

Analysis of Trichloroanisole in Wine Using Headspace-GC/MS

There are instances of wine being polluted and spoiled as a result of trichloroanisole, which exists in cork plugs. Because it has a low odor threshold, monitoring requires highly sensitive analysis. Conventional analysis methods include the purge & trap method and thermal desorption method, which

have a high degree of enrichment (Application News M172, etc). The features of headspace methods are easy operation and low contamination. This Application News introduces the results of the analysis of trichloroanisole in wine using headspace-GC/MS.

■ Analytical Conditions

Headspace sampler : TurboMatrix HS40 w/vial shaker (Perkin Elmer)
 Device : GCMS-QP2010 (SHIMADZU)
 Column : Rtx-5 MS (Restek, 30 m × 0.25 mm I.D. df = 1.0 μm)

HS	Sample Amount	: 10 mL	Sample Temp.	: 80 °C (30 min)
	Injection Time.	: 1.5 min	Needle Temp.	: 100 °C
	Transfer Temp.	: 180 °C	Agitation	: ON
	Carrier Gas Press.	: 150 kPa		
GC	Injector Temp.	: 220 °C		
	Column Temp.	: 35 °C (1 min)→15 °C/min→250 °C (5 min)		
MS	Interface Temp.	: 220 °C		
	Ion Source Temp.	: 200 °C		
	Monitor Ion	: 197, 210, 212		

■ Total Ion Chromatogram and Mass Spectrum

Fig.1 shows the total ion chromatogram and mass spectrum of a sample in which 400 ng/L of 2, 4, 6- trichloroanisole was added to wine.

