The Importance of System Conditioning After Installing a New Gas Chromatography Column

Kory Kelly
Phenomenex Inc., 411 Madrid Ave., Torrance, CA, USA

Introduction

If you have ever purchased and installed a new gas chromatography column and seen high signals when the oven temperature is raised for conditioning, your first impulse might be to despair that you have just purchased a 'bad' column. The truth is that the increased signal is common and expected during the conditioning step when new components are installed.

After a new column/component installation, detectors show increased response in signal that will decrease slowly over time with the constant increased temperature. One misconception of most users is that this rise in the baseline is solely due to excess or unstable stationary phase being purged from the GC column (column bleed). The observed rise in baseline may be a combination of many causes that collectively can be called system bleed. In fact, at temperatures below 200ºC, almost all background signal is the result of system noise.

Experimental

For these experiments, two instruments were used from Agilent Technologies (Palo Alto, California, USA). The first was a 6890 gas chromatograph equipped with a flame ionization detector (FID). The second was a 6890 gas chromatograph with a 5973 MSD. Multiple capillary columns were used and will be identified in the text when appropriate. All gases were of UHP grade with helium as carrier gas.

Results & Discussion

Detector background signal will usually have a linear response with respect to temperature either because of contamination or the physics involved within the detector.

During new column installation, detectors are sometimes allowed to cool for convenience. If the column is connected to the detector during conditioning, the detector can become contaminated. When the detector is heated following column installation an increased signal will be observed that can be interpreted as column bleed as in Figure 1. An increase in detector temperature from a constant temperature of 275ºC to 350ºC caused a signal increase of 6 pA!

The effect would be greater if the detector had been constant at room temperature for an extended period of time. It is not uncommon for more sensitive detectors such as Electron Capture Detectors (ECD) to show very high signals that may persist for days after prolonged dormancy.

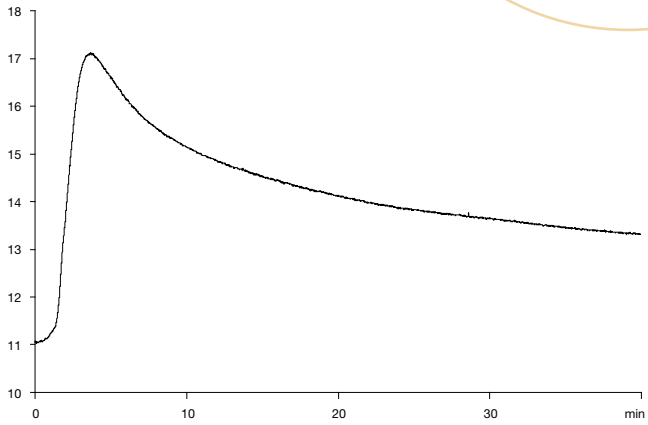

Figure 1. Chromatogram resulting when a FID detector was raised from a temperature of 275ºC to 350ºC.

Repetitive heating and cooling of detectors can cause seal and ferrule distortion allowing leaks to form. This might introduce oxygen into the system resulting in increased signal response.

If the detector temperature remains constant, other causes for baseline increases are still possible. It is likely that when the new column is installed, new ferrules were also installed. Ferrules absorb gases and other substances that offgas when heated. Figure 2 shows a spectrum that was obtained when an uncoated capillary column, which does not contain stationary phase, was installed using new ferrules. The signal seen in Figure 2 is due to system noise only and not column bleed. Notice that the intensity of the signal is very high and would be greater than most analyte peaks. This would decrease the signal to noise ratio making detection limits much worse than if the system was conditioned and the baseline minimized.

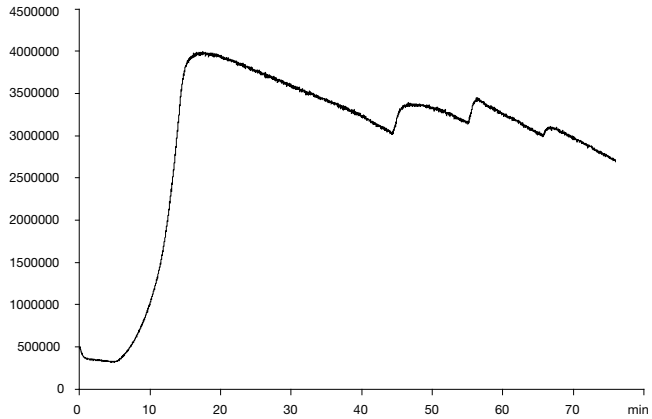

Figure 2. MSD system bleed with new ferrule and capillary column with no stationary phase. Program: 40ºC for 5 min to 320ºC at 30ºC/min for 30 min to 340ºC at 30ºC/min for 10 min to 360ºC at 30ºC/min for 10 min to 370ºC at 30ºC/min for 10 min.

