Gas Chromatography Problem Solving and Troubleshooting

Question

In your article, you have mentioned a way to rejuvenate columns that have experienced peak-shape deterioration. Our laboratory always backflushes a column after several hundred injections. It seems to improve the performance, and we do this to enhance the column life. What are your thoughts on this routine operation?

Answer

If this procedure is working in your laboratory, why change? Column backflushing is one of the easiest procedures to follow and is the most useful when the cause of peak-shape deterioration is debris from the HPLC system accumulating on the inlet frit of the column. As you may know, debris is generated in the HPLC system by seal wear in the pumping systems and the injectors. Additionally, debris can possibly be introduced into the system from the sample and mobile phase. In the context of this discussion, the column is a great depth filter. The accumulation of debris will occur on the column inlet and cause the sample distribution on the head of the column as well as the flow through the system to be uneven. The sum total of clogging that the column inlet will have is the same result that would also be seen from a channeled bed structure. Thus, backflushing would remove that debris and the column will perform satisfactorily again.

In my experience, most of the problems with column performance deterioration are a result of either (*a*) degradation of the packing because of adverse or harsh mobile phase conditions such as exposing a bonded phase to low pH (it should be noted that some bonded phases will not experience this deterioration) or (*b*) system debris clogging the inlet frit. Generally, when clogging occurs, an increase in backpressure should be observed before or during the observation that the peak performance is deteriorating. However, this effect may not be very large at the onset. I would suggest that you monitor the backpressure and the pressure over the several hundred analyses that you run before you backflush the column. Also, you should observe the pressure after the backflush, it should be lower than when the process began. Backflushing routinely may remove the debris before it has time to work its way into the porosity of the frit, thus making it easier to remove than waiting until the frit is "really" clogged.

If the backflushing does not work, it may be that the debris is embedded into the pores of the frit. In this case, the next step would be to change the inlet frit. Changing a frit, however, means having to remove the column endfitting, and this opens the possibility of disturbing the column's bed structure. However, if backflushing does not work, what have you to lose? Changing the frit is often successful, and if you "goof", your column will have to be replaced—the same result that you would have had if the backflushing did not improve the peak shape.

If debris is causing shortened column lifetime, you should consider using an inline filter. Using this low dead-volume device before the column makes maintenance easier, because you would only have to remove the housing and change the filter rather than backflush the system. Of course, if your protocol is presently set up, use it. However, if you are asked to improve this method or develop a new method, consider using an inline filter.

Another possibility could be to use an inline guard column. This guard column could be changed periodically to maintain column peak-shape performance. However, if the problem is debris clogging the inlet filter, then the use of a guard column is a more expensive solution than an inline filter. A guard column is appropriate when chemical contamination of the bonded phase is the cause of the peak-shape deterioration.

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 it 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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