Gas Chromatography Problem Solving and Troubleshooting

Question:

My flame-ionization detector (FID) goes out about 1.5 min after I make a sample injection. It occurs for the samples in water, but not for the ones in methanol. How do I prevent the flame from being extinguished? Is the water damaging my FID?

Answer:

There is no damage to the FID, but the large amount of water is extinguishing the flame. Unfortunately, there is little that can be done to consistently avoid this problem. Decreasing the injection volume is one possible solution. This approach is most successful for Megabore direct, splitless, and on-column injections because these deposit large volumes of the injection solvent into the column. Another technique is to add enough methanol to the aqueous sample to create a final methanol concentration of 10–20%. As long as the column does not separate the water and methanol, the presence of flammable methanol may prevent the flame from going out. Obviously, this technique is not suitable if sample dilution or alteration is not possible. Sometimes, increasing the hydrogen flow to the FID by 5–10 mL/min may prevent the flame from going out. It may be more difficult to light the FID flame if the hydrogen flow is changed by more than 10 mL/min. Finally, installing a retention gap or guard column may help (1).

Question:

Upon installing a new capillary column, some peaks exhibited tailing that was not present for previous columns of the same description. After checking the GC and conditions for possible changes, none were noted. However, I noticed that the color of the new column was lighter than the previous ones. Is it possible that I received the wrong column or that the lighter tubing is responsible for the increase in peak tailing?

Answer:

The color of the column has no effect on the chromatographic performance. The color is determined by the polyimide coating on the outside of the fused-silica tubing. The chromatography occurs inside of the tubing, thus any chromatographic differences cannot be attributed to the outside of the column. It is just a coincidence that the problem occured with a lighter colored column. The increased peak tailing is due to an undetected system problem, accidental contamination of the new column, or a normal variation in the activity level of columns from this specific manufacturer. Any changes in retention, peak shape or width, or separation cannot be correlated to the color of the column.

Differences in the polyimide coating have two noticeable effects. One batch of tubing may be stronger than another, making it less likely to break or more difficult to cut. Some of the time required to condition a column can be attributed to the polyimide coating (2). One column may stabilize (i.e., condition) slightly faster than another due to differences in the polyimide coatings. Columns become darker with use, especially if they are exposed to high temperatures for long periods of time.

References

1. D. Rood. Gas chromatography problem solving and troubleshooting. *J. Chromatogr. Sci.* **34:** 64 (1996).
2. D. Rood. Gas chromatography problem solving and troubleshooting. *J. Chromatogr. Sci.* **35:** 343 (1997).

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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