The composition of the ferrule will also effect the system bleed profile. Figure 3 shows a spectrum with the same conditions as Figure 2 with the exception that the ferrules in Figure 2 were composed of 85% Vespel - 15% graphite. Figure 3 used a 60% Vespel - 40% graphite ferrules. Notice the intensity of the bleed for figure 3 is much less than the spectrum in Figure 2. After the completion of the same oven program, the signal in Figure 3 is 10% of the signal in Figure 2.

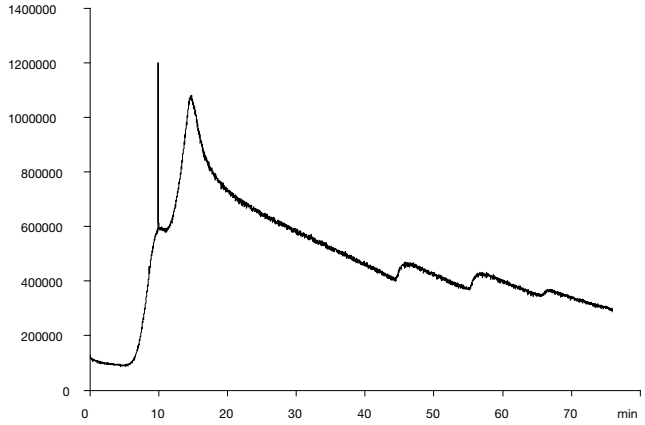

Figure 3. MSD system bleed with new 60/40 Vespel-Graphite ferrules and a capillary column with no stationary phase. Oven program is the same as in Figure 2.

Figure 4 is a mass spectrum of the bleed seen from the system during conditioning when using capillary tubing with no stationary phase. The major ions seen are 17, 18, 28, 32, and 44, and 64. Most masses can be easily explained by common gases adsorbing on the ferrule such as water, carbon monoxide, nitrogen, oxygen, and carbon dioxide. Since these same ions are indicators of a gas leak or contaminated vacuum chamber, an air and water check was run and passed before analysis. Subsequent runs after initial conditioning showed drastically reduced signals for these ions, where an air leak would likely remain constant.

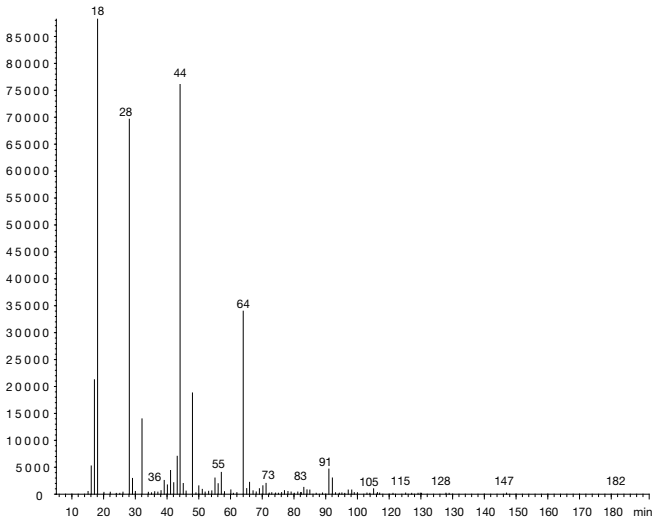

Figure 4. Mass spectrum of system bleed ions with capillary column with no stationary phase.

Figure 5 shows spectra obtained when new 60/40% Vespel-Graphite ferrules were installed with a Phenomenex Zebron™ ZB-5 column using MSD. The top trace is the total ion current resulting from total system bleed.

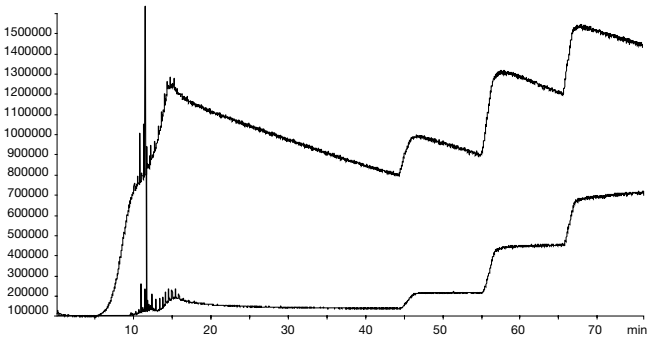

Figure 5. Chromatogram of system bleed with new ferrule. Top trace is total ion current. Bottom trace is from ions associated with column bleed. Both spectra on same scale.

The bottom trace is the result of subtracting the ions associated with system bleed from the top trace and represents bleed from column conditioning only. This figure shows how much of the bleed profile is actually due to the column relative to "system bleed." At normal operating conditions, over 90% of the ion intensity is completely due to system bleed derived from ferrule offgassing.

Conclusion

Installation of any column should be followed by a heating cycle to condition the system. During this conditioning cycle, the entire system is being conditioned, not only the column. To prevent detector contamination, it is recommended that you do not connect the column to the detector. Ferrule composition can also determine the extent of conditioning needed, as it is the key component responsible for most offgassing. Injector contributions (not covered here) should also be considered when determining system bleed and may appear as peaks.