

## ***Gas Chromatography Problem Solving and Troubleshooting***

### **Question:**

The baseline noise in my GC-MS suddenly increased, and I was told the ion source was dirty. After cleaning the source and replacing the filaments, the noise was slightly reduced but still excessively high. Then I checked for leaks, cleaned the injector, replaced the column and traps, and installed a new gas cylinder, but the noise was still high. Finally I disassembled and rinsed the transfer line, which solved the problem. The column passes through the heated transfer line and terminates at the source. Since no sample comes in contact with the transfer line, how did it become dirty?

### **Answer:**

Probably one or more small column ferrule particles became trapped in the transfer line. The ferrule particle was probably generated during column installation. When the transfer line column nut was tightened, a small piece of the ferrule was broken off. This typically occurs when the column nut is overtightened. Some transfer line designs are prone to producing ferrule particles upon overtightening of the column nut. The vacuum of the mass spectrometer pulls the ferrule particle into the body of the transfer line. Depending on the design of the transfer line, the ferrule particle then becomes lodged in the line, trapped at the end of the line, or passes completely through and enters the ion source. Column ferrules contain graphite, which is a very good absorbent. Compounds absorbed by the graphite slowly desorb when the ferrule is heated, especially under vacuum. These released compounds are swept into the mass spectrometer, thus producing a response. Because the ferrule impurities are released on a somewhat constant basis, the overall effect is a rise in the baseline and/or an increase in baseline noise. Over time, the ferrule impurities often become depleted, and the background signal may improve. Sometimes the background signal remains high until the responsible particle is removed. If the ferrule particle enters the ion source, the noise level usually remains high until source cleaning removes the particle. It is possible, but more difficult and less likely, to get a ferrule particle trapped in a standard GC detector.

Column ferrules are usually made of 100% graphite or a 40% graphite-60% vespel mixture. Graphite ferrules are much softer and easier to seal. Upon overtightening, they sometimes produce small particles or flakes of graphite. These graphite particles can get trapped in the injector and detector fittings. A future column installation may push the particles into the injector or detector. Column contamination with solid particulates usually causes peak shape problems and/or baseline instability and noise. If overtightening is avoided, graphite ferrules can be used multiple times, and particles are less likely to be generated. Most graphite ferrules seal when the column nut is turned approximately one quarter turn beyond finger-tight. Graphite-vespel ferrules are much harder than graphite ferrules. They can be slightly more difficult to seal and do not produce small particles unless severely overtightened. Most graphite-vespel ferrules seal when the column nut is turned approximately three quarters of a turn beyond finger-tight. It is not unusual for graphite-vespel ferrules to fuse or tightly adhere to the column tubing. This may render them difficult to reuse. Graphite-vespel ferrules are usually recommended for use with GC detectors with sealed cells or units (e.g., ECD or TCD) and GC-MS transfer lines. Although graphite ferrules function properly with these detectors, a trapped ferrule particle may result in the need for major maintenance or repair of the detector. Ferrules made of 100% graphite can be used for the injector regardless of the detector.

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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**Reader's tip:**

Hans-Martin Mueller of Werthenstein Chemie AG in Switzerland has shared an easy-to-incorporate technique to prevent water from extinguishing the FID flame. This topic was discussed in the August 1997 Troubleshooting article (1). If the initial temperature of the GC column is set at a low value, water condenses at the front of the column. In most columns, water does not condense in a tight band or film but as a series of droplets spread out over a long length of column. This creates a low concentration band of water eluting from the column (i.e., the amount of water per milliliter of carrier gas is low). Because the water concentration is low, the FID flame remains lit. If the initial column temperature is too high, the water does not condense at the front of the column. Instead the water passes through the column in a tight band at a high concentration. This introduces enough water into the FID at one time to extinguish the flame.

Determining the best initial column temperature may require some experimentation. Depending on the polarity of the stationary phase, the temperature where the water forms droplets instead of a film is different. For most stationary phases, the best initial temperature will probably be 40–80°C (Mueller uses 70°C for 3 min). In general, water droplets are formed at higher initial column temperatures as stationary phase polarity increases.

**References**

1. D. Rood. Gas chromatography problem solving and troubleshooting. *J. Chromatogr. Sci.* **35**: 404 (1997).