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Application Data Sheet

No. 73

GCMS
Gas Chromatograph Mass Spectrometer

High-Sensitivity Analysis of 2,4,6-Trichloroanisole in Wine Using Headspace-Trap GC/MS

< Introduction >
2,4,6-trichloroanisole (TCA) emitted from wine corks can taint wine and cause an objectionable odor. Due to the low threshold value for sensing the odor, highly sensitive measurements are required for monitoring. Conventionally, TCA was measured using methods such as purge and trap, which is very effective in concentrating samples, or thermal desorption. The HS-20 headspace sampler includes a trap function that is able to concentrate headspace gases. This Data Sheet describes an example of high-sensitivity measurement of TCA in wine using an HS-trap GC/MS system. The structure of TCA is illustrated in Figure 1 and the mass spectrum is shown in Figure 2.

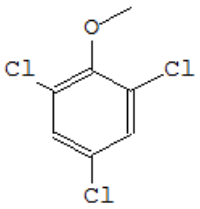

Fig. 1: Structure of TCA

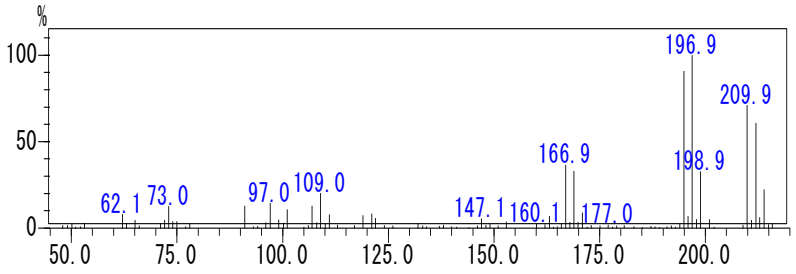

Fig. 2: Mass Spectrum of TCA

■Equipment and Analytical Conditions

Table 1: Analytical Conditions

HS-20	GCMS-QP2010 Ultra		
Mode	Trap	GC Unit	
Equilibrating time	30 min	Column	Rxi-5ms 0.32 mm I.D. × 60 m L., df=1.0 μm
Oven temp.	60 °C	Column oven temp.	50 °C (1 min) – 10 °C/min – 300 °C (5 min)
Sample line temp.	260 °C	Carrier gas control	Constant pressure
Transfer line temp.	260 °C	Carrier gas pressure	180 kPa
Trap equilibrating temp.	80.0 °C	Injection mode	Splitless
Trap cooling temp.	80.0 °C	Sampling time	3 min
Trap desorbing temp.	280.0 °C	Additional Flow	
Vial pressurizing time	2.0 min	APC1	100 kPa
Pressure equilibrating time	0.1min	APC3	50 kPa
Load time	0.1 min	MS Unit	
Load equilibrating time	0.1 min	Interface temp.	280.0 °C
Dry purge	5 min	Ion source temp.	230.0 °C
Injection time	20 min	Solvent elution time	14 min
Needle flush	20 min	Measurement start time	15 min
Injection cycle	3 cycles	Measurement end time	20 min
Cycle time	50 min	Measurement mode	SIM
Sample loading volume	5 mL	Selected ions (*m/z*)	211.9, 209.9, 196.9, 194.9
		Event time	0.2 sec

■Sensitivity

A wine sample spiked with the equivalent of 1 ng/L TCA was measured by SIM using the HS-trap method (Fig. 3).

The results show how the system was able to analyze low concentrations of TCA with high sensitivity.

A wine sample spiked with the equivalent of 100 ng/L TCA was measured by SIM using the headspace-GC/MS method and HS-trap method, as shown in Figures 4 and 5. A comparison of both shows that the HS-trap method provided about 10 times higher sensitivity.

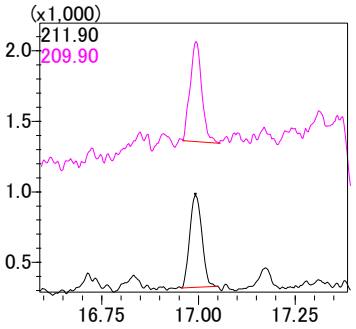

Fig. 3: SIM Chromatogram of TCA in Wine Measured Using HS-Trap (Wine spiked with 1 ng/L TCA)

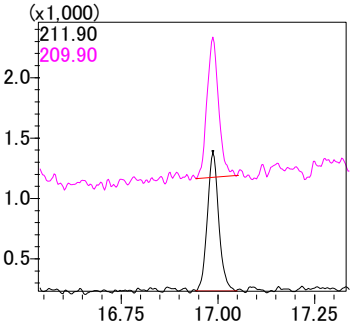

Fig. 4: SIM Chromatogram of TCA in Wine Measured Using Conventional Headspace GC/MS (Wine spiked with 100 ng/L TCA)

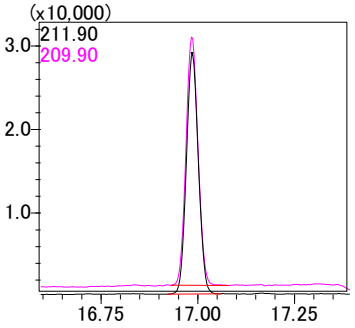

Fig. 5: SIM Chromatogram of TCA in Wine Measured Using HS-Trap (Wine spiked with 100 ng/L TCA)

■Linearity and Repeatability

Linearity was confirmed by adding specific concentrations of trichloroanisole to wine (from 1 to 100 ng/L, as shown in Figure 6). The results showed good linearity.

3 ng/L of trichloroanisole was added to wine to test the repeatability (n = 5) of peak area (Table 2). Results showed good repeatability, with a CV value not exceeding 5 %.

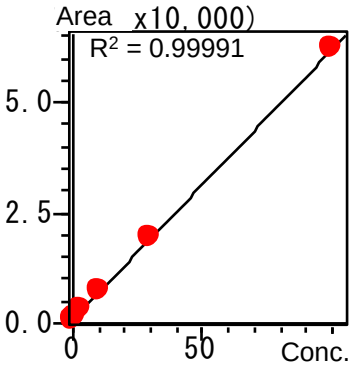

Fig. 6: Linearity of TCA (Wine spiked with 1-100 ng/L TCA)

Table 2: Area Repeatability of TCA (n=5, wine spiked with 3 ng/L TCA)

AREA	1	2	3	4	5	Average	%RSD
TCA m/z 211.9	3,103	3,051	2,925	3,020	2,742	2,968	4.79 %

■Conclusion

This Data Sheet describes an example of high-sensitivity measurement of trichloroanisole in wine using an HS-trap GC/MS system. The results showed that the system was easily able to measure even a few nanograms per liter. This also confirmed that an HS-trap-GC/MS system using the HS-20 headspace sampler is effective in monitoring trichloroanisole in wine.

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