Gas Chromatography Problem Solving and Troubleshooting

Question

Much to my horror, an associate in our lab injected raw liquid coffee onto our GC–MS system to determine whether or not the cafeteria had switched the regular and decaf pots. He claimed a little water wouldn't hurt the system, but I thought water on fused silica capillary columns was taboo because it attacks the stationary phase. Isn't that why we have moisture traps to remove water from the carrier gases we use? The resulting chromatogram had poor chromatography for several early-eluting peaks, but the caffeine peak was well-defined. When I later ran a test mixture, the column performance was fine, the mass spectrometer tuning and sensitivity were unchanged, and my associate seemed vindicated. What is the deal with water-based samples on a capillary GC column?

Answer

Aqueous-based samples can be analyzed on fused silica capillary columns that use bonded phases; however, these columns can be damaged over time by exposure to water at high temperatures. Some analysts recommend lower temperatures for aqueous-based samples because that minimizes column damage, resulting in loss of performance. But water itself is tricky because it has a high vapor volume and can create problems with pressure bursts in the injector. This can lead to oddly-shaped peaks like the early eluters you mentioned and irreproducible results. This problem can be addressed by lowering the injection volume.

I think the the real issue here is injection of a dirty sample. Aqueous samples can bring salts and metals (and who knows what other impurities in various coffees), which accumulate on the column and degrade performance over time. This requires removal of some of the column (or guard column, if installed) to restore performance. You can get away with a few injections of dirty samples without serious degradation, as your own result shows. If the coffee were strong and dressed up with lots of cream and sugar, it would be of particular concern because these additives have nonvolatile components that can decompose and leave residues on the column over time.

In the future, I suggest that your associate not analyze trivial samples on operational analytical systems. By the way, I tried the same experiment with our cafeteria coffee (no cream, no sugar) on a GC–MS system using a split injection on a DB5 column and got a very nice caffeine peak, identifiable by a mass spectral library search.

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,* 6600 W. Touhy Ave., Niles, IL 60714-4516. All questions/answers are reviewed to ensure completeness. The *Journal* reserves the right not to publish submitted questions/answers.

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