Gas Chromatography Problem Solving and Troubleshooting

Question

Upon changing to a low bleed version of my column, some of the separations got worse. What happened? Is there anything I can do to restore the separations?

Answer

The explanation for the separation change is probably related to the different methods that column manufacturers use to obtain low bleed columns. The three most common approaches are selecting the lowest bleed columns and designating those as low bleed columns, altering the manufacturing process but not the stationary phase, and altering the stationary phase. Depending on the column manufacturer's approach and the sample compounds, noticeable separation differences may be obtained with a regular and corresponding low bleed column. Noticeable separation differences between equivalent low bleed columns from different manufacturers may also occur because different polymer structures or manufacturing processes may have been used.

Because a significant separation change was observed, it is highly probable that the stationary phase in the low bleed column is different from the one in the regular column. For silicon-based stationary phases (i.e., polysiloxanes), lower bleed phases are often obtained by incorporating phenyl groups into the polymer backbone (Figure 1). These polymers are commonly called arylenes. Because the structure of the polymer is altered, obtaining identical stationary phase selectivity is not possible, regardless of the adjustments made in other parts of the polymer structure. For most analyses, any separation differences are inconsequential; however, there are cases where the separation differences result in significant peak coelution problems. Problems are most common for samples containing large numbers of target analytes, especially those composed of numerous isomers or congeners (e.g., dioxins and PCBs). Sometimes slight adjustments to a temperature program or using a higher efficiency version of the column (e.g., smaller diameter) rectifies the problem, but not always.

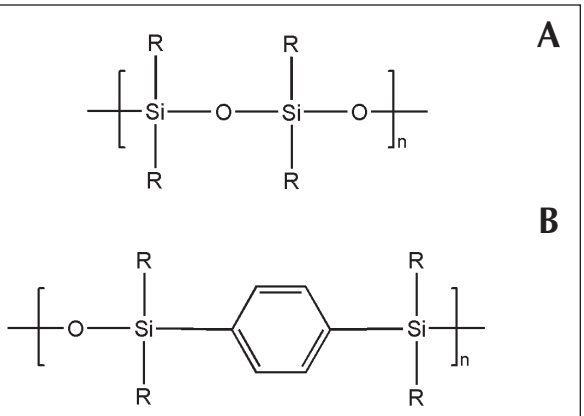

Figure 1. Polysiloxane (A) and arylene (B).

Most polysiloxane stationary phases have some phenyl content, thus adjusting for the effect of the additional phenyl in the arylene polymer backbone is feasible. For 100% dimethylpolysiloxanes, which contain no phenyl, using an arylene to obtain a low bleed column is not a good option because the phase selectivity is significantly altered by the phenyl. Altering the column manufacturing process or improving the purity of the polymer are the usual approaches to obtaining low bleed 100% dimethylpolysiloxane columns. The separation behavior of a low bleed column made in this manner is expected to be identical to the corresponding regular column. Selecting only the lowest bleed regular columns and calling them low bleed 100% dimethylpolysiloxane columns is another approach. Whether they are selected from the population of regular columns or held to lower bleed specifications during the testing process, there is no change to the stationary phase or process, thus identical separation behavior is expected for the two types of columns.

Although many low bleed columns are marketed or implied to be equivalent to their regular counterpart, separation differences may be readily evident for the same analyses. Separation difference should be expected if a modified polymer such as an arylene is used to obtain the low bleed behavior. Contacting the column manufacturer may be necessary because it may not be apparent if a structurally different stationary phase is used in the low bleed 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,* 6600 W. Touhy Ave., Niles, IL 60714-4516. All questions/answers are reviewed to ensure completeness. The *Journal* reserves the right not to publish submitted questions/answers.

Dean Rood
Associate Editor