Gas Chromatography Problem Solving and Troubleshooting

Question:

To speed up my analysis, I switched from helium to hydrogen as the carrier gas in my GC–MS. Although I did gain analysis speed, I lost sensitivity. What happened?

Answer:

The recommended average linear velocity for hydrogen is about two times that of helium. This also means that the corresponding volumetric flow rate of hydrogen is about two times greater than that of helium. This flow rate difference is rarely an issue with standard GC detectors; however, carrier gas flow rates can be an important consideration in GC–MS systems. Mass spectrometers are maintained under a vacuum using vacuum pumps. Many benchtop GC–MS systems have pumping capacities of 1–2 mL/min. Using higher carrier gas flow rates does not harm the vacuum pump or the MS; however, a loss of sensitivity might occur. For 0.25-mm-i.d. columns, typical carrier gas flow rates are 2–4 mL/min for hydrogen and 1–2 mL/min for helium. Hydrogen often exceeds the recommended flow rate for many benchtop GC–MS systems, thus a loss of sensitivity may occur relative to helium. In addition, excessively high carrier gas flow rates might interfere with the proper tuning of the MS.

Lowering the flow rate of hydrogen to the MS minimizes the sensitivity loss. Lowering the average linear velocity would reduce the flow rate but would also eliminate the main reason for using hydrogen as the carrier gas (shorter analysis times) and perhaps cause an efficiency loss. A better alternative is to change to a 0.18- or 0.20-mm-i.d. capillary column. At the same average linear velocity as for a 0.25-mm-i.d. column, the carrier gas flow rate is lower for these smaller diameter columns. Because the carrier gas flow rate is reduced (while still maintaining column efficiency and short analysis times), this often allows the use of hydrogen as a carrier gas without a measurable loss of GC–MS sensitivity. The high efficiency of smaller diameter columns often allows the use of shorter columns and further reduces analysis times.

There have been a few anecdotal reports of hydrogen explosions in a GC–MS. If a power loss occurs, hydrogen continues to flow into the MS. It is suspected that there are potential areas where small pockets of hydrogen may accumulate if the MS is off for a prolonged period. When the power is resupplied, the accumulated hydrogen may ignite when the MS begins to heat. In practice, the probability of this type of hydrogen ignition is extremely low. GC–MS systems equipped with electronic pressure or flow control injectors turn off the gas flows when a power outage occurs, thus eliminating the possibility of this type of problem.

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