Gas Chromatography Problem Solving and Troubleshooting

Question

Because of drift to slightly shorter retention times during the past year, I was told that my GC oven needs to be recalibrated. Is this correct and how do I recalibrate the oven?

Answer

There are a number of more likely causes of the retention time drift than the oven, thus they need to be investigated before recalibrating the GC oven. A minor and often unnoticed change in the carrier gas average linear velocity can cause a measurable shift in retention time. Checking and resetting the velocity if needed should return the retention times close to their original values unless the column is much shorter than before. An older column in use for a prolonged period may have been periodically trimmed, thus the length would be considerably shorter than when the column was new. Not only are retention times shorter as a result of the shorter length column, the actual carrier gas average linear velocity is different than thought because it is being calculated using the original column length. Most other causes of retention shifts are more sudden (versus slowly drifting over time) or caused by a gross problem. These causes should be fairly obvious and easy to discover (e.g., different column, wrong oven temperature, different sample components or contamination, large sample concentration changes, and different carrier gas).

When other explanations for the retention shift can be eliminated, a few considerations or questions need to be explored before calibrating the GC oven. The importance and criticality of an accurate GC oven temperature needs to be weighed against the cost and labor of calibrating a GC oven. If a standard is injected to determine compound retention times and samples are compared with this specific standard, the need for an accurate oven temperature is less important. The inaccuracy in the retention times does not negatively affect the quantitative accuracy of the analysis except in a few select cases. The precision or reproducibility of the oven temperature is more important. The retention times of the sample are compared with a standard analyzed only minutes or hours before. If the retention times are slowly drifting over a period of weeks or months, the analytical results generated during one day will not be significantly affected.

If retention times are required to be within a range such as one specified in a method or SOP, accuracy becomes more important. Retention times falling outside of the range may result in noncompliance or may require extensive recalibration of peak identification data stored in data-reporting software. Even if carrier gas average linear velocities and column dimensions are within specifications, a slightly inaccurate oven temperature may still result in retention times being outside of the designated ranges. An example is shown in Table I of the impact of an oven temperature inaccuracy. A 1°C decrease in oven temperature resulted in a retention time increase of 0.17 min for

Table I. Comparison of Small Oven Temperature Differences*

	1-Methylnaphthalene			1-Undecanol		
	Retention time (min)	k	Retention index	Retention time (min)	k	Retention index
125°C	6.17	3.78	1324.51	7.68	4.95	1371.63
1°C lower†	6.34	3.91	1323.75	7.94	5.16	1371.63
0.3°C lower‡	6.22–6.25	3.82–3.84	1324.25–1324.40	7.74–7.80	5.00–5.05	1371.63

* Chromatographic conditions: DB-5ms column (30 m × 0.25 mm, 0.25 µm), split injector at 250°C with a 1:100 ratio, FID detector at 300°C, and hydrogen as the carrier gas at 38.8 cm/s.
† Average of six injections per temperature, measured.
‡ Average of six injections per temperature, calculated.

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Associate Editor

1-methylnaphthalene and 0.21 min for 1-undecanol. More importantly, the retention time or retention factor (k) shift for each compound was not the same—the relative retention or separation between the two compounds was no longer the same. This was also reflected by the different shifts in the retention indices for the two compounds (actually no retention index shift for 1-undecanol). Even for an 0.3°C difference, the retention time and index shifts were significant.

A less obvious problem may also occur with slight changes in column temperature. Because all compounds do not shift by the same amount with a change in oven temperature (as seen in Table I), it is possible for a change in resolution to occur. For two barely or partially resolved peaks, if one of the peaks shifts by a different amount than the other, then the two peaks will move closer or further apart. If they move closer, a loss of resolution will occur. A resolution increase will occur if the peaks move further apart. Resolution changes of 5–10% can occur depending on the compounds and column. In general, columns with more polar stationary phases are more severely affected by changes in oven temperature. The retention time and index shifts are greater and the differences between compounds more varied. This makes an accurate oven temperature more important and visible when using columns with highly polar stationary phases.

Calibrating a GC oven requires a very accurate temperature probe and a proper calibration knowledge of the specific GC model. A qualified service engineer should perform the calibration because inaccuracies may significantly affect retention times, retention indices, and peak resolution. Before the oven is recalibrated, the requirements of the analysis and the criticality of the results need to be balanced against the effort and cost of recalibration.