SHIMADZU APPLICATION NEWS

LAAN-A-G-E014

● GAS CHROMATOGRAPHY

No.G253

Introduction of a Multidimensional GC System

Accurate analysis of trace-level compounds in samples with complex matrices, such as refined petroleum products, flavor and fragrance formulations, and environmental samples, is not easy due to the difficulty in completely separating the target compounds from other compounds in the matrix. In the past, the approach to resolving this issue was to investigate the effectiveness of sample pretreatment procedures, separation columns and selective detectors, etc. However, with the recent adoption of multidimensional GC (MDGC) systems in which high resolution is achieved relatively easily, high-accuracy analysis has become possible, samples that contain trace-level analytes, such as those mentioned above. An MDGC system is a GC system incorporating two types of columns, so that components that cannot be separated by the first column are heart-cut and introduced into the second column for detailed separation. This Application News introduces such an MDGC system.

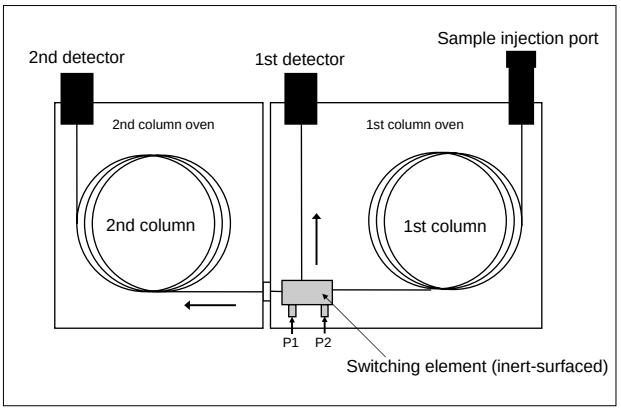

Fig.1 Configuration of an MDGC System

■ Configuration of an MDGC System

Fig.1 shows a configuration diagram of an MDGC system. MDGC systems can consist of combinations of two GC units as well as a GC and a GC/MS. The use of dual ovens allows the two columns to be set to different temperatures, enabling easy setting of the analytical conditions. Compounds that have eluted from the first column can be routed either to the first detector or to the second column (on the second detector side) utilizing the differential pressure-driven high-performance switching element.

Fig.2 shows a chromatogram of gasoline as one example of analysis. Gasoline is composed of a great many compounds, including hydrocarbons and aromatic series. This sample has been spiked with three types of ethers; however, they are not separated from the other matrix compounds by the first column. By heart-cutting the fraction containing the ethers and introducing this fraction into the second column, the ethers were completely separated from the remainder of the sample matrix.

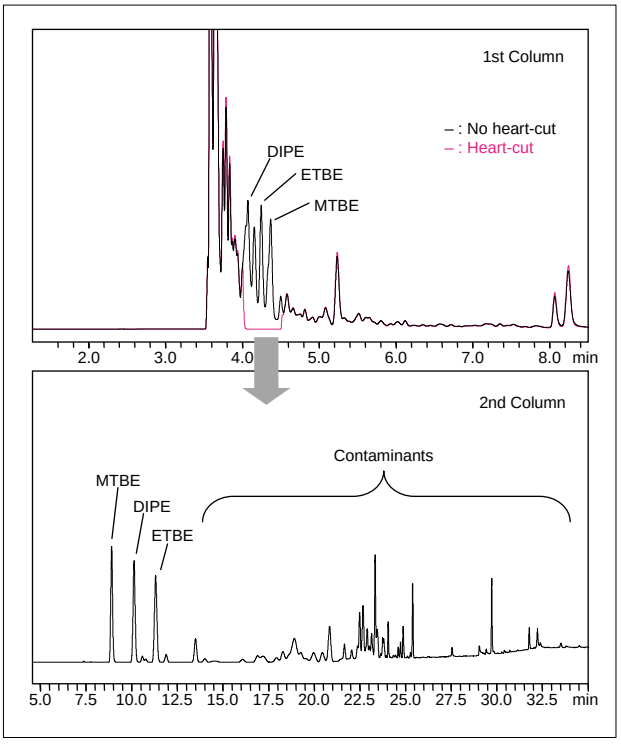

Fig.2 Analysis of Gasoline

The MDGC system achieves flow-line switching utilizing differential pressure provided by the APC (advance pressure controller). The mechanism is arranged such that the constituents eluted from the first column outlet are either introduced into the first detector or the second column (on the second detector side), depending on the change in the differential pressure at the first column outlet and at the second column inlet.

