Gas Chromatography Problem Solving and Troubleshooting

Question

After using my capillary column for a few weeks, some of the peaks begin to broaden or tail. Upon removing the column from the gas chromatograph, about 4–5 cm of the detector end is noticeably darker than the rest. If this dark section is removed, the peak shape problem is eliminated, but it returns a few weeks later. What is causing the discoloration, and how can it be prevented? How many times can I cut off the dark section of the column before it becomes too short?

Answer

Gathering a little more information will shed some insight into this problem. The most notable item was the use of an electron capture detector (ECD) at 375°C. The polyimide coating used on most capillary tubing starts to discolor with prolonged exposure to temperatures around 350°C or higher. The columns slowly turn a darker brown color; however, this does not damage the tubing unless 360°C is exceeded for prolonged periods. Excessively heated tubing may become brittle and more susceptible to breakage. High-temperature polyimide-coated tubing capable of withstanding 400°C is available, but it is more expensive and less robust than standard polyimide-coated tubing. The high temperature of the ECD resulted in the tubing discoloration. Most other gas chromatographic detectors are used at 300°C or lower, thus tubing discoloration rarely occurs. The 375°C ECD temperature exceeded the upper temperature limit of the column. The high temperature damaged the stationary phase and degraded the surface deactivation, which results in localized column activity. Usually a short section of column with high activity near the exit end does not result in any chromatographic problems unless the damage is quite severe. Some compounds, especially those with highly active functionalities (e.g., carboxylic acids, 1° and 2° amines, diols, etc.), are particularly sensitive to column activity and may exhibit peak tailing or broadening. Relatively inactive compounds such as hydrocarbons, ethers, and some ketones may not be affected by the active section of column. If the sample contains a mixture of active and inactive compounds, the active compounds would exhibit the earlier onset of peak shape problems. Besides lowering the ECD temperature, there is little that can be done to prevent the column discoloration and activity problem. Changing the ECD temperature often causes a change in response (i.e., sensitivity) and linear range, which may not be acceptable.

Cutting or trimming of capillary columns is a common practice and is often used as a column maintenance technique. Trimming is used to remove a small section of damaged or contaminated column. It is more common for the injector end of the column because nonvolatile compounds accumulate more frequently in this location. Also, column damage caused by destructive sample components is most severe at the front of the column. Trimming the exit or back of the column is done when tubing is extensively exposed to high-detector or transfer-line temperatures. Cutting 5–10 cm from a 15-m or longer capillary column has an insignificant impact on compound retention times. However, repeated trimmings eventually result in decreases in the retention times. As long as the shifting retention times do not cause major difficulties (e.g., extensive adjustment of calibration tables or curves), the accuracy and precision of the qualitative and quantitative data is not compromised. As the column becomes shorter, there is a loss of peak separation, but more importantly, a loss of peak resolution. There is a square root relationship between column length and resolution. This means a large length of column can be removed without large resolution losses. For example, if 1 m is cut from a 30-m column, the resolution loss is around 2%. Even if 15 m is cut from a 30-m column, the resolution loss is around 30%. The amount of acceptable retention time decrease and resolution loss are the determining factors when deciding the maximum amount of column that can be trimmed before it is too short.

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