

Application Data Sheet

No.84

GC-MS

Gas Chromatograph Mass Spectrometer

Higher Sensitivity Analysis of 2-Methoxy-3-Isobutylpyrazine (MIBP) in Wine Using GC-MS/MS(PCI)

2-Methoxy-3-isobutylpyrazine (MIBP) is an aromatic substance with the fragrance of bell peppers. It is found in sauvignon blanc (a type of grape used for white wine) and cabernet sauvignon (a type of grape used for red wine), and gives the wines a favorable aroma. MIBP, which has a significant impact on the flavor of wine, has an extremely low threshold value in sensory tests, on the order of a few ng/L. Since wine contains many components, concentration and selective separation & detection are essential to analysis.

The trace amounts of MIBP in wine were selectively detected by utilizing the MonoTrap[®] silica monolithic absorbent for collection and concentration, and a GC/MS/MS (GCMS-TQ8030) in positive chemical ionization (PCI) mode. The MRM acquisition mode was used to monitor specific transitions for the compound of interest.

Experimental

A standard MIBP solution was added to commercially-available sauvignon blanc (produced in Chile in 2012) and cabernet sauvignon (produced in Chile in 2012) at different concentrations (0 ng/L, 1 ng/L, 5 ng/L, 10 ng/L, and 20 ng/L). The samples were heated for 1 hour at 50 °C, and then the gaseous phase MIBP was collected using MonoTrap[®] RGPS TDNote (GL Sciences, P/N: 1050-74202). After collection, the MonoTrap[®] RGPS TD was measured with the analysis conditions shown in Table 1.

Note: MonoTrap[®] RGPS TD: This is formed by applying a polydimethylsiloxane coating to a silica substrate, and applying end caps, with graphite carbon added to the absorbent.

Table 1: Analysis Conditions

Autosampler:	AOC-5000 Plus
OPTIC Liner Auto Exchange System:	CDC + LINEX
Multipurpose Injection Port:	OPTIC-4
GC-MS:	GCMS-TQ8030
Column:	InertCap 17MS (Length: 30 m, 0.25 mm I.D., df=0.25 µm), (GL Sciences, P/N: 1010-20142)
[OPTIC-4]	
Initial Temp.:	35 °C
Ramp Rate:	10 °C/sec
Hold Temp.:	250 °C
Hold Time:	300 sec
Column Flow1:	5 mL/min
Column Flow Time:	320 sec
Column Flow2:	1.5 mL/min
Split Flow1:	5 mL/min
Split Flow Time:	320 sec
Split Flow2:	50 mL/min
[GC]	
Column Oven Temp.:	40 °C (5 min) → (10 °C/min) → 280 °C (5 min)
[MS]	
Ion Source Temp.:	200 °C
Interface Temp.:	250 °C
Ionization Method:	Positive chemical ionization (PCI)
Reagent Gas:	Isobutane (60 kPa)
Acquisition Mode:	MRM
Event Time:	0.3 sec
Monitor Ion and Collision Energy (CE):	m/z 167.1 > 124.1 (20 V) m/z 167.1 > 135.1 (15 V)

Results

Fig. 1 shows the calibrations curves for sauvignon blanc and cabernet sauvignon via the standard addition method. Favorable results were obtained for the correlation coefficient (R) between area and concentration for each MIBP-spiked sample, with 0.9999 for the sauvignon blanc and 0.9998 for the cabernet sauvignon. Fig. 2 shows the MRM chromatograms for the MIBP in the wines. Table 2 shows the results of quantitatively analyzing each wine 3 times via the standard addition method and the repeatability. The respective concentrations of MIBP in the wines were 5.4 ng/L for the sauvignon blanc and 12.1 ng/L for the cabernet sauvignon. In addition, favorable results of 3 % RSD were obtained for the repeatability.

