Gas Chromatography Problem Solving and Troubleshooting

Question

One of the capillary columns in my dual column assembly degraded faster than the other. This should not happen because both columns are exposed to the exact same GC conditions and samples. Why is one column failing before the other column? Is the one column defective?

Answer

Dual columns are typically composed of two significantly different columns and a length of deactivated fused-silica tubing connected by a Y-connector or splitter. The deactivated tubing is installed into a single injector, and each column is installed into individual detectors. Sometimes, a two-holed ferrule is used to install the two columns into the same injector, and the deactivated tubing and Y-connector are not needed. With either setup, the injected sample is split into two portions with each going into a single column and detector. Each column is exposed to the same sample and GC conditions, and it seems reasonable that both columns should last the same amount of time. However, there are a number of factors and circumstances that may make this an incorrect premise.

All columns do not have the same lifetime under the same GC conditions. This is true even for identical columns from the same manufacturer. The longevity differences between individual columns may be enough to account for the lifetime difference. Lifetime variations between different types of columns are expected, and column stability and robustness can vary dramatically for different stationary phases. Column degradation accelerates as the operating temperature approaches the column's upper temperature limit. Columns have different upper temperature limits, which can be a critical factor when using dual columns. The highest oven temperature that can be used is restricted by the column with the lowest temperature limit. One column may be routinely exposed to temperatures near or at its temperature limit, while the other column (with the higher temperature limit) is maintained well below its temperature limit. For this type of situation, it is expected that the column with the lower temperature limit degrades faster than the column with the higher temperature limit. Elevated column bleed levels and increased activity are the primary symptoms of normal column degradation. Other symptoms are probably caused by some other malfunction or condition.

Oxygen significantly accelerates column degradation at higher temperatures. Some columns are more prone to oxygen-induced damage than others and degrade faster with excessively high levels of oxygen in the carrier gas. A leak in the regulators, gas lines, injector, septa, or column fittings may introduce air into the carrier gas. The resulting elevated oxygen level would degrade the more susceptible column much faster than the other. A leak at the junction of the Y-splitter and a particular column would introduce air only into that specific column. The other column would not be exposed to the oxygen, thus it would not degrade as quickly. Higher oxygen damage susceptibility or a column-specific leak may explain the variable column lifetimes.

Depending on the sample and the extraction or preparation technique, column contamination is a possibility. For reasons not completely understood, some types of columns are more susceptible to contamination than others. Some may retain contaminating (i.e., very low volatility) compounds more than others and suffer performance problems. Column contamination is a common problem and often mimics the symptoms of a degrading column. Many contaminated columns are not permanently damaged and can be solvent rinsed to remove the contaminants. Unfortunately, disassembling the dual column is recommended before solvent rinsing the columns.

Small debris may become lodged inside one of the columns or in one arm of the Y-splitter. Usually, peak-shape problems are experienced with partial column blockage. If one of the dual detectors is malfunctioning, it may appear that one of the columns is at fault. One quick test is to switch the columns in the detectors. If the problem follows the column, the problem is related to the column. If the problem stays with the detector, there is something wrong with that particular detector. Some compounds or samples exhibit poor peak shapes or response with some types of columns. If the problem occurs after a period of successful results, this cause can be eliminated. If the problem has always been present with a new column, there is the possibility of some type of sample-column mismatch.

Uneven column degradation rates are normal, thus there may not be a problem or defect with the column or GC. Before a variable column degradation rate is dismissed as normal, the possibility of other problems needs to be investigated. Leaks, contamination, excessively high temperatures, incorrect analysis conditions, sample issues, or a GC malfunction may be the cause of the variable column lifetimes.

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 them 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.

Dean Rood
Associate Editor