SHIMADZU APPLICATION NEWS

LA180-E011

● GAS CHROMATOGRAPHY

No.G213

Reduction of Analysis Time in Capillary GC (Part 5)
Factors Related to Analysis Time (Analysis with Temperature Program)

The previous Application News G212 introduced basic information on reducing analysis time in isothermal analysis. It presented the results of investigations into how the analysis time and resolution change due to changes of the column temperature, carrier gas linear velocity, column length, or liquid-phase film thickness. This Application News introduces similar tests conducted for analysis with temperature program.

The retention time and resolution for toluene, ethyl benzene, m-xylene + p-xylene, and o-xylene were investigated using a nonpolar column.

Fig. 1 shows the chromatograms for heating rates of 5˚C/minute, 10˚C/minute, and 20˚C/minute. Table 1 shows the retention times and resolutions. Increasing the programming rate by 4 times halved the analysis time, with a 20% to 30% drop in resolution.

Fig. 2 shows the chromatograms for carrier gas linear velocities of 20cm/s, 40cm/s, and 80cm/s (at 5˚C/minute heating rate). Table 2 shows the retention times and resolutions. Increasing the carrier gas linear velocity by 4 times approximately halved the analysis time but caused no apparent drop in resolution.

Fig. 3 shows the chromatograms for column lengths of 30 m and 60 m (at 10˚C/minute heating rate and 40 cm/s linear velocity). Table 3 shows the retention times and resolutions. Halving the column length resulted in about 2/3 the analysis time with approximately 10% to 20% drop in resolution.

Fig. 4 shows the chromatograms for the liquid-phase film thickness of 1µm and 0.25µm (at 5˚C/minute heating rate and 40cm/s linear velocity). Table 4 shows the retention times and resolutions. Reducing the film thickness to 1/4 resulted in approximately 1/2 the analysis time, with approximately 10% to 20% drop in resolution.

The results above indicate that the analysis time for analysis with temperature program can be approximately halved by increasing the heating rate by four times, increasing the carrier gas linear velocity by four times, reducing the column length to 1/4, or reducing the liquid-phase film thickness to 1/4. However, with most of these methods, poorer resolution accompanies the reduced analysis time. Of these methods, increasing the carrier gas linear velocity appears to have comparatively little detrimental affect on the resolution.

Therefore, the time of analysis with temperature program can be most easily reduced by increasing the carrier gas linear velocity, as is the case for isothermal analysis. If resolutions between each components are sufficiently high, the analysis time can also be efficiently reduced by increasing the heating rate in the range where the separation state is not deteriorated. The other methods require replacing the column, with associated cost and work.

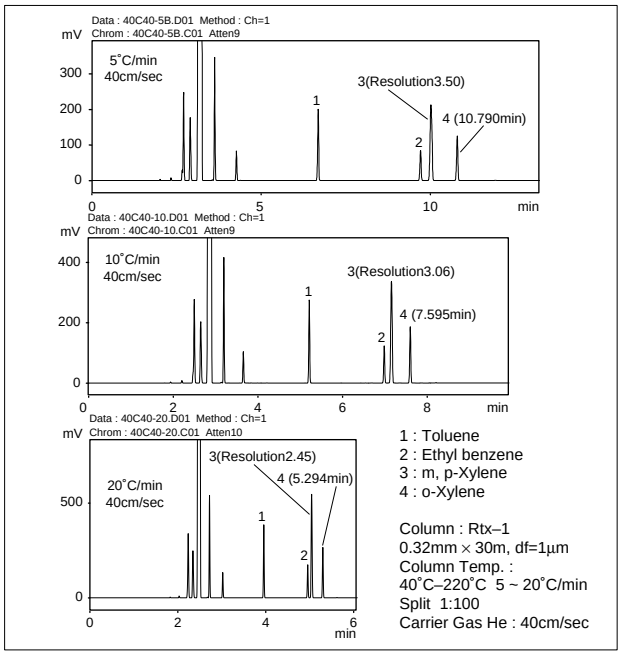

Fig. 1 Factors Related to Analysis Time 1 – Relationship between Heating Rate and Retention Time

Table 1 Factors Related to Analysis Time 1 – Relationship between Heating Rate and Retention Time

	5˚C/min 40cm/sec		10˚C/min 40cm/sec		20˚C/min 40cm/sec	
	Rt (min)	Resolution	Rt (min)	Resolution	Rt (min)	Resolution
Toluene	6.685		5.209		3.952	
Ethylbenzene	9.706	47.88	6.980	40.87	4.951	32.16
m, p-Xylene	10.011	3.50	7.151	3.06	5.041	2.45
o-Xylene	10.790	8.73	7.595	7.88	5.294	6.79

