Gas Chromatography Problem Solving and Troubleshooting

Question:

I have been experiencing some retention time instability with my portable GC compared with the benchtop GC in the lab. Because of the small oven size of the portable GC, the column cage has to be smaller than 3.5 inches in diameter. The smallest diameter cage available from various capillary column manufacturers was 5 inches. A fused-silica column was removed from its cage and wound around a cylinder that was approximately 3.5 inches in diameter. High-temperature Kapton tape was then wrapped around the tubing in 4 places to hold the coil in place. The column was placed on the bottom of the GC oven and installed into the injector and detector as usual. Is the retention time instability related to the column configuration, or are retention times less precise with portable GCs?

Answer:

Probably both the column configuration and the portable GC are contributing to the retention time instability problem. Many of the portable or mini-GCs that use heated air to heat the column do not have the oven temperature accuracy or precision of the larger benchtop GCs. Most GCs do not heat the column directly, but use heated air that circulates through the GC oven in which the column resides. Uniform, accurate, and precise temperature control and air flow in the oven are critical for reproducible retention times. The larger benchtop GCs have substantially more space for the heaters, fans, and vents necessary to control flow and temperature of the air circulating through the GC oven. Portable GCs have severe space limitations, thus compromises are sometimes made. Some inexpensive models have exposed heating elements that are shielded only by a crude wire mesh. The sections of the column closest to the heating element may be exposed to a higher temperature than other sections. The fan is often placed directly in the oven, thus the heated air circulates in a fairly uncontrolled flow pattern. Because the air does not flow in an even manner around the column, uniform heating of the column does not occur. Combined with the exposed heating element, temperature control in the oven is less accurate and precise for these types of portable or mini-GCs. This leads to less-stable retention times when compared to benchtop GCs. In some cases, peak broadening may occur because of the uneven heating of the column. Even with better-designed ovens, the small size of portable GCs makes it difficult to match the accuracy and precision of a well-designed full-sized GC. Some portable GCs embed the column in a solid material and the entire assembly is heated. Because circulating heated air is not used, the aforementioned problems are eliminated.

Rapid heat transfer from the air to the column is required for proper heating of the column. If the heated air cannot freely flow around all of the column tubing, the heat transfer may not occur in the desired manner. The column is not uniformly heated to the same temperature as the oven. If the column tubing is bunched or coiled together so that some of the tubing is not directly exposed to the air (i.e., in the center of a large bundle of tubing), it may not be at the same temperature as tubing at the outer edges of the bundle. If isothermal temperature conditions are being used, the center of the bundle will eventually heat to the set temperature. Using a temperature program (especially with fast ramp rates) may result in inconsistent column temperatures with tightly bundled or coiled columns. Minimal overlap or bundling of the tubing does not cause any problems, but large bundles may lead to retention time instabilities or peak-broadening problems. If a column needs to be wound into a bundle or coil, make sure that no more than about 3 layers of tubing overlap in one area.

Laying column tubing directly onto the floor of the GC oven does not allow the heated air to flow around the bottom coils of the column. Again, this will lead to uneven heating of the column, thus retention reproducibility problems. Also, the walls of the oven are often not the same temperature as the air within the oven. Because the GC monitors the oven temperature using the air temperature, sections of the column are not at the desired temperature while other sections are at the correct temperature. Retention time reproducibility and peak-broadening problems may occur. If a cage-less column is being used, care must be taken to prevent the column from touching the walls of the oven. A homemade column hanger or support needs to be invented. Several short pieces of wire (or paper clips) can often be fashioned into a suitable column support or hanger. Remember to file the wire ends in order to eliminate any sharp edges that could scratch the tubing and lead to 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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