Gas Chromatography Problem Solving and Troubleshooting

Question

I changed my carrier gas from helium to hydrogen. Although much shorter retention times were obtained, there was a noticeable loss in resolution. I thought that using hydrogen as a carrier gas would reduce the retention times and increase resolution. Why did the resolution decrease instead of increase?

Answer

The optimal average linear velocity for hydrogen and helium are different, thus method adjustments are needed when changing between the two carrier gases. For most capillary columns the optimal average linear velocity is 25–40 cm/s for helium and 40–80 cm/s for hydrogen. The best average linear velocity is dependent on a number of variables including column dimensions and compound retention. Primarily because of differences in gas viscosities, hydrogen and helium require a different head pressure in order to obtain their respective optimal average linear velocities for a specific size column. When changing carrier gases, the same head pressure cannot be used for both gases if optimal performance is desired.

An example of the head pressure differences between helium and hydrogen are shown in Table I. Helium at 10 psig delivered the average linear velocity that resulted in the highest resolution (0.82) in this example. Using hydrogen at 10 psig resulted in a significant drop in resolution (0.52); however, the retention times were more than two times lower. Adjusting the hydrogen head pressure to 6 psig provided the highest resolution (0.73), but it was still lower than the best obtained with helium (0.82). Compared with helium, the retention times were about 25% lower with a resolution loss of about 11%. This specific example shows that higher efficiency or resolution is not always obtained with hydrogen, but shorter analysis times are. Although the loss with hydrogen was fairly small at its optimal average linear velocity, the loss was much larger when it was used at the same head pressure as helium.

A 15-m column was used in this example. For longer and smaller diameter columns and

Table I. Comparison of Optimal Average Linear Velocities*

Carrier gas	Head pressure (psig)	Average linear velocity (cm/s)	Retention time for propyl benzoate (min)	Retention time for 1-decanol (min)	Resolution
Helium	10	36.6	6.51	6.59	0.82
Hydrogen	10	82.2	2.86	2.88	0.52
Hydrogen	6	48.4	4.88	4.94	0.73

* The column used was a DB-5ms (15 m × 0.25 mm, 0.25 μm). The injector was in split mode and set at 100°C. The detector used was a flame ionization detector set at 300°C.

Table II. Comparison of Resolution Losses Above the Optimal Average Linear Velocities

Carrier gas	Head pressure (psig)	Average linear velocity (cm/s)	Time for propyl benzoate (min)	Retention time for 1-decanol (min)	Resolution
Hydrogen	6	48.4	4.88	4.94	0.73
Hydrogen	8	65.1	3.65	3.69	0.68
Helium	10	36.6	6.51	6.59	0.82
Helium	15	54.5	4.38	4.43	0.68

* The column used was a DB-5ms (15 m × 0.25 mm, 0.25 μm). The injector was in split mode and set at 100°C. The detector used was a flame ionization detector set at 300°C.

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Associate Editor

more highly retained compounds, the resolution obtained with hydrogen became equivalent or better than those obtained with helium, and the analysis times were substantially shorter (1). One less obvious advantage with hydrogen carrier gas was its lower relative loss of efficiency or resolution as the average linear velocity increased beyond the optimum (Table II). For hydrogen, increasing the velocity by about 17 cm/s decreased resolution by 0.05, and an approximate 18-cm/s velocity increase resulted in a 0.14 resolution loss for helium. This means that hydrogen can be used at very high velocities with a less severe loss in resolution, thus it is often the best carrier gas when the shortest analysis times are desired.

Very small diameter capillary columns (e.g., 0.05- and 0.10-mm i.d.) require very high head pressures to obtain optimal linear velocities. Sometimes, hydrogen is the only carrier gas than can be used, because hydrogen requires about half the head pressure of helium at their respective optimal velocities. The primary drawback with hydrogen is its flammability. Although hydrogen fires or explosions are relatively rare when used as a capillary column carrier gas, the appropriate safety precautions need to be exercised.

References

1. D. Rood. *A Practical Guide to the Care, Maintenance, and Troubleshooting of Capillary Gas Chromatographic Systems*, 3rd ed. Wiley-VCH, Weinheim, Germany, 1999.