

Application Data Sheet

No. 118

GC-MS

Gas Chromatograph Mass Spectrometer

Analysis of Organic Solvents and Specified Chemical Substances in a Working Environment Using Two Different Columns (1)

Measurements of a working environment are mandated by Japan's Industrial Safety and Health Act for the purpose of preventing employee health problems caused by toxic factors in the workplace. These toxic factors include organic solvents and specified chemical substances. Many of these are measured using the solid sampling - gas chromatograph method.

This article investigates the analysis of 58 organic solvents (including some specified chemical substances) subject to the working environment measurement. A gas chromatograph mass spectrometer (GC-MS) was used, which effectively optimizes analysis and heightens analytical accuracy. This investigation used two analysis columns with different liquid phases.

From the results of this investigation, it was found that using two different columns made it possible to separate the 58 organic solvents with heightened identification accuracy.

Experiment

For 56 of the 58 organic 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.) In the same way, methanol and isopropyl alcohol standards were diluted with purified water 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.)

These standard stock solutions were then measured using the analysis conditions in Table 1.

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 μA (high concentration)
Injection port temp.:	230 °C	Measurement mode:	Scan mode
Injection mode:	Split	Measurement mass range:	<i>m/z</i> 20 to 250
Split ratio:	20	Event time:	0.3 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

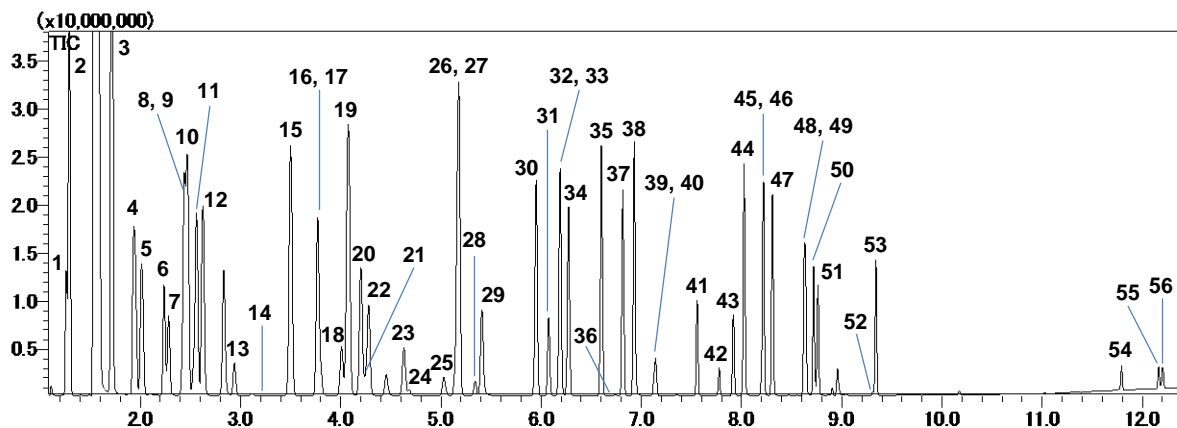
*4 Code No.: 10638 (Restek Corp., Shimadzu GLC)

*5 Code No.: 10968 (Restek Corp., Shimadzu GLC)

Analysis Results

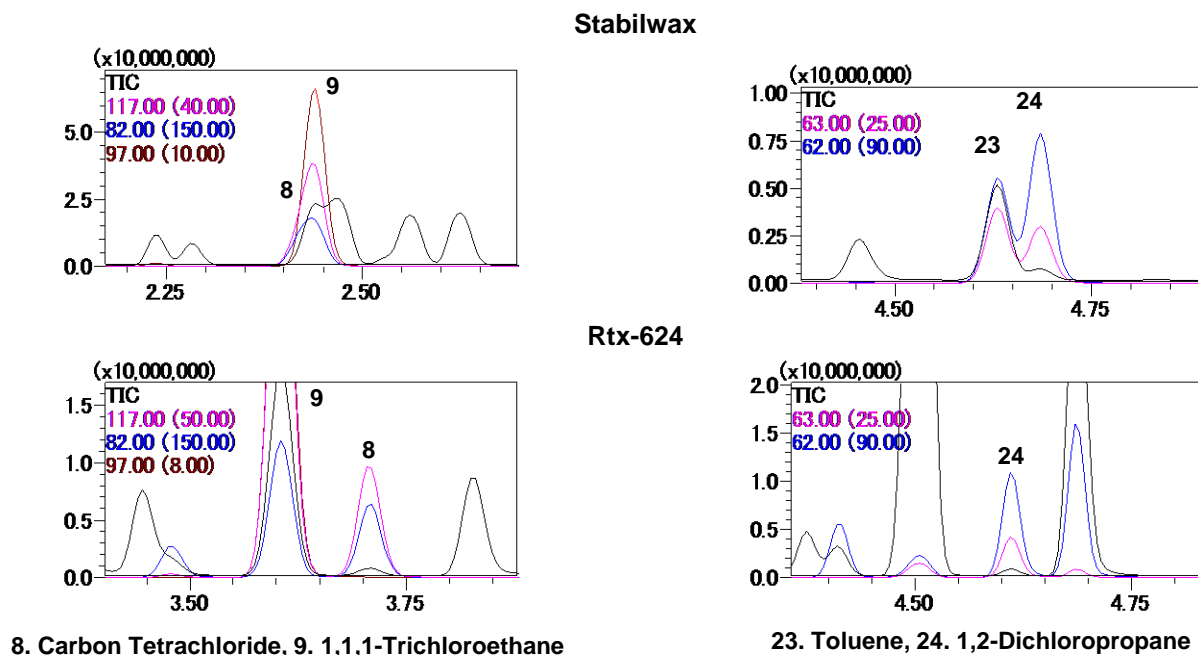
Fig. 1 shows the total ion current chromatogram obtained by measuring the 56 organic solvents eluted with carbon disulfide using analysis conditions 1. Separation was impossible in terms of retention time or *m/z* between carbon tetrachloride and 1,1,1-trichloroethane, and between toluene and 1,2-dichloropropane, respectively. Accordingly, they were analyzed using analysis conditions 2. The results showed that it was possible to separate these components using the retention times (Fig. 2). Methanol and isopropyl alcohol were measured using analysis conditions 3 (Fig. 3).