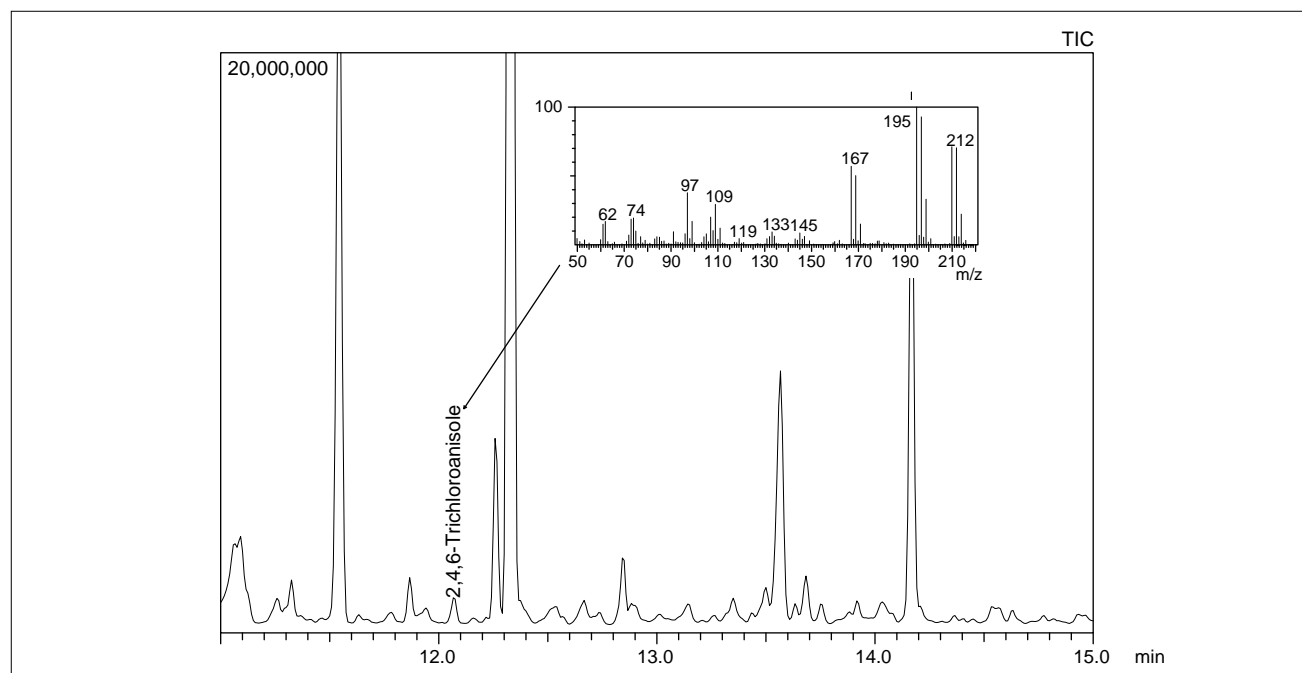


Fig.1 Total Ion Chromatogram and Mass Spectrum

■ Sensitivity

Fig.2 shown the SIM Chromatogram of a sample where the level of trichloroanisole in the wine was adjusted to by 4 ng/L. Using the headspace-GC/MS method, there are limits on injection volume and thus there is the possibility of insufficient sensitivity. However, as seen in Fig.2, even with the headspace-GC/MS method sufficient sensitivity is achieved.

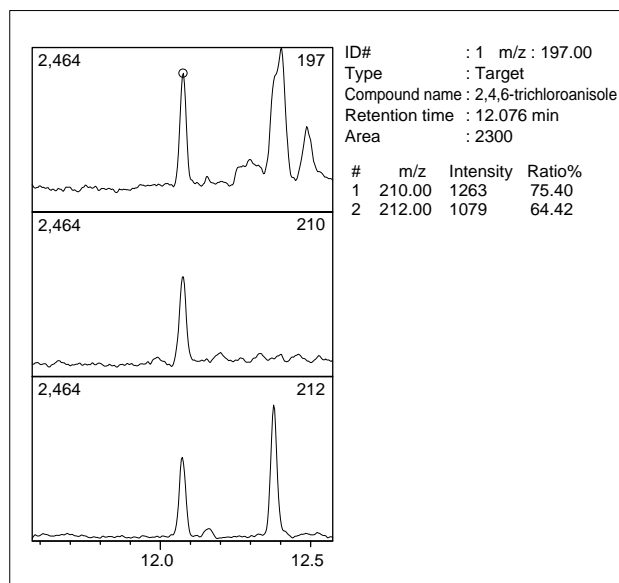


Fig.2 SIM Chromatogram (4 ng/L)

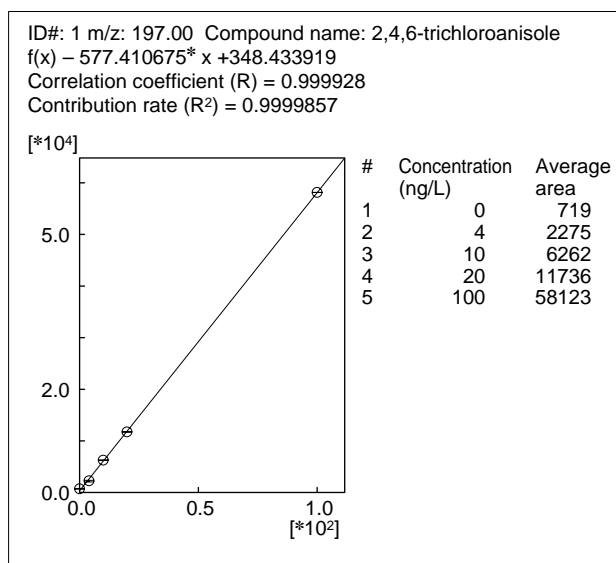


Fig.3 Calibration Curve (4 -100 ng/L)

■ Calibration Curve and Repeatability

In wine in which the compound was not included, trichloroanisole was added to achieve a specific concentration and then created the calibration curve. Those results are shown in Fig.3. Table 1 shows repeatability. The excellent result (%RSD<6 %) for a 4 ng/L sample was obtained.

Table 1 Repeatability (4 ng/L n=5)

Compound name	Analysis	Analysis	Analysis	Analysis	Analysis	RSD (%)
	1	2	3	4	5	
2,4,6- trichloroanisole	2056	2300	2345	2357	2318	5.47

■ Conclusion

This Application News explored the analysis of trichloroanisole in wine using headspace-GC/MS. The results confirmed that it is possible to achieve sufficient measurements even at the few ng/L level.

Thus, we were able to confirm that the headspace-GC/MS method is effective in the monitoring of trichloroanisole in wine.

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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