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Application Data Sheet

No.119

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Organic Solvents and Specified Chemical Substances in a Working Environment Using Two Different Columns (2)

Application Data Sheet No. 118 presented the successful separation of 58 organic solvents (including some specified chemical substances) subject to the working environment measurement, using two columns (Stabilwax and Rtx-624) with different liquid phases.
For the purpose of quantifying the 58 organic solvents automatically and continuously, analysis was performed using the Twin Line MS system, where the outlets from two different columns were connected to the MS unit simultaneously.
This Application Data Sheet introduces the results of investigating the quantitative analysis of 58 organic solvents using the Twin Line MS system.

Experiment

For the 56 solvents that are to be eluted with carbon disulfide, standard solvents were diluted with carbon disulfide to obtain twice the control concentration for each solvent and a standard mixture stock solution was prepared. (Preparation assumed that the amount of actual working environment sample collected was 1 L, and the amount of eluted solvent was 1 mL.) The mixed standard stock solution was further diluted with carbon disulfide to prepare standard solutions at concentrations of 1/1, 1/5, 1/10, 1/50 and 1/100 the control concentration, respectively. In the same way, methanol and isopropyl alcohol standards were diluted with purified water to prepare standard mixture solutions at concentrations of 2×, 1/1, 1/5, 1/10, 1/50 and 1/100 the control concentration, respectively.
These prepared standard solutions and standard stock solutions were measured using the analysis conditions in Table 1. High-sensitivity and high-accuracy analysis was possible by using the Smart SIM automatic method creation function.

Table 1: Analysis Conditions

Gas chromatograph mass spectrometer: GCMS-QP2020

GC		MS	
Column*1, 3:	Stabilwax (30 m × 0.25 mm I.D., 0.5 μm)*4	Ion source temp.:	200 °C
Column*2:	Rtx-624 (30 m × 0.25 mm I.D., 1.4 μm)*5	Interface temp.:	240 °C
Sample injection quantity:	1 μL*1, 2, 0.5 μL*3	Ionization current:	20 mA (high concentration)
Injection port temp.:	230 °C	Measurement mode:	SIM mode
Injection mode:	Split	Measurement mass range:	See Table 2.
Split ratio:	20 Note 1)	Event time:	0.2 sec
Control mode*1, 3:	Constant linear velocity (47 cm/sec)		
Control mode*2:	Constant linear velocity (49 cm/sec)		
Oven temp.*1:	50 °C (1 min) → (5 °C/min) → 70 °C → (25 °C/min) → 240 °C (2.5 min)		
Oven temp.*2:	50 °C (1 min) → (10 °C /min) → 80 °C → (40 °C/min) → 200 °C → (25 °C/min) → 230 °C (1.5 min)		
Oven temp.*3:	50 °C (1 min) → (10 °C/min) → 70 °C → (25 °C/min) → 240 °C (2 min)		

*1 Analysis conditions 1: 54 organic solvents
*2 Analysis conditions 2: Carbon tetrachloride and 1,2-dichloropropane
*3 Analysis conditions 3: Methanol and isopropyl alcohol
*4 Code No.: 10638 (Restek Corp., Shimadzu GLC)
*5 Code No.: 10968 (Restek Corp., Shimadzu GLC)
Note 1: Change the value depending on the amount of sample collected and the concentration range of the calibration curve.

<Twin Line MS System>

The outlets for two different columns were connected simultaneously to the MS unit, so data could be acquired for the different columns without compromising the vacuum in the MS unit. (A small quantity of helium gas flowed through the column that was not used for analysis to prevent column degradation.)

・Features of the System
1) There is no connector from the injection port to the MS unit, so there are no concerns about adsorption or contamination.
2) Provides smaller retention time fluctuation than the flow line switching system.
3) The simple system construction provides little possibility of carrier gas leakage.
4) For column maintenance, only a Vespel ferrule and a nut need replacement.
5) Use of two different columns can widen the scope of the investigation of analysis conditions when adding target components.

Analysis Results

Of the 56 organic solvents eluted with carbon disulfide, Fig. 1 shows the SIM chromatograms for methyl cellosolve (control concentration: 0.1 ppm), measured with analysis conditions 1, and carbon tetrachloride and 1,2-dichloropropane, measured with analysis conditions 2. For methyl cellosolve, sufficient sensitivity was obtained at the control concentration (standard solution concentration: 0.3 mg/mL). Carbon tetrachloride and 1,2-dichloropropane were able to be selectively detected using the Rtx624.

For the 56 organic solvents, calibration curves were created with the concentrations ranging from 1/100 the control concentration (1/10 for methyl cellosolve) to twice the control concentration. The correlation coefficients (R) for the calibration curves were at least 0.998, which were favorable results. In addition, the standard solutions at 1/00 the control concentration (standard solution at the control concentration for methyl cellosolve) were measured five times to calculate the repeatabilities. Favorable results were obtained for all components, with a repeatability of 4% max. Favorable results were also obtained for methanol and isopropyl alcohol, with a correlation coefficient (R) for the calibration curve of 0.999 min. and a repeatability of 3% max. The results are shown in Table 2.

From the results of investigating analysis of 58 organic solvents using the Twin Line MS system, it was proved that automatic and continuous measurements were possible while maintaining quantitative accuracy.