The use of two different columns made it possible to separate all 58 organic solvents with improved identification accuracy.



1. n-Hexane, 2. Ethyl ether, 3. Methylcyclohexane, 4. Acetone, 5. Methyl acetate, 6. trans-1,2-Dichloroethylene, 7. Tetrahydrofuran, 8. Carbon tetrachloride, 9. 1,1,1-Trichloroethane, 10. Ethyl Acetate, 11. Isopropyl acetate, 12. Methyl ethyl ketone, 13. Dichloromethane, 14. Benzene, 15. n-Propyl acetate, 16. cis-1,2-Dichloroethylene, 17. Trichloroethylene, 18. Methyl isobutyl ketone, 19. Isobutyl acetate, 20. 2-Butanol, 21. Chloroform, 22. Tetrachloroethylene, 23. Toluene, 24. 1,2-Dichloropropane, 25. 1,4-Dioxane, 26. 1,2-Dichloroethane, 27. n-Butyl acetate, 28. Methyl n-butyl ketone, 29. Isobutyl alcohol, 30. Isopentyl acetate, 31. Ethylbenzene, 32. p-Xylene, 33. 1-Butanol, 34. m-Xylene, 35. n-Pentyl acetate, 36. Methyl Cellosolve, 37. o-Xylene, 38. Isopentyl alcohol, 39. Cellosolve, 40. Chlorobenzene, 41. Styrene, 42. Cellosolve acetate, 43. Cyclohexanone, 44. 2-Methylcyclohexanone, 45. N,N-Dimethylformamide, 46. 3-Methylcyclohexanone, 47. 4-Methylcyclohexanone, 48. Butyl Cellosolve, 49. Cyclohexanol, 50. cis-2-Methylcyclohexanol, 51. trans-2-methyl-cyclohexanol, 52. 1,1,1,2-Tetrachloroethane, 53. ortho-Dichlorobenzene, 54. o-Cresol, 55. p-Cresol, 56. m-Cresol

Fig. 1: Total Ion Current Chromatogram for 56 Organic Solvents (Stabilwax)



8. Carbon Tetrachloride, 9. 1,1,1-Trichloroethane

23. Toluene, 24. 1,2-Dichloropropane

Fig. 2: Separation Between Carbon Tetrachloride and 1,1,1-Trichloroethane, and Between Toluene and 1,2-Dichloropropane (Upper: Stabilwax; Lower: Rtx-624)

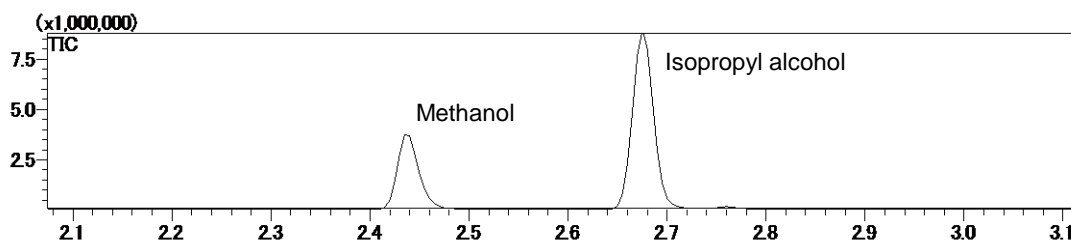


Fig. 3: Total Ion Current Chromatogram for Methanol and Isopropyl Alcohol (Stabilwax)