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Application News

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GC/MS

Analysis of DMTS in Alcoholic Drinks Using SPME Arrow

Dimethyl trisulfide (DMTS) is known as an off-flavor compound in aged alcoholic drinks. This is due to an oxidation degradation which is caused by enzymes in the liquor. Thus, the ability to control the quality of alcoholic drinks by measuring DMTS has been attracting growing attention.

Additionally, many uses of solid phase micro extraction (SPME) to concentrate DMTS have been reported.

This report introduces the analysis of DMTS by the vapor phase extraction method and soaking extraction using SPME Arrow, which was shown to be more effective for concentration than the conventional SPME method.

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Materials

Standards for calibration curves

The DMTS standard was diluted with ethanol, and the standard solutions were prepared to make a concentration of 0.05 – 2 µg/L when 1 µL of the solution was spiked with 10 mL of 10% ethanol solution.

Standards used for calibration curves were prepared by mixing 3 g of sodium chloride with 10 mL of 10% ethanol solution in a 20-mL screw cap vial and adding 1 µL of each of the DMTS standard solutions.

Alcoholic drink samples

Two different Japanese sakes were prepared for this measurement. Each Japanese sake sample was diluted with purified water to make ethanol at a concentration of 10%. Samples used for analysis were prepared by mixing 3 g of sodium chloride with 10 mL of each of the diluted samples in a 20-mL screw cap vial.

Analytical Conditions

The instruments used and analytical conditions are shown in Table 1.

Table 1 Analytical Conditions

GCMS	: GCMS-QP™2020 NX
Autosampler	: AOC-6000
Column	: InertCap-PureWAX (Length: 30 m, I.D.: 0.25 m, df: 0.25 µm)
SPME Arrow conditions	
SPME Arrow	: DVB/Carbon WR/PDMS (Vapor phase: O.D.: 1.1 m, film thickness: 120 µm, length: 20 mm) (Soaking: O.D.: 1.5 mm, film thickness: 120 µm, length: 20 mm)
Conditioning Temp.	: 270 °C
Pre Conditioning Time	: 5 min
Incubation Temp.	: 35 °C
Incubation Time	: 5 min
Stirrer Speed	: 250 rpm (vapor phase)/0 rpm (soaking)
Sample Extract Time	: 30 min (vapor phase)/15 min (soaking)
Sample Desorb Time	: 2 min (250 °C: GC injection temperature)
GC conditions	
Injection Temp.	: 250 °C
Injection Mode	: Split (split ratio: 20)
Purge Flow Rate	: 3.0 mL/min
Control Mode	: Constant linear velocity (50.5 cm/min)
Column Oven Temp.	: 40 °C (2 min) → (30 °C /min) → 90 °C → (3 °C /min) →110 °C → (30 °C /min) → 250 °C (5 min)
MS conditions	
Interface Temp.	: 250 °C
Ion Source Temp.	: 200 °C
Measurement Mode	: SIM
Event Time	: 0.3 sec
Monitor Ion	: *m/z* 126, 79

Calibration Curves

Calibration curves are shown in Fig. 1. For both vapor phase extraction and soaking extraction, good linearity was obtained in the concentration range of 0.05 – 2 µg/L.

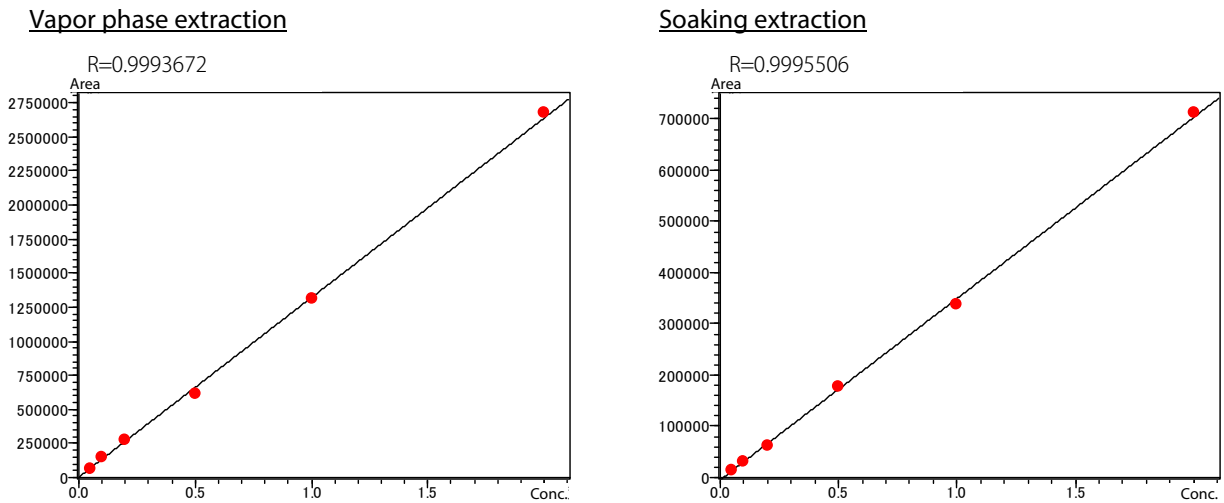

Fig. 1 Calibration Curves

SIM Chromatograms

Fig. 2 shows SIM chromatograms of Japanese sakes (blank) and Japanese sake samples spiked with 0.05 µg/L of DMTS.
We confirmed that DMTS can be detected successfully from different types of Japanese sake.

