Technical Overview

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Why Do Loss of Resolution and Retention Time Shift Occur After Installing a New GC Column?

When a new column is installed on an instrument that has been running an application for a long time, and all the instrument parameters are set to the same values as before, the resolution between a pair of critical analytes can drop unacceptably, creating concern that the column is defective.

The chromatograms that accompany columns like these will show that the retention times of the poorly resolved peaks on the new column have shifted. This difference indicates a change in linear velocity of the carrier gas, since the run conditions were the same.

Why did the retention times of those compounds shift under seemingly identical run conditions?

The answer is that the run conditions are not identical because the columns are not truly identical. In any manufacturing process, products are built to tolerances. In the case of a GC capillary column, these tolerances include diameter and length. Even small differences in diameter and length will affect the actual velocity and flow rate through a column at a given head pressure.

The Dutch chemist J.J. van Deemter established the relationship between carrier gas velocity and efficiency. As shown in Figure 1, the number of theoretical plates—and the resultant resolving power—is greatly influenced by linear velocity. By adjusting the carrier gas velocity so that two compounds are eluted at the same retention time as before, the resolution can be restored to the desired level (Figure 2).

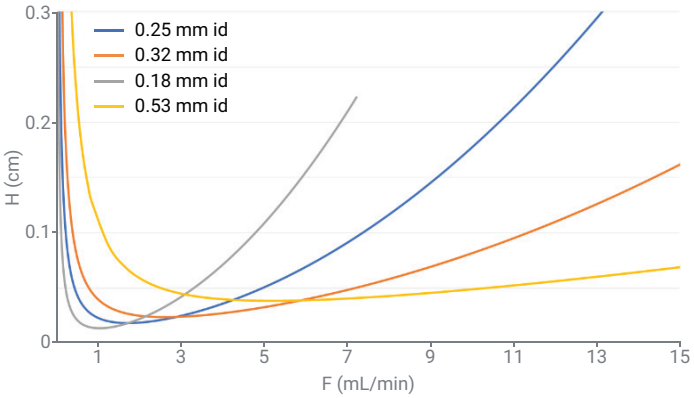

Figure 1. Van Deemter curves for common GC column internal diameters (mm id). The Y-axis shows the height of a theoretical plate (H), which is inversely proportional to the number of theoretical plates per meter. The smaller the H, the greater the efficiency of a column.

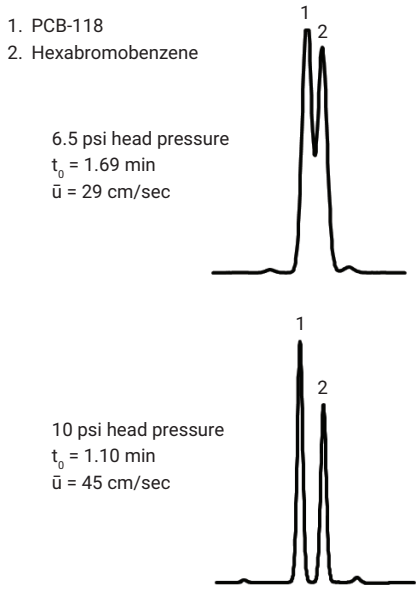

Figure 2. Agilent HP-5ms 30 m × 0.25 mm id, 0.25 µm.

Gas chromatography instruments do not have a velocity and flow-measuring device built in. The linear velocity displayed is calculated from the head pressure and the column dimensions that the user sets. If nominal values (30 m length, 0.25 mm diameter) are used, the instrument automatically calculates flow and linear velocity data for a 30 m × 250 µm column. Many columns do not have these exact dimensions, which makes it imperative to measure the actual linear velocity by injecting a nonretained compound (such as methane). By using its retention time and the actual length of the column, average linear velocity can be calculated with Equation 1. The flow rate can be calculated by incorporating the column radius with Equation 2.

Equation 1.

$$\bar{u} = L/t_0$$

Equation 2.

$$F = \pi r^2 L/t_0$$

Where:

ū = average linear velocity (cm/s)

F = average flow rate (mL/min)

L = length of column in cm

t_0 = retention time of nonretained peak

r = radius of column in cm

To operate under truly identical conditions, the column head pressure must be adjusted to match the retention time of a nonretained compound from the previous column. Relying solely on instrument features such as electronic pneumatics control (EPC) can lead to incorrect settings and cause problems.

To get a better feel for the effect that column dimensions can have on the head pressure/linear velocity relationship, please use the Agilent Pressure Flow Calculator, which is downloadable for free from the Agilent web site.

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