Gas Chromatography Problem Solving and Troubleshooting

Question:

All of the peaks in my chromatogram suddenly started to tail. I injected a column test mixture, and the same problem occurred. I trimmed about 5 cm from each end of the column; however, this did not fix the problem. The amount of column bleed has not increased. What is wrong with the column, and can it be repaired?

Answer:

There are several possible causes of this peak tailing problem. Although the column is the most likely source, there are other possibilities that also need to be investigated. Because the peak tailing occurred after the column was successfully installed and used, the column installation and injection techniques are not suspect. A poorly installed column or very slow injection speed can cause peak tailing. Usually, the earlier eluting peaks exhibit the worst tailing for these types of problems. Check for unintended changes in the injector and detector. If the injector or detector temperature is too low, peak tailing such as that seen in Figure 1A may occur. An excessively low split ratio or detector auxiliary (makeup) gas flow may also cause this type of peak tailing. Finally, a problem with the injector liner such as compaction or movement of silylated glass wool, breakage, or accumulation of solid debris (e.g., septa particles) may have occurred. If all of these types of problems have been eliminated as sources, then the column is the likely culprit.

There are only a few column problems that can cause peak tailing. A poorly made capillary column may exhibit tailing or missphapened peaks. Because the column performed satisfactorily for a time, this possibility can be eliminated. Activity (i.e., lack or loss of inertness) causes peak tailing for active compounds such as alcohols, carboxylic acids, and amines. Columns become more active with use, and the resulting peak tailing usually occurs gradually and not suddenly. The test mix chromatogram exhibited peak tailing for all of the compounds, even nonactive compounds such as the hydrocarbons (peaks 2, 4, 6, and 9 in Figure 1). Severe peak tailing for nonactive compounds indicates that column activity is not the cause. Only active compounds would exhibit tailing with an active column.

Damage to the stationary phase caused by exceeding the upper temperature limit for a prolonged time, constant exposure to oxygen at higher temperatures, and/or repeated injections of mineral acids or bases (e.g., sulfuric acid, sodium hydroxide) often result in peak tailing. Active compounds are the first to exhibit peak tailing, and the stationary phase has to be severely damaged to obtain peak tailing for nonactive compounds. The injection of a column test mixture is a good method of determining which compounds types are exhibiting peak tailing. If there is enough damage to cause nonactive compounds to tail, there will be an excessive amount of column bleed. Because the column bleed did not rise by a substantial amount, column damage is an unlikely source of the peak tailing.

The most common cause of peak tailing for nonactive compounds is column contamination. These contaminants are relatively nonvolatile, and they accumulate in the column over time. These types of contaminants usually originate in the sample and are species such as polymeric materials, salts, and proteins. Another contaminant is solid debris such as tiny slivers of septa, ferrule, glass, or other particles that fall into the column and become trapped. Usually, these types of contaminants are contained in the very front of the column. Depending on their shape and the column diameter, they may migrate several meters into the column. Trimming a few centimeters from the front of the column may remove the contaminated portion, but trimming several meters may be necessary. Because trimming a few centimeters of the column did not work, and the other possibilities have been eliminated, a more severe column contamination problem is likely the cause of the peak tailing seen in Figure 1A. One meter of the front of the column was removed, and the column was tested again (Figure 1B). The peak tailing problem disappeared; thus, some type of severe contamination or solid debris was present in the first meter of the column. If a large portion of the column was contaminated, it may have been necessary to solvent rinse the column in order to remove the contaminants (1). Although solvent rinsing removes many contaminants, some may remain and render the column unusable.

Whenever a peak tailing problem occurs, all of the possible causes need to be investigated. It is very easy to be led astray

