Gas Chromatography Problem Solving and Troubleshooting

Question

Sometimes a series of spikes randomly appears in my chromatograms. Re-installing the capillary column or cleaning the FID does not fix the problem. The spikes only go away after installing a new FID jet. What is causing the spikes?

Answer

FID spiking can originate from a number of sources. Because replacing the FID jet eliminated the spiking, the problem is probably directly related to the FID jet. Assuming the FID jet is not physically damaged, the only failure related to FID jets is contamination. Deposits can accumulate on the jet, especially when analyzing high molecular-weight compounds or using very thick film capillary columns or high loading percentage packed columns. Usually, dirty FID jets result in elevated baseline noise and loss of sensitivity. Because cleaning the FID did not eliminate the problem, it seems logical that a contaminated jet is not the source of the problem. Only after a thorough and detailed investigation was this conclusion proven to be wrong and the source of the FID jet contamination found. Surprisingly, the technique used to install the capillary column indirectly resulted in the jet contamination.

One technique that is used to install a capillary column in an FID is to push the column into the FID until it stops (i.e., it hits the tip of the jet). The column is then pulled down approximately 1 mm and the column ferrule is tightened to seal and hold the column in place. When carefully performed, this technique often results in satisfactory installation of the capillary column in the FID; however, when improperly performed, FID jet contamination may occur. When tightening the ferrule, the column is pulled slightly upward. If the column was not pulled down far enough before tightening the ferrule, the end of the column would be strongly forced into the tip of the FID jet. This often results in breakage or shattering of the column end, which generates a number of very small pieces of fused-silica and polyimide. Afterwards, when the FID flame is lit, the jet becomes extremely hot. At this high temperature, the pieces of polyimide melt and fuse to the inner portion of the jet. Eventually, microscopic pieces of the polyimide contaminants flake off of the jet, resulting in the spikes. Pushing a fine wire through the jet, soaking in solvent, or ultrasonicating the jet usually does not remove all of the fused-silica or polyimide contaminants. The jet often appears to be clean and no foreign particles are observed inside of the jet. If an FID jet has become contaminated with pieces of a fused-silica capillary column, replacing the FID jet is the easiest and probably the best method to eliminate the spikes.

To avoid this type of FID contamination, the proper capillary column installation technique needs to be practiced. The insertion distance of the capillary column into the FID is provided in the GC manual. Usually, a distance from the tip of the column to a reference point (such as the top of the ferrule or bottom of the column nut) is stated. If this distance is used and never exceeded, contamination of the FID jet by small pieces of broken column should not occur. The only exception would be cases in which a graphite ferrule is severely overtightened. Upon overtightening, a graphite ferrule becomes extremely compressed and the column can move upward by 2–3 mm, which results in the column end being broken inside of the FID jet. With the proper installation technique and properly tightened ferrules, contamination of the FID jet by broken pieces of column should not occur.

Other sources of spiking include particle loss from PLOT columns, a dirty or rusty FID collector, and an electrical problem. Gently tapping the FID collector with the plastic handle of a screwdriver (do not use the metal end or any metallic object such as a wrench) usually dislodges some of the collector contaminants. If spiking is observed that roughly corresponds with the tapping, then collector contamination or rusting is a very likely source. Cleaning the collector usually fixes this problem. Examining the various cables and connectors between the FID and the electrometer board may uncover loose connections or damaged cables. A faulty electrometer board may also cause spiking, but this is fairly rare. It is usually best to consult with a qualified service engineer before attempting any type of board level repair or indepth investigation.

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