Gas Chromatography Problem Solving and Troubleshooting

Question:

There are several commercially available solutions that claim to improve column performance, extend column life, or repair damaged columns. Are the enhancements worth the cost and effort of using these types of solutions?

Answer:

Conditioning or rejuvenation solutions are rarely effective with capillary columns and only occasionally effective for packed columns. These reagents are intended to react with active sites in a column. Active sites often cause compound adsorption that results in peak tailing or a reduction in peak size for active compounds. Active compounds are defined as those with a hydroxyl (–OH), amine (–NH), or thiol (–SH) functional group. Certain stationary phases may develop active sites after prolonged use. While these sites contribute very little to column activity, it is believed that they may catalyze column bleed.

For capillary columns, active sites are primarily found on the tubing surface. For fused-silica tubing, these active sites are called silanols and are composed of a hydroxyl group attached to a silicon atom (Si–OH). Present-day capillary columns are well deactivated, thus new columns contain a minimal number of silanols. Typical procedures involve passing several millimeters of solvent-diluted reagent through the column. For the reaction to occur, the solvent needs to penetrate the stationary phase and come in contact with the tubing surface. With the relatively low number of silanols and the minimal contact of the reagent with the tubing surface, treating a new capillary column does not decrease activity or increase column life.

Packed columns have a stationary phase coated onto the surface of small particles called supports. These supports are often active and can be a major source of peak tailing or size loss. Deactivated supports are available; however, the variety of these supports is limited. Depending on the type of support, injecting a conditioning solution may reduce the amount of column activity. Numerous injections or treatments with the reagent may be necessary because of the very large surface areas of most supports. Over time, the deactivation may be lost and the column may require additional injections or treatments.

Polysiloxane stationary phases (commonly and improperly called silicones) are commonly used in packed and (especially) capillary columns. It is commonly believed that as polysiloxane stationary phases degrade with normal use, a silanol group is created at the end of the polymer chain. This silanol catalyzes further degradation of the stationary phase. Unfortunately, another silanol group is regenerated as part of the degradation process, thus the process is self-sustaining. The creation and elution of stationary phase degradation products is responsible for the bulk of column bleed. If the continued creation of silanol groups could be eliminated or reduced, the amount and progression of column bleed could theoretically be reduced. Because the conditioning reagents can react with silanol groups, treating the column should reduce column bleed. In practice, little to no reduction in column bleed is obtained, especially for capillary columns. Treating older columns with elevated column bleed does not reduce column bleed either.

Any stationary phase that contains hydroxyl, amine, or thiol groups should not be treated with any type of deactivation or conditioning solution. Because the reagents react with these types of groups, the reagent will alter the chromatographic properties of the stationary phase. Irreversible alteration or damage to the stationary phase often occurs, rendering the column unusable.

There are no benefits from treating capillary columns, whether old or new, with conditioning or rejuvenating reagents. Sometimes older, non-bonded capillary columns exhibit some improvement after treatment with conditioning reagents. Since most current capillary columns are bonded, using conditioning or rejuvenating agents is often a waste of time, effort, and expense. Some packed columns may benefit from treatment with these types of reagents. Careful selection of a more inert support often reduces the need for additional deactivation and is often an easier and more reliable route to achieving a more inert packed column.

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.

Dean Rood
Associate Editor