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Application News

No. G302A

Gas Chromatography

An Examination of the Effects of High/Low Column Cooling Rates Using Nexis™ GC-2030

Gas chromatography is a technique for separating components within a sample by utilizing the adsorption and desorption of components from a column when it is heated. The adsorption and desorption differ according to the column's liquid phase type. After analysis, the column must be cooled down to the initial temperature, but the rate at which the column is cooled may affect the column's liquid phase. Until now, priority was placed on shortening cooling times in order to shorten analysis cycles. However, the Nexis GC-2030 now allows operators to select a column cooling rate of their choice. By selecting the optimum cooling rate, the life time of the column can be maximized.

This article compares the baseline noise and the S/N ratio of peaks according to different cooling rates when using the Nexis GC-2030 cooling program.

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Column Liquid Phase

Fig. 1 shows the liquid phase model of a column coated with methyl phenyl polysiloxane. Each of the individual figures show the liquid phase structure in differing states: Fig. 1-1 at a high temperature, Fig. 1-2 at a low temperature, and Fig. 1-3 after rapid cooling.

At a high temperature, molecular mobility is increased and the liquid phase expands whereas at a low temperature the molecular mobility is suppressed and the liquid phase contracts. If the temperature change from high to low is gradual, the liquid phase expands and contracts without damaging its structure. However, if the column oven is cooled rapidly, the liquid phase is damaged as shown in Fig. 1-3 in which side chains with a large molecular structure in the liquid phase polymer are cut off.

The following sections study the affects of this damage on analysis results with respect to the stability and noise of the baseline and S/N ratios.

Baselines

Table 1 lists the gas chromatography (GC) analytical conditions used in this study and Fig. 2 shows the enlarged chromatogram of nine compounds. Using methanol as a solvent, a sample was prepared so that it contains the nine compounds indicated in Fig. 2 each at a concentration of 1 vol%. This sample solution was analyzed continuously 100 times using the analytical conditions listed in Table 1 with differing column cooling rates. Fig. 3 shows an enlarged view of the baseline of the 100th analysis with a low cooling rate (red line) and that with a high cooling rate (black line).

Looking at Fig. 3, we can see that the noise of the baseline is greater with the high cooling rate compared to that with the low cooling rate. This is because side chains in the column's liquid phase are cut off when using a high cooling rate and those chains are detected when the column is heated the next time around.

This result indicates that the cooling rate affects both the liquid phase of the column and the analysis results.

Table 1 GC Analytical Conditions

Model	: Nexis GC-2030/AOC-20i
Injection Mode	: Split mode
Injection Volume	: 1.0 µL
Split Ratio	: 1:50
Injection Temp.	: 250 °C
Carrier Gas	: He
Carrier Gas Control	: Constant linear velocity (30 cm/sec)
Column	: SH-50 (30 m × 0.32 mm I.D., 1.00 µm) *1
Column Temp.	: 40 °C (0 min.) - 4 °C/min. - 280 °C (0 min.)
Detector	: FID
Detector Temp.	: 320 °C
Detector Gas	: He 32.0 mL/min, Air 200 mL/min
Makeup Gas	: He (24 mL/min)

