Gas Chromatography Problem Solving and Troubleshooting

Question:

When I've finished with the GC for the week, I turn off all of the GC gases to avoid wasting gas over the weekend. Upon returning on Monday morning, it takes 3–4 h for the GC signal to stabilize. I was told that I might be damaging the column or GC. Is this true? What are the best conditions for storing a GC when it is not being used?

Answer:

Heating a column without carrier gas flow results in rapid damage to the column. Even if the column oven is off, the short lengths of column inside of the injector and detector are often at temperatures of 200°C or above. The occurrence and severity of any column damage depends on a wide range and number of intertwined variables (temperatures, times, injector and detector design, column). The safe and conservative recommendation is to always maintain carrier gas flow in the column whenever the oven, injector, or detector is heated. Although this is overly cautious for some situations, it is the most dependable method for maintaining the integrity of the column.

While there is no carrier gas flow, various contaminants may accumulate in the injector. Septum bleed compounds, residues from previously injected samples, and other types of undesirable contaminants build up in the injector because there is no carrier gas flow to sweep away these materials. When the carrier gas flow is re-established, some of this accumulated material is transported into the column and eventually elutes from the column. Noisy and unstable baselines or ghost peaks are the most common symptoms associated with this type of problem. Several hours or more of waiting may be needed before the symptoms subside.

Another source of the temporary GC signal instability may be the detector. Some detectors require several hours to one day to stabilize after only 1–2 h without gas flow. Temperature- or flow-sensitive detectors such as ECDs and TCDs are particularly noted for this type of behavior. Many GC–MS systems should not be left under vacuum without column carrier gas flow. The hydrogen and air flame detectors (e.g., FID, FPD) can be repeatedly turned off and on without any major problems. With the exception of flame gases, turning off or changing the detector gas flows may results in several hours of waiting until the baseline or sensitivity returns to normal.

When a GC is not being used, it is recommended to leave the column temperature at 35–40°C. Leaving the column at a higher temperature often reduces column life. Also, a leak or a loss of carrier gas does not result in damage to the column at the lower temperature. Immediately before the next use, the column needs to be heated for 15–30 min at a higher temperature (usually the final temperature in the temperature program). If a GC is not going to be used for 2–3 days, it is probably best to leave all gas flows and temperatures at their usual operating values. For a split/splitless capillary injector, the split flow can also be reduced to save on unnecessary use of expensive carrier gas. Some of the automatic or electronic control flow systems have a gas saving feature. It reduces the split flow during periods of non-use. Care must be taken to ensure that all flow rates are returned to their recommended or previous values. If a GC is not going to be used for one week or longer, it often makes sense to reduce or turn off the gas flows or, perhaps, the temperatures. It is usually best to maintain ECDs and TCDs above 100°C and with at least a minimal flow of gas.

Usually, economics and convenience dictate whether turning off the gas flow is reasonable. As long as a column or detector is not heated, turning off the gases does not cause any long-term damage. Several hours or days may be required before the GC is completely stabilized.

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