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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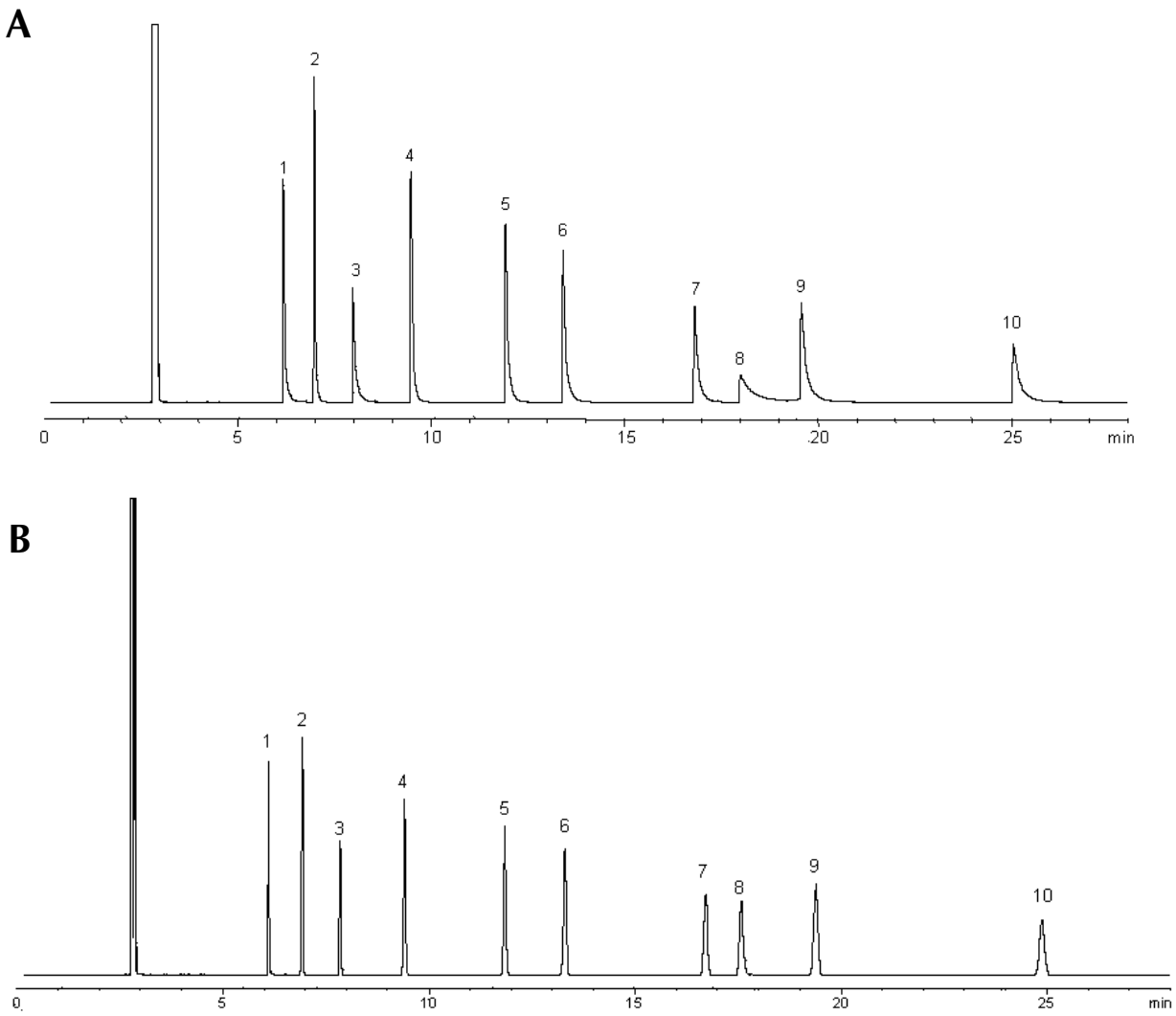

Figure 1. Test mixture chromatogram for original column (A) and original column after removing 1 m from the front (B). Chromatographic conditions for both columns: column, DB-210 (60 m x 0.25 mm, 0.50 µm); split injector, 250°C, 1:100 split ratio; FID detector, 300°C; carrier gas, hydrogen at 37.0 cm/s; column temperature, 120°C. Peaks: 1, 1-octanol; 2, tridecane; 3, 2,6-dimethylphenol; 4, tetradecane; 5, 2,6-dimethylaniline; 6, pentadecane; 7, methyl decanoate; 8, 1-undecanol; 9, hexadecane; 10, methyl undecanoate.

when considering only some of the possibilities or jumping to conclusions. By also considering GC factors and observing any patterns or trends, it is much easier to reduce the number of possibilities. Gathering facts such as when the peak tailing first occurred, which types of compounds are tailing (i.e., active or nonactive), and whether the amount of column bleed is elevated may aid in finding the source of the problem.

References

1. D. Rood. Gas Chromatography Problem Solving and Troubleshooting. *J. Chromatogr. Sci.* **33:** 596 (1995).