The switching mechanism greatly influences the analytical accuracy of the MDGC system. Since there is almost no change in outlet pressure at the first column before and after switching in the Shimadzu method, fluctuations in retention time are effectively suppressed.

■ Repeatability of an MDGC System in Heart-Cut Analysis

Here we conducted multiple analyses of a standard sample mixture of benzene, toluene, ethylbenzene, p-xylene, m-xylene, o-xylene, styrene and cumene, using a different number of heart-cuts for each analysis (heart-cut at 0 to 4 retention time segments). The chromatograms obtained from the first column elution are shown in Fig.3, and the respective peak retention times are listed together in Table 1. Even in the analyses using multiple heart-cuts, excellent repeatability is obtained with almost no fluctuation in retention times.

In addition, eight replicate analyses were conducted using heart-cuts at four retention time segments. The chromatograms obtained from elution of the second column are shown in Fig.4; the retention times and area values obtained from these chromatograms are listed together in Tables 2 and 3, respectively. Excellent repeatability was obtained for both retention time and area values.

These results demonstrate that heart-cut analysis with excellent repeatability can be conducted with the high-accuracy switching mechanism adopted in Shimadzu's MDGC system.

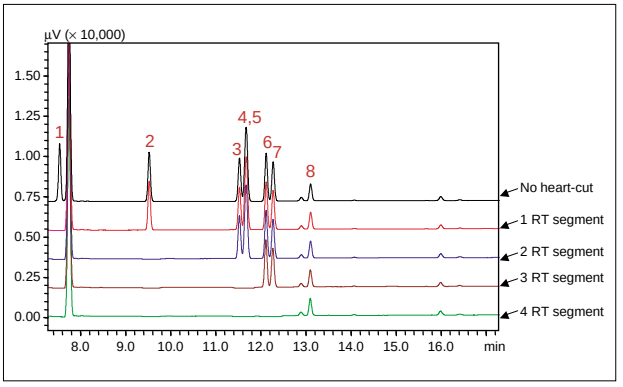

Fig.3 Chromatograms of a Standard Solution (1st Column)

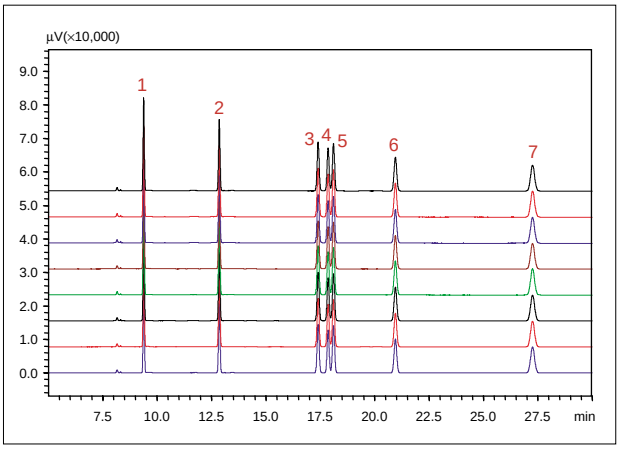

Fig.4 Chromatograms of a Standard Solution (2nd Column)

Table 1 Repeatability of Retention Time (1st Column)

	Peak 2	Peak 3	Peak 6	Peak 7	Peak 8
	Toluene	Ethylbenzene	Styrene	o-xylene	Cumene
No heart-cut	9.524	11.527	12.119	12.273	13.106
1 RT segment	9.524	11.527	12.119	12.274	13.104
2 RT segments	–	11.525	12.116	12.271	13.103
3 RT segments	–	–	12.111	12.267	13.098
4 RT segments	–	–	–	–	13.097
Avg.	9.524	11.526	12.116	12.271	13.102
SD	0.000	0.001	0.004	0.003	0.004
RSD (%)	0.000	0.010	0.031	0.025	0.030