*1 P/N: 227-36167-01

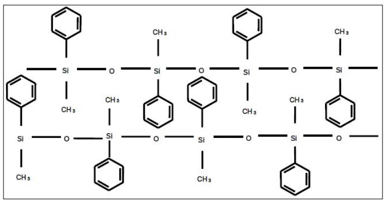

Fig. 1-1 Model of Liquid Phase Structure at High Temperature

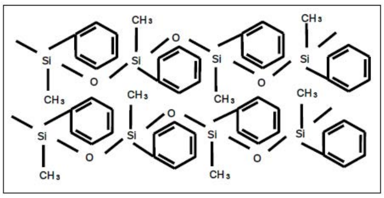

Fig. 1-2 Model of Liquid Phase Structure at Low Temperature

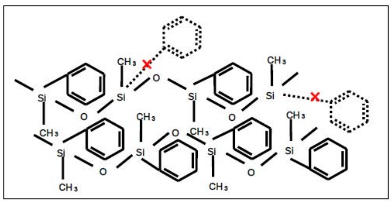

Fig. 1-3 Model of Liquid Phase Structure After Rapid Cooling

Fig. 1 Model Diagrams of the Column Liquid Phase

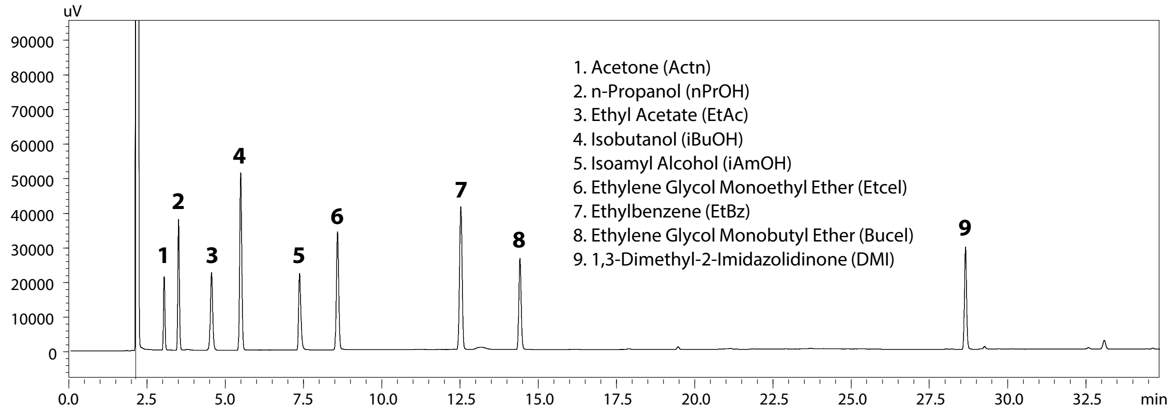

Fig. 2 Enlarged Chromatogram of Nine Compounds

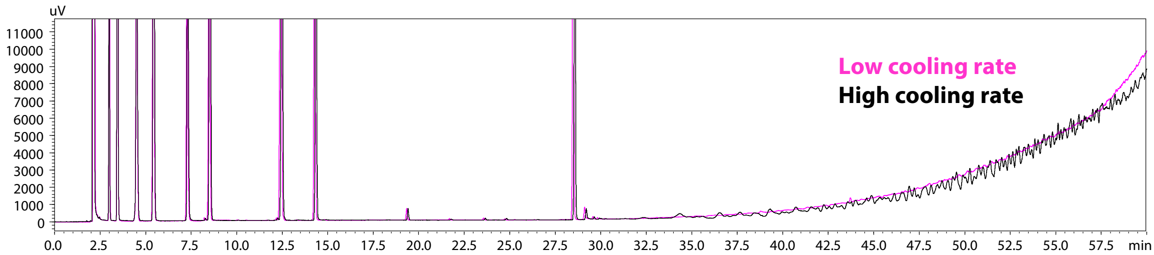

Fig. 3 Enlarged View of Baselines from the 100th Analysis with a High Cooling Rate and with a Low Cooling Rate

Table 2 Comparison of Noise and S/N Ratios

		Low Cooling Rate			High Cooling Rate		
		5th	50th	100th	5th	50th	100th
	Noise	128	81	76	631	284	259
1	Acetone	252	403	390	31	68	73
2	n-Propanol	310	494	525	60	129	151
3	Ethyl Acetate	170	264	297	34	65	81
4	Isobutanol	409	653	695	83	178	204
5	Isoamyl Alcohol	179	289	304	36	80	90
6	Ethylene Glycol Monoethyl Ether	270	430	460	55	120	136
7	Ethylbenzene	369	575	590	64	129	136
8	Ethylene Glycol Monobutyl Ether	211	346	362	43	97	107
9	1,3-Dimethyl-2-Imidazolidinone	249	358	378	50	105	115

Baseline Noise and S/N Ratios

Table 2 summarizes the baseline noise and the S/N ratios of the nine compounds from the 5th, 50th, and 100th analyses when using a fast cooling rate and when using a low cooling rate.

The values in Table 2 indicate that the baseline noise is smaller and S/N ratios are higher when using a low cooling rate compared to when using a high cooling rate.

When using a test method involving S/N ratios, using a low cooling rate is likely to be effective.

Conclusion

When performing analyses repeatedly on a gas chromatograph, the cooling conditions for the oven may affect the liquid phase of the column depending on its type.

Until now, gas chromatographs were designed with an openable flap on the back side so that cooling times can be shortened and thereby shorten analysis times.

The Nexis GC-2030 allows operators to select the optimum cooling rate for the column that is used. This can maximize the column performance over a long use period while also enabling the acquisition of favorable analysis results.

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