Gas Chromatography Problem Solving and Troubleshooting

Question:

When using a PLOT capillary column with an FID, I usually get one or more sharp spikes in the chromatograms. The spikes appear at random and only occur in the GC with PLOT columns. Is the PLOT column damaged?

Answer:

Spikes are usually caused by particulate matter entering a detector. Spiking is most common with dirty FID and NPD detectors. Particles fall off of the dirty collector and cause a spike upon impact with the jet. One technique to test for this possibility is to gently tap the plastic handle of a screwdriver or similar item against the collector assembly. (Do not use a metallic item, such as a wrench, because it will cause a spike to appear.) If spikes are obtained upon tapping, it is a good indication that the collector has become coated with solid debris and needs to be cleaned. In rare cases, an electrical problem can cause spiking. The spike usually occurs at the same time as some type of external event, such as a valve switch, the start up of other instruments plugged into the same circuit, or a similar event.

Another source of particles are PLOT columns. Most PLOT column stationary phases are composed of a thin layer of small particles adhering to the inner wall of the capillary tubing. These particles can detach and be transported out of the column by the flowing carrier gas. Particle loss from alumina and molecular sieve PLOT columns is fairly unusual. Many of the pora-plot or pora-pak type of PLOT columns suffer from particle loss. Loose particles are generated upon bending or cutting the column, such as during column installation. Sometimes the frequency of loose particle formation, and subsequent spiking, decreases after the column has been installed for a few days. Some columns continuously lose particles and cause detector spikes without any apparent pattern.

If spiking caused by PLOT columns is a problem, there is one technique that may reduce the number of spikes. The dislodged particles can often be trapped using a short length of WCOT capillary column attached to the back of the PLOT column. This particle trap is a short piece of capillary column coated with 3–5 µm of a 100% dimethylpolysiloxane (e.g., DB-1, HP-1, Rtx-1, SPB-1, etc.) or 5% diphenyl–95%dimethylpolysiloxane (e.g., DB-5, HP-5, Rtx-5, SPB-5, etc.) stationary phase. The diameter should be the same or larger than the PLOT column (one with the same diameter is recommended). The particle trap length should be just long enough to protrude from the detector fitting by several centimeters; thus the actual length is governed by the column insertion distance as specified for the model of GC or GC–MS being used. The piece of column acting as the particle trap is attached to the PLOT column using a suitable union. The type of union is not critical, as long as it has a very small internal volume and it does not leak.

The particle trap will need to be replaced as it becomes plugged with particles. As the piece of column becomes plugged, an increase in retention times may be observed along with an increase in the number of spikes. It is tempting to use a longer piece of tubing to reduce the replacement frequency, but the separations obtained with the PLOT column may be affected by longer lengths of the particle trap. Usually, the shortest reasonable length of particle trap is used to minimize its affect on the separations.

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