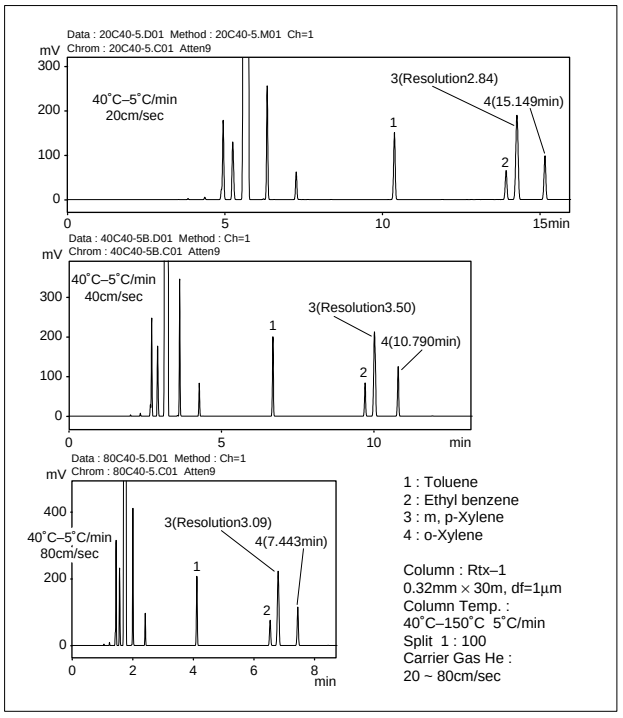

Fig. 2 Factors Related to Analysis Time 2 – Relationship between Carrier Gas Linear Velocity and Retention Time

Table 2 Factors Related to Analysis Time 2 – Relationship between Carrier Gas Linear Velocity and Retention Time

	5˚C/min 20cm/sec		5˚C/min 40cm/sec		5˚C/min 80cm/sec	
	Rt (min)	Resolution	Rt (min)	Resolution	Rt (min)	Resolution
Toluene	10.371		6.685		4.112	
Ethylbenzene	13.916	36.29	9.706	47.88	6.530	37.91
m, p-Xylene	14.258	2.84	10.011	3.50	6.794	3.09
o-Xylene	15.149	7.29	10.790	8.73	7.443	7.47

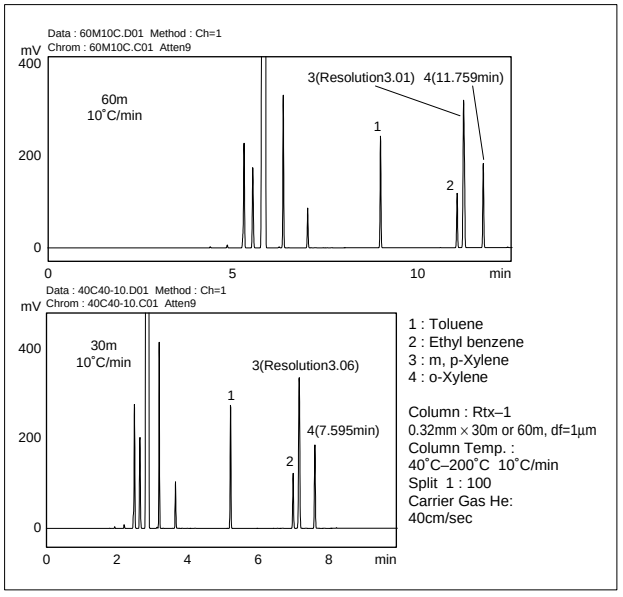

Fig. 3 Factors Related to Analysis Time 3 – Relationship between Column Length and Retention Time

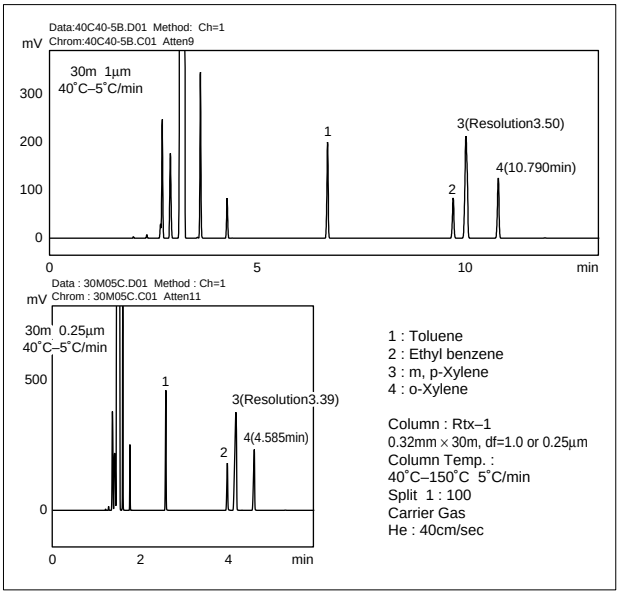

Fig. 4 Factors Related to Analysis Time 4 – Relationship between Column Film Thickness and Retention Time

Table 3 Factors Related to Analysis Time 3 – Relationship between Column Length and Retention Time

	30m 40cm/sec		60m 40cm/sec	
	Rt (min)	Resolution	Rt (min)	Resolution
Toluene	5.209		8.985	
Ethylbenzene	6.980	40.87	11.052	44.56
m, p-Xylene	7.151	3.06	11.228	3.01
o-Xylene	7.595	7.88	11.759	9.01

Table 4 Factors Related to Analysis Time 4 – Relationship between Column Film Thickness and Retention Time

5˚C/min	30m×0.25mm, 0.25µm		30m×0.25mm, 1µm	
	Rt (min)	Resolution	Rt (min)	Resolution
Toluene	2.573		6.685	
Ethylbenzene	3.971	41.10	9.706	47.88
m, p-Xylene	4.165	3.39	10.011	3.50
o-Xylene	4.585	6.77	10.790	8.73

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Printed in Japan 3100-06325-10A-IK