Gas Chromatography Problem Solving and Troubleshooting

Question

I have a GC–MS equipped with a small capacity (1 mL/min) vacuum pump and manual carrier gas controls. I was told that the carrier gas flow rate into the mass spectrometer exceeded the pumping capacity of the vacuum system and this may cause a decrease in sensitivity. How do I determine the carrier gas flow rate since it cannot be directly measured with a flow meter?

Answer

The carrier gas flow rate at the column outlet can be calculated using available column and system parameters. The flow rate at the column outlet is required because the flow rate into the MS is the desired measurement. Equation 1 is used to calculate the carrier gas flow rate at the column outlet for vacuum outlet conditions:

$$F = 60\pi r^2 \left(\frac{T_{ref}}{T}\right)\left(\frac{2p_i}{3p_{ref}}\right)\bar{u} \qquad \text{Eq. 1}$$

where F is the flow rate at the column outlet (mL/min), r is the column radius (cm), T is the column temperature (K), T_{ref} is the reference temperature (298 K), p_i is the inlet pressure (dyn/cm^2), p_{ref} is the reference pressure (1.013 × 10^6 dyn/cm^2), and $\bar{u}$ is the average linear velocity (cm/s).

It should be noted that a different equation is used for nonvacuum column outlet conditions. Some of the equation terms are different from standard or familiar GC units and need to be carefully converted into the required units. Also, the inlet pressure is the gauge pressure plus the ambient pressure.

A column outlet carrier gas flow rate of 1.2 mL/min was obtained from the following parameters used in the GC–MS system. The column used was 30-m × 0.32-mm i.d. (r = 0.016 cm), the column temperature was 100°C (373 K), the inlet pressure was 2 psig (137,895.2 dyn/cm^2 + 1.013 × 106 dyn/cm^2 = 1,150,895.2 dyn/cm^2) (1 psi = 68,947.6 dyn/cm^2), and the average linear velocity (helium) was 40.2 cm/s. Because 1 mL/min was the vacuum pump capacity, the flow of 1.2 mL/min exceeded the pump limit with a resulting decrease in sensitivity.

An average linear velocity of 40.2 cm/s for helium is above the recommended range of velocities. Decreasing the inlet pressure to 1.0 psig (which is the lowest inlet pressure suitable for this system) resulted in an average linear velocity of 37.8 cm/s. Using these values, a column outlet flow rate of 1.0 mL/min was obtained, which is at the limit of the vacuum pump capacity. Inlet pressures below 2 psig can be very difficult to set and control. Retention time reproducibility problems are common at very low pressures unless the GC is specifically designed to handle very low inlet pressures. One method used to reduce the carrier gas flow rate is to use a smaller diameter column. Using a 30-m × 0.25-mm-i.d. column at 32.0 cm/s (inlet pressure = 7 psig) resulted in a column outlet flow rate of 0.7 mL/min. The 7-psig pressure was easier to set and control, and the lower flow rate resulted in better sensitivity. The 0.25-mm-i.d. column had lower sample capacity, but better peak resolution was obtained.

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 it 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.

Dean Rood
Associate Editor