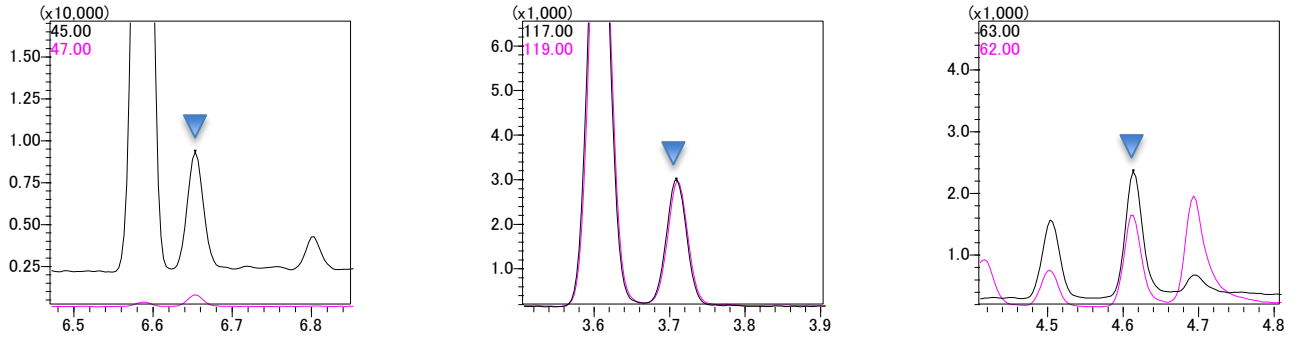

Fig. 2: SIM Chromatograms for Methyl Cellosolve, Carbon Tetrachloride, and 1,2-Dichloropropane
(Methyl cellosolve: 0.3 mg/mL, carbon tetrachloride: 0.3 mg/mL, 1,2-dichloropropane: 0.04 mg/mL)

Table 2: Calibration Curve Linearity and Repeatability
(1/100 the control concentration, except for methyl cellosolve, which was at the control concentration; n=5)

Name of Compound	Ions Monitored	%RSD	Correlation Coefficient: R	Name of Compound	Ions Monitored	%RSD	Correlation Coefficient: R
n-Hexane	86, 56	3.6	0.999	p-Xylene	106, 105	2.4	0.999
Ethyl ether	74, 45	3.3	0.999	1-Butanol	56, 43	1.8	0.999
Methylcyclohexane	82, 70	3.8	0.998	m-Xylene	106, 105	2.1	0.999
Acetone	58, 39	3.1	0.999	n-Pentyl acetate	70, 61	2.4	0.999
Methyl acetate	74, 59	2.6	0.999	Methyl Cellosolve	45, 47	3.4	0.999
trans-1,2-Dichloroethylene	96, 98	2.9	0.999	o-Xylene	106, 105	2.3	0.999
Tetrahydrofuran	72, 71	2.4	0.999	Isopentyl alcohol	70, 55	2.1	0.999
1,1,1-Trichloroethane	99, 97	2.8	0.999	Cellosolve	59, 72	2.3	0.999
Ethyl Acetate	70, 45	2.7	0.999	Chlorobenzene	112, 114	2.1	0.999
Isopropyl acetate	87, 59	3.0	0.999	Styrene	104, 78	2.3	0.999
Methyl ethyl ketone	72, 57	2.4	0.999	Cellosolve acetate	72, 59	2.1	0.999
Dichloromethane	84, 86	2.9	0.999	Cyclohexanone	98, 55	2.0	0.999
Benzene	78, 77	3.7	0.999	2-Methylcyclohexanone	112, 84	1.5	0.999
n-Propyl acetate	73, 42	3.5	0.999	N,N-Dimethylformamide	73, 30	2.7	0.999
cis-1,2-Dichloroethylene	96, 98	1.8	0.999	3-Methylcyclohexanone	112, 97	1.9	0.999
Trichloroethylene	130, 132	2.6	0.999	4-Methylcyclohexanone	112, 83	1.1	0.999
Methyl isobutyl ketone	100, 85	1.4	0.999	Butyl Cellosolve	87, 75	1.8	0.999
Isobutyl acetate	73, 56	2.1	0.999	Cyclohexanol	82, 67	2.3	0.999
2-Butanol	59, 41	1.8	0.999	cis-2-Methylcyclohexanol	96, 81	1.3	0.999
Chloroform	85, 83	1.4	0.999	trans-2-methyl-cyclohexanol	96, 81	0.7	0.999
Tetrachloroethylene	166, 164	2.4	0.999	1,1,2,2-Tetrachloroethane	83, 85	2.5	0.999
Toluene	91, 92	2.4	0.999	ortho-Dichlorobenzene	146, 148	2.2	0.999
1,4-Dioxane	88, 58	2.2	0.999	o-Cresol	107, 108	2.7	0.999
1,2-Dichloroethane	49, 64	3.2	0.999	p-Cresol	107, 108	1.3	0.999
n-Butyl acetate	73, 61	2.2	0.999	m-Cresol	107, 108	1.2	0.999
Methyl n-butyl ketone	58, 100	1.7	0.999	Carbon tetrachloride*1	117, 119	1.4	0.999
Isobutyl alcohol	43, 42	2.2	0.999	1,2-Dichloropropane*1	63, 62	1.9	0.999
Isopentyl acetate	70, 55	2.1	0.999	Methanol*2	31, 29	2.1	0.999
Ethylbenzene	106, 65	1.9	0.999	Isopropyl alcohol*2	45, 43	1.1	0.999

*1: Substances (two types) to be measured using carbon disulfide as the elution solvent and Rtx624 as the analysis column
*2: Substances (two types) to be measured using purified water as the elution solvent and Stabilwax as the analysis column
The other 54 substances are subject to measurement using carbon disulfide as the elution solvent and Stabilwax as the analysis column.

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Shimadzu Corporation
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