Gas Chromatography Problem Solving and Troubleshooting

Question:

I was informed that capillary columns do not have a specific front and back. Upon reversing the direction of one of my capillary columns, I obtained a significant difference in the retention times. This implies that capillary columns are directional. Is this a correct conclusion?

Answer:

With only a few exceptions, capillary columns are not directional, and their performance is the same regardless of the installation direction. Some types of columns are more prone to directional differences. Long-length, thick-film, or small-diameter columns are more likely to suffer from a directional difference. A directional difference is even more likely if a column has more than one of these characteristics.

If a directional difference occurs, it indicates that the front portion of the column is different than the back portion. This difference is in the stationary phase film thickness; in other words, the film thickness is different at the two column ends. Usually, the difference in film thickness occurs as a gradient along the column length. In other words, the film gradually becomes thicker or thinner along the length of the column. One way to illustrate this occurrence is to cut the column in half and compare the retention of the two halves.

Figure 1 shows a test mixture chromatogram for a 60-m column exhibiting a directional difference. Figure 2 shows the same test mixture for each of the 30-m halves. Pentadecane (peak 8) retention times of 15.99 and 11.40 min are obtained for the two halves. A retention time difference of less than 0.5 min would normally be expected for this type of column. Comparing pentadecane retention factors (k) for all three columns further illustrates the film thickness differences. If the film thickness was uniform throughout the column, the same retention factor would be obtained for the original column and the two halves. The retention factor for the original 60-m column is 8.06 (Figure 1). Retention factors of 9.12 and 6.50 were obtained for the two halves (Figures 2A and 2B). These retention factors indicate that the average film thickness of the two columns are 0.8 and 0.55 µm, respectively.

Sometimes, a longer column is cut into shorter lengths. This is done to save money, because longer columns cost less per meter than shorter columns. Although this practice usually works, the occasional directional difference in columns may result in the shorter columns having significantly different retention characteristics.

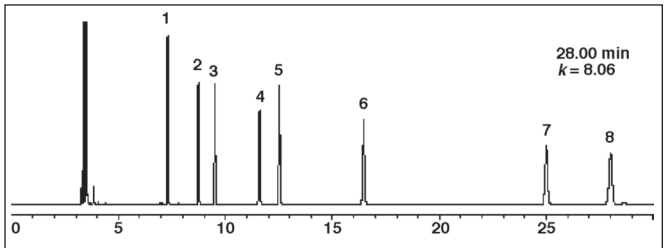

Figure 1. Test mixture chromatogram for the original 60-m column. Chromatographic conditions: column, 5% diphenyl–95% dimethyl polysiloxane (60 m × 0.32-mm i.d., 0.7-µm average film thickness); split injector, 250°C, 50:1 split ratio; FID detector, 300°C; carrier gas, helium at 32.4 cm/s; column temperature, 150°C. Peaks: 1, 1,6-hexanediol; 2, 4-chlorophenol; 3, methyl nonanoate; 4, 4-propylaniline; 5, tridecane; 6, 1-undecanol; 7, acenaphthylene; 8, pentadecane.

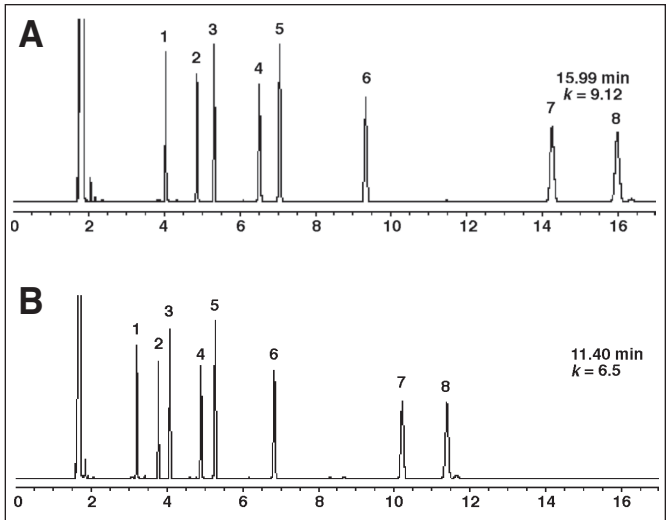

Figure 2. Test mixture chromatograms for the 30-m halves. Chromatographic conditions: column, 5% diphenyl–95% dimethyl polysiloxane (30 m × 0.32-mm i.d.); carrier gas, helium at 31.7 cm/s (A) and helium at 32.9 cm/s (B). The injector, detector, column temperature, and peaks are the same as in Figure 1.

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.

Dean Rood
Associate Editor