Table 2 Repeatability of Retention Time (2nd Column)

	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7
	Benzene	Toluene	Ethylbenzene	p-xylene	m-xylene	o-xylene	Styrene
1st	9.378	12.852	17.396	17.855	18.102	20.950	27.259
2nd	9.378	12.851	17.396	17.855	18.103	20.949	27.256
3rd	9.378	12.851	17.394	17.854	18.101	20.948	27.252
4th	9.379	12.852	17.395	17.855	18.102	20.947	27.253
5th	9.379	12.851	17.396	17.853	18.103	20.948	27.255
6th	9.379	12.852	17.396	17.854	18.103	20.949	27.260
7th	9.379	12.852	17.396	17.855	18.103	20.950	27.256
8th	9.379	12.852	17.396	17.855	18.102	20.948	27.249
Avg.	9.379	12.852	17.396	17.855	18.102	20.949	27.255
SD	0.001	0.001	0.001	0.001	0.001	0.001	0.004
RSD (%)	0.006	0.004	0.004	0.004	0.004	0.005	0.013

Table 3 Repeatability of Peak Area (2nd Column)

	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7
	Benzene	Toluene	Ethylbenzene	p-xylene	m-xylene	o-xylene	Styrene
1st	109359	103286	98122	87393	100635	89242	103014
2nd	107650	102643	97770	87110	100270	89068	102838
3rd	107101	101808	96848	86377	99298	88344	101769
4th	108111	102053	96502	86214	99104	88183	101575
5th	107626	103159	98621	88180	101378	89808	103262
6th	108431	102794	97105	86489	100080	89016	102532
7th	107747	102812	98140	87346	100671	89322	102600
8th	105965	102231	98078	87469	100747	89448	102993
Avg.	107749	102598	97648	87072	100273	89054	102573
SD	986.76	525.11	742.80	668.14	764.48	547.21	605.10
RSD (%)	0.91	0.51	0.76	0.77	0.76	0.61	0.59

Table 4 Analytical Conditions

Model	: MDGC-2010, AOC-20i, GCsolution
1st Det	: FID
2nd Det	: FID
Fig. 2 Analytical Conditions	
1st Column	: Rt-TCEP 60 m × 0.25 mm I.D. df=0.4 µm
2nd Column	: DB-1 30 m × 0.32 mm I.D. df=3.0 µm
1st Column Temp.	: 35 ˚C (5 min)-5 ˚C/min-100 ˚C-20 ˚C/min -130 ˚C (10 min)
2nd Column Temp.	: 35 ˚C (20 min)-20 ˚C/min-200 ˚C-15 ˚C/min -240 ˚C (10 min)
Carrier Gas	: He 300 kPa
Switting pressure	: 120 kPa
INJ Temp.	: 135 ˚C
1st DET Temp.	: 250 ˚C
2nd DET Temp.	: 250 ˚C
Injection Method	: Split
Split ratio	: 1 : 300
Injection Volume	: 0.2 µL
Fig. 3, 4 Analytical Conditions	
1st Column	: DB-1 60 m × 0.32 mm I.D. df=5.00 µm
2nd Column	: ZB-WAX 30 m × 0.32 mm I.D. df=0.5 µm
1st Column Temp.	: 100 ˚C-10 ˚C/min-230 ˚C (10 min)
2nd Column Temp.	: 50 ˚C
Carrier Gas	: He 311 kPa
Switting pressure	: 180 kPa
INJ Temp.	: 300 ˚C
1st DET Temp.	: 310 ˚C
2nd DET Temp.	: 250 ˚C
Injection Method	: Split
Split ratio	: 1 : 100
Injection Volume	: 1.0 µL

NOTES:

✻This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

SHIMADZU

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