Helium Quality Affects Thermal Desorber Calibration

Joseph Romagnano: joroma@cc.usu.edu
Bruce Bugbee: bruce.bugbee@usu.edu

For more Studies and Results: www.usu.edu/cpl

Thermal desorption technology increases the sensitivity of gas chromatography, but it also can concentrate contaminants from any gas stream that passes over a trap.

If contaminants interfere with the elution of the compound of interest, it is impossible to get a clean blank run (no sample applied yet there is still a peak) and the calibration curve will not pass through zero (Fig. 1, top line). This may be the result of contamination in either the gases used to blend the standards (trap tubes) or gases used internally by desorber (cold trap). However, when combined with an inability to get a clean zero, the evidence suggests that the problem is with gases internal to the instrument. The carrier gas, which passes through the cold trap at several stages of operation, is the most likely source. We compared contamination from two He standards (Fig. 2).

Conclusion

The total hydrocarbon contamination specification in helium cylinders is more important than using UHP Grade helium.

Figure 1. The effect of carrier gas hydrocarbon contamination on zero offset. Ultra-high purity helium was specified at 500 ppb THC. Technical grade helium was specified at 100 ppb THC.
Figure 2. Chromatograms generated with and without contamination demonstrate residual peak interference. Although peak shape for a 2 nmol mol$^{-1}$ (parts per billion, ppb) standard appeared adequate (a), a control blank still had a residual peak at the same retention time (b). Adjusting column temperature and pressure programs did not separate the contaminant peak from the ethylene peak. Although ultra-high purity (UHP) grade helium (99.9995% purity) was used, the gas contained 500 ppb total hydrocarbon contamination (THC) per cylinder. Technical grade helium (99.995% purity) with 100 ppb THC, coupled with an inexpensive hydrocarbon filter (Scottgas #5344H, ~$50) removed the residual peak (d).