Gas Chromatography Problem Solving and Troubleshooting

Question:

It is routine practice in our lab to cut about 20 cm from the front and back of the capillary column whenever one of the peaks starts to tail. This technique often repairs the problem. There is concern that after a few cuts, the column will become too short and its performance will be severely reduced. How many times can a column be trimmed?

Answer:

In theory, reducing the length of the column decreases the total number of theoretical plates and peak resolution. In practice, the loss with each cut is so small that it is rarely noticeable in the chromatogram. After numerous cuts, the column length is significantly shortened and a decrease in peak resolution eventually becomes evident. At some point, the column becomes too short to provide the required peak resolution. The length of column that can be removed before there is a detrimental effect on peak resolution depends on a number of variables, with original column length and amount of peak resolution being the most important.

Based on chromatographic theory, resolution and column length is related by a square root function. For example, doubling the column length does not increase resolution by 100% but by the theoretical amount of 41%. In practice, the resolution gain is usually 25–35%. Halving the column length does not decrease resolution by 50% but by 21%. In practice, the resolution loss is usually 12–15%. This square root relationship means that a large length of column has to be removed before a significant loss in resolution occurs.

For a 30-m column, a loss of 1 m results in only a 1.7% decrease in resolution. The column would have to be shortened to 25 m before a 10% loss in resolution occurs. Shorter columns tolerate less trimming before resolution losses become too severe. A loss of 1 m from a 15-m column results in a 3.4% loss in resolution. A 15-m column would have to be shortened to 12.2 meters to obtain a 10% resolution loss. This is a 2.8-m loss as compared to a 5-m loss for a 30-m column. Columns can continue to be shortened until the amount of resolution becomes too small. Greater column length reduction can be tolerated when the original peak resolution is larger and the original column is longer. Retention times decrease as the column length is shortened, thus small losses in retention times have to be acceptable with each trimming.

When injecting dirty samples, trimming the front of a capillary column often (but not always) improves the performance of a column. Sample-induced damage or performance degradation usually occurs in the front portion of the column. This section of column suffers the greatest amount of contamination from non-volatile sample residues or damage from chemical attack. This damaged or contaminated section is removed by cutting away the tubing, thereby restoring column performance. Usually, any damage or contamination is in the first meter of the column, so it is rarely necessary to cut off more than 1 m from the front of the column. In most cases, trimming 10–50 cm from the front of the column is sufficient to remove the most contaminated or damaged section. This means that 5–25 trimmings are possible before a notable loss in resolution occurs.

Trimming the back of the column is often unnecessary. It only serves to shorten the column because sample-induced damage or contamination is usually localized within the first meter of the column. There is one situation that may warrant trimming the back of the column. If the back portion of the column resides in a detector or transfer line maintained at a temperature much higher than the upper temperature limit of the column, it might suffer some minor damage. Fortunately, any damage in this part of the column rarely degrades peak shapes because the sample spends a very short amount of time in the last length of column. If trimming the back of the column does not improve peak shapes, the practice should be stopped. If fused-silica tubing is visibly darker upon removal from a high-temperature detector or transfer line, it is advisable to trim off the darkened portion before re-installing the column. Prolonged exposure to temperatures above 350°C often darkens the polyimide coating. Above 350°C, the polyimide coating may weaken, leading to column breakage.

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.

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