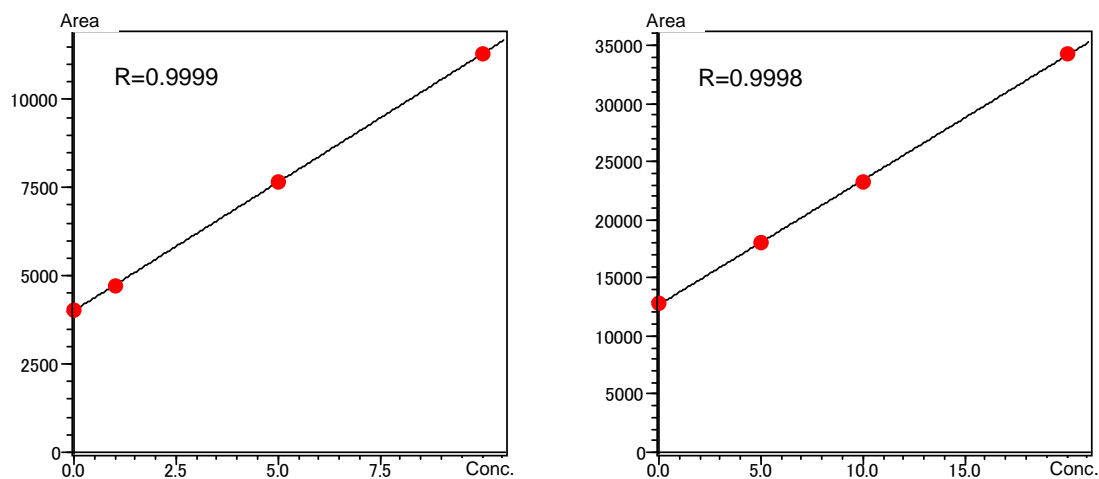


Fig. 1: Calibration Curves for the Wines via the Standard Addition Method

Left: Sauvignon Blanc (Concentrations of 0 ng/L, 1 ng/L, 5 ng/L, and 10 ng/L)

Right: Cabernet Sauvignon (Concentrations of 0 ng/L, 5 ng/L, 10 ng/L, and 20 ng/L)

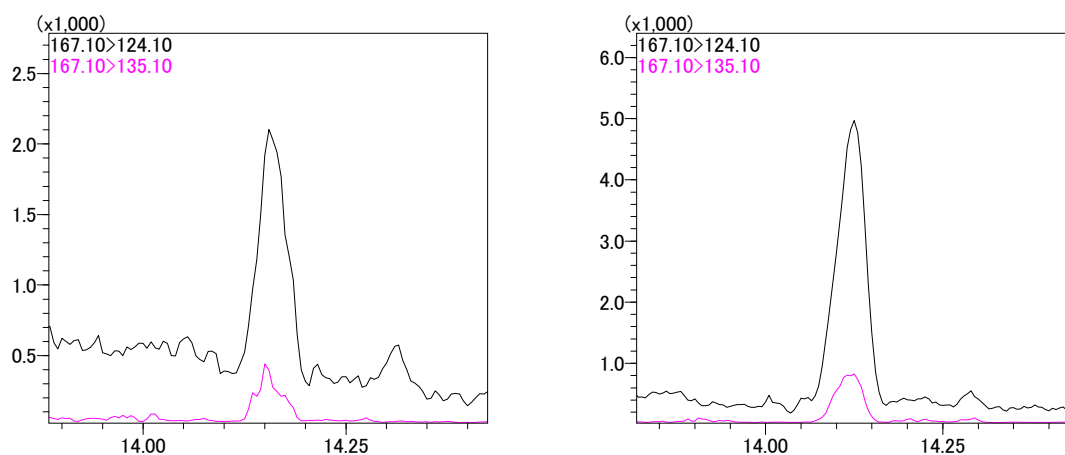


Fig. 2: MRM Chromatograms for the MIBP in the Wines (Left: Sauvignon Blanc, Right: Cabernet Sauvignon)

Table 2: Quantitative Results for the MIBP in the Wines via the Standard Addition Method
(Concentration Units: ng/L, and the Repeatability (n=3))

Wine Type	1	2	3	Average	Standard Deviation	C.V. (%)
Sauvignon Blanc	5.5	5.3	5.3	5.4	0.1	2.47
Cabernet Sauvignon	11.8	12.3	12.1	12.1	0.2	1.91

Conclusion

The trace quantities of MIBP in the wines were collected and concentrated by the MonoTrap® RGPS TD, and then selectively detected by utilizing the GC-MS/MS in PCI mode with MRM acquisition mode. It was thus possible to detect MIBP at the ng/L level with high sensitivity.

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