# Gas Chromatography Problem Solving and Troubleshooting

***Question:***

After about 50 injections of a water sample, I notice that some of my peaks begin to tail. Did I damage my capillary column with the water?

***Answer:***

If the capillary column stationary phase was bonded and cross-linked, the water probably did not damage the column. Most capillary columns have bonded and cross-linked stationary phases. Consult the column manufacturer if there is any doubt or question about the stationary phase. Sometimes, after prolonged use at higher temperatures, very polar stationary phases (especially polyethylene glycol) can be slightly damaged by water. If any damage occurs, it is after hundreds of injections of water and many hours at high column temperatures. Thinner film columns are also more susceptible to this type of damage. Higher than normal column bleed and peak tailing for active compounds are typical symptoms of a damaged stationary phase. These symptoms are not unique to stationary phase damage; thus, they cannot be used as an absolute indicators that stationary phase damage has occurred.

It is impurities in the water that are usually indirectly responsible for the peak tailing. Unless the water is thoroughly deionized, it may contain inorganic or ionic species. The water may also contain large molecular weight or high boiling point compounds. All of these types of nonvolatile compounds do not travel through the column; they accumulate at the front of the column. Eventually, they interfere with the proper interaction between the stationary phase and the compounds in subsequently injected samples. This often causes peak tailing, especially for compounds that contain hydroxy, amine, and aldehyde functionalities. In severe cases, the residues can interfere with all the sample compounds, which results in extreme cases of peak shape distortion. When injections with an organic solvent do not result in these types of problems, the erroneous conclusion that the water was responsible for the problem is reached. Many of the inorganic or ionic species are poorly soluble in organic solvents. Even if the organic solvent and water-based samples are handled in the same manner, the amount of residue is substantially higher in the water-based sample. The difference is not in the sample solvent but in the amount of nonvolatile contaminants present in each sample solvent.

Extracting the problem-causing compounds from the water sample is one solution. There are a variety of solid-phase extraction cartridges or minicolumns that can easily remove metals, salts, charged organic compounds, or high molecular weight organic compounds from a water sample. Another method to prevent the buildup of residue in the column is to use a retention gap or guard column. A guard column is 1–10 m of deactivated fused-silica tubing attached to the front of the analytical column. A glass pressfit union with sealing resin (1) or a metal union can be used to connect the two pieces of tubing. The nonvolatile compounds accumulate in the guard column tubing instead of inside the analytical column, thus they do not interfere with the interaction between the stationary phase and the compound. Also, the residence time of the sample in the guard column is very short, thus the contaminating residues do not have the opportunity to interact with a sample. Eventually, the guard column becomes thoroughly contaminated with the accumulated nonvolatile residues, and the front section of the guard column has to be cut off or the guard column has to be replaced.

***Reference***

1. D. Rood. Problem Solving and Troubleshooting. *J. Chromatogr. Sci.* **32:** 252 (1994).

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.

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