Repeatability and Recovery

Fig. 2 shows the repeatability (CV values) and the spike-and-recovery. We obtained favorable results with ≤ 12% of repeatability for both vapor phase extraction and soaking extraction. The spike-and-recovery was ≥ 70% for soaking extraction, but was < 70% in Company B's Japanese sake when vapor phase extraction was used.

Conclusion

This report compared vapor phase extraction and soaking extraction in the measurement of DMTS in alcoholic drinks using SPME Arrow. The results showed that a higher recovery was obtained by the soaking extraction method. Vapor phase extraction takes 30 minutes, but the soaking extraction can reduce this time by half.
These results indicate that soaking extraction is more effective for analysis of DMTS when the SPME Arrow method is used.

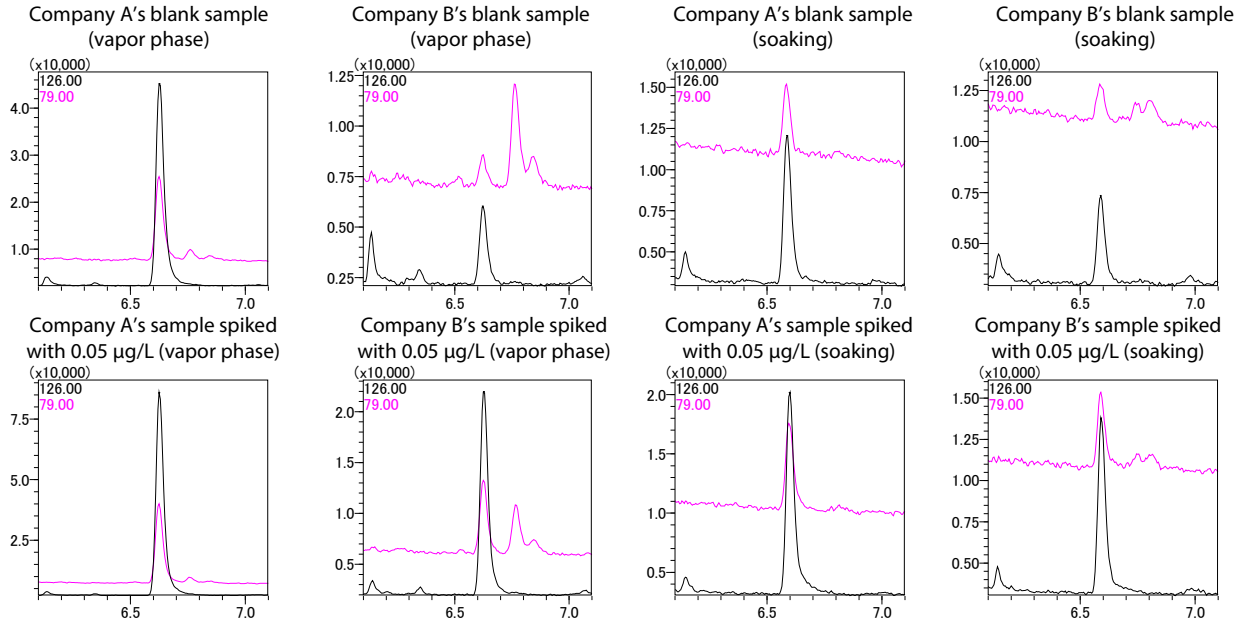

Fig.2 SIM Chromatograms of DMTS in Japanese Sake

Table 2 Repeatability and Recovery

		Vapor phase extraction			Soaking extraction		
		Mean value (µg/L)	CV value (%)	Recovery rate (%)	Mean value (µg/L)	CV value (%)	Recovery rate (%)
Company A	blank	0.088	5.5	-	0.077	2.5	-
	Spiked with 0.05 µg/L	0.146	3.2	116.0	0.114	5.9	74.0
	Spiked with 1 µg/L	0.923	3.0	83.6	0.934	5.1	85.7
Company B	blank	0.005	12	-	0.043	9.3	-
	Spiked with 0.05 µg/L	0.036	3.0	63.2	0.088	2.6	88.8
	Spiked with 1 µg/L	0.661	4.5	65.7	0.817	4.8	77.4

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