

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

### **Question**

I had several columns that needed testing. I was told to use a Grob mix instead of the column manufacturer's test mixes. The test chromatogram for one of the columns was missing a peak. Is this indicative of a column problem? What is the advantage of using the Grob test mix instead of the manufacturer's mixes?

### **Answer**

The Grob test mix was developed as a means to evaluate capillary GC columns using a uniform and consistent procedure (1,2). Although the Grob test mix and procedure differ from many column manufacturer's test mixes and procedures, the same type and utility of information is essentially obtained. The missing peak in one of the test chromatograms may be an expected result or it may be indicative of a column problem.

The Grob test mix and procedure uses the same compounds and temperature conditions for all columns. Many column manufacturers use test mixes that are specifically formulated for the stationary phase being tested and temperature conditions optimized for the specific column. The Grob mix is more convenient because the same test mix and conditions are used for all columns. One drawback is the possibility of coelutions. If two test compounds coelute, some of the information provided by these compounds is lost. This may be the reason for the missing peak in one of the test chromatograms. If two of the test compounds are coeluting, it may appear that one of the peaks is missing. Changing the temperature program may help resolve any of the coeluting compounds; however, this defeats the purpose of having universal test conditions. The injection of individual compounds may be necessary to identify any coeluting test compounds. Coelutions are not a problem with manufacturer-specific test mixes and conditions. Any missing peaks will probably be a column or test-sample problem. If different stationary phases or column manufacturers are used, a collection of test mixes is required.

The Grob test procedure uses a temperature program. Using a temperature program results in narrower peaks than isothermal conditions. Narrow peaks can conceal small amounts of peak tailing for some compounds. If the same compounds were analyzed using isothermal conditions, more peak tailing would probably be apparent. The difference in peak tailing is caused by the differences in the test conditions and not a change or inconsistency with the column. The same applies to theoretical plate measurements. Narrower peaks translate into higher plate numbers when compared with isothermal conditions (even when the peaks have the same retention for both temperature conditions). Although column comparisons can be made for the exact same conditions and test mix, valid comparisons cannot be made if the conditions and mixes are different.

The compounds used in a column manufacturer's test mixes are very similar to the ones in the Grob test mix (some of them may even be the same). The Grob mix and some manufacturer test mixes contain dicyclohexylamine, 2-ethylhexanoic acid, and 2,3-butanediol. These compounds are very active and exhibit some tailing for nearly every column. They are also very susceptible to adsorption (i.e., partial or complete loss of peak size). If the missing peak corresponds to one of these compounds and is not coeluting with another peak, there is a problem with the column or sample. Many of the compounds in test mixes are reactive and the samples may degrade over time, especially when improperly stored (not in a sealed vial and in a refrigerator). Make sure the test sample is intact before concluding that the column is malfunctioning. If the test mix is sound and an active compound peak is missing, adsorption is occurring. The most likely cause is column activity; however, column contamination can also result in the same outcome. The identity of the missing peak is important. If it is an active compound, then test-sample degradation, column adsorption, and column contamination have to be considered and investigated. If it is an inactive compound, other problems have to be considered such as coelution or sample error. More information about test-mix compounds can be found in reference 3.

There are advantages and disadvantages to each type of test mix. The universal nature of the Grob mix and procedure is

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very appealing. The column manufacturer test-mix and procedure is more specific, but different mix and test conditions are needed for different columns. Because capillary GC columns are individually tested and the test chromatogram is included with each column, a reference point is available. A test chromatogram generated with your GC can be compared with the one provided with the column (the test mix and conditions need to be the same). This is not possible with the Grob mix and procedure unless it was originally used to test the column. Arguments can be made about the superiority of each test procedure. The most important factor is that the same test sample and conditions need to be used when making comparisons.

## References

1. K. Grob, Jr., G. Grob, and K. Grob. *J. Chromatogr.* **156**: 1 (1978).
2. K. Grob, Jr. and K. Grob. *J. Chromatogr.* **207**: 291 (1981).
3. D. Rood. Gas Chromatography problem solving and troubleshooting. *J. Chromatogr. Sci.* **34**: 96–97 (1996).