exposes untreated glass, and thus active sites. It is probably better to replace the entire liner if the glass wool becomes fouled with sample residues.

The purpose of *Chromatography Problem Solving and Troubleshooting* is to have selected experts answer chromatographic questions in any of the various separation fields (GC, GC–MS, HPLC, TLC, SFC, HPTLC, open column, etc.). If you have questions or problems that you would like answered, please forward these to the *Journal* editorial office with all pertinent details: instrument operating conditions, temperatures, pressures, columns, support materials, liquid phases, carrier gas, mobile phases, detectors, example chromatograms, etc. In addition, if you would like to share your expertise or experience in the form of a particular question accompanied by the answer, please forward to JCS Associate Editor, *Chromatography Problem Solving and Troubleshooting,* P.O. Box 48312, Niles, IL 60714. All questions/answers are reviewed to ensure completeness. The *Journal* reserves the right not to publish submitted questions/answers.

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Associate Editor