SHIMADZU

Application Data Sheet

No.17

GCMS

Gas Chromatograph Mass Spectrometer

Paint Thinner Analysis Utilizing Headspace GC-MS

Paint thinners, which are utilized to lessen the viscosity of paints, consist primarily of toluene, and contain other substances such as ethyl acetates and alcohols. As these compounds have anesthetic and excitatory properties, “paint sniffing”, or abuse by intentional inhalation, has become a social problem, leading to regulations targeting the inhalation of paint thinner for such purposes [1, 2]. Since GC-MS systems are often utilized in place of GC in scientific crime detection, this experiment investigated the ability of Headspace GC-MS to measure ethyl acetate, toluene, and methanol.

Experiment

Samples

Samples consisted of commercially-available paint thinner; samples of paint thinner and water in 2 separate phases, with either water or paint thinner as the primary ingredient; commercially-available adhesive; and tissue paper with paint thinner added.

Pretreatment

5 µL of commercially-available paint thinner was placed in a headspace vial (22 mL), which was then sealed with a headspace cap (aluminum cap: butyl rubber/PTFE).

Samples of paint thinner mixed with water, as well as paint thinner and water in 2 separate phases, were agitated, after which 5 µL samples were quickly extracted and placed in headspace vials. The vials were then sealed with headspace caps.

Approximately 50 mg to 100 mg of commercially-available adhesive was placed in a headspace vial, which was then sealed with a headspace cap.

In addition, 5 µL of commercially-available paint thinner was added to a sheet of tissue paper. The tissue paper was placed in a headspace vial, which was then sealed with a headspace cap, creating a sample consisting of tissue paper with paint thinner added.

Instrument

A direct connection between headspace and the GC-MS was adopted to minimize carrier gas consumption, and a Press-Tight connector (P/N: 221-38102-91) was used for the connection between columns. The analysis conditions are shown in Table 1.

Table 1: Analysis Conditions

HS	: TurboMatrix HS		
GC-MS	: GCMS-QP2010 Ultra		
[HS]		[GC]	
Headspace mode	: Constant	Vaporization chamber temperature: 200℃	
Injection time	: 0.02 min. injection *)	Column	: Rtx®-BAC2 (30 mL. x 0.32 mmI.D., df=1.2 µm Restek Co.)
Zone temperature settings: (O/N/T)		Column oven temperature: 40℃ (5min)→(40℃/min)→200℃ (1min)	
Oven temperature	: 60℃	Carrier gas	: Helium
Needle temperature	: 100℃	[MS]	
Transfer temperature:	150℃	Interface temperature: 230℃	Ion source temperature: 200℃
Sample shaker	: OFF	Solvent elution time	: 0.7 min Data sampling time: 1 – 10 min
GC cycle time	: 20 min	Measurement mode	: can Mass range: *m/z* 29-300
Pressurization time	: 1 min	Event time	: 0.5 sec Emission current: 150 µA (high sensitivity) Detector
Uptake time	: 0 min	voltage	: -0.25 kV (relative value) *
Warming time	: 15 min		
HS carrier gas pressure: 70 kPa			

*Note: The headspace sampler injection time and detection voltage must be optimized since they can differ depending on equipment status.

Results

The total ion current chromatogram obtained from measurements of commercially-available paint thinner is shown in Fig. 1. We succeeded in separating the three components of paint thinner in 10 minutes, and could identify them from the mass spectrum. Fig. 2 illustrates the total ion current chromatograms obtained from measurements of the sample of paint thinner mixed with water; the samples of paint thinner and water in 2 separate phases; the commercially-available adhesive; and the tissue paper with paint thinner added. Methanol, ethyl acetate, and toluene were detected in all of the samples. With the commercially-available adhesive, although the toluene peak in the total ion current chromatogram was small, the peak was clearly detected with the mass chromatogram (m/z 91, 92).

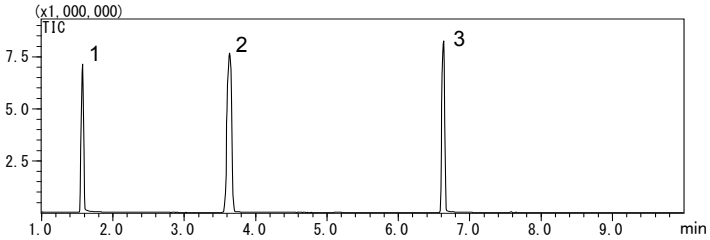

Fig. 1: Total Ion Current Chromatogram of Commercially-Available Paint Thinner
1 = methanol; 2 = ethyl acetate; 3 = toluene

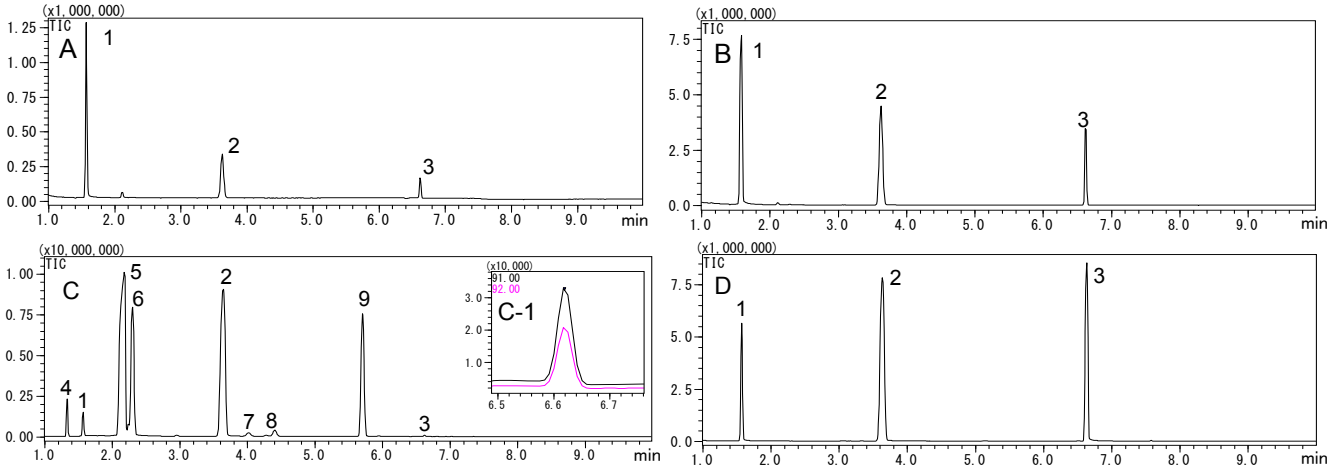

Fig. 2 Total Ion Current Chromatograms
(A: Sample with paint thinner mixed with water; B: Sample with paint thinner and water in 2 separate phases; C: Commercially-available adhesive; D: Tissue paper with paint thinner added)
1 = methanol; 2 = ethyl acetate; 3 = toluene; 4 = acetaldehyde; 5 = ethanol; 6 = acetone; 7 = 1,2-ethoxymethoxyethane; 8 = benzene, 9 = diethyl acetalC-1; Mass chromatogram for peak 3 (toluene) (m/z 91, 92)

Summary

Under the analysis conditions investigated, methanol, ethyl acetate, and toluene, the components of paint thinner, were separated with an analysis time of 10 minutes. This approach can evidently be applied to the measurement of various types of samples, including undiluted paint thinner. As this system can also be applied to the measurement of alcohol in blood, the type of measurement can be switched without replacing the column.

References

[1] Osamu Suzuki, Mikio Yashiki, editors: Dokuyakubutsu Bunseki Jissen Handbook – Chromatography-wo-cyushin-toshite (Practical Handbook for the Analysis of Toxic Pharmaceuticals, with a Focus on Chromatography) Jiho, Inc., Tokyo 2002.
[2] The Pharmaceutical Society of Japan, editors: Dokuyakubutsu-shiken-ho-to-cyukai 2006 – Bunseki, Dokusei, Taisho-ho (Testing Methods and Annotation for Toxic Pharmaceuticals 2006 – Analysis, Toxicity, and Coping Methods) Tokyo Kagaku Dojin, Tokyo, 2006.

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