AAS
APPLICATION NOTES

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The determination of manganese in white spirits by flame atomic absorption spectrophotometry

GBC

Introduction

The determination of manganese in white spirits has usually been performed by a spectrophotometric technique based on the colour reaction of this metal. The sample was digested, then MnII was oxidized to MnO_4 using potassium periodate or ammonium persulphate in the presence of Ag^+ as a catalyst[1]. However, these methods are tedious and the reproducibility is unsatisfactory. Ribereau-Gayon et al[2] measured μg/L concentrations of manganese in wine by flame atomic absorption spectrometry after a pre-concentration procedure. Wine and brandy containing 42-556 μg/L manganese have been measured directly with a kinetic-spectrofluorometric method by Perez-Bendito[3].

The National Health Ministry of the Peoples Republic of China has set maximum levels of manganese at 2 mg/L for alcoholic beverages. Consequently a direct flame AAS method was developed. The method is simple and fast, and was applied to the measurement of 15 beverages taken from the Chinese market.

Experimental

Instrumentation

The GBC atomic absorption spectrophotometer was used. This instrument allows the construction of a calibration curve with up to 10 standards. The sample concentrations were then calculated in the desired units by the instrument from the graphed calibration curve. An air-acetylene flame was used and the operating parameters are listed in Table 1.

Element	Mn
Beam Mode	Double Beam
Wavelength (nm)	279.5
Slit Width (nm)	0.2
Atomization	Air-Acetylene
Lamp Current (mA)	5.0
EHT (gain)	– 444 V
Scale Expansion	1.000
Integration Time (sec)	1

Table 1: Operating parameters

Calibration

A stock standard manganese solution (100 mg/L) was provided by the Beijing Fifth Institute. Working standards of 0.25, 0.50, 1.00, 2.00, 4.00 and 8.00 mg/L were prepared by dilution of the stock solution in ethanol (ethanol absolute AR grade) and deionised water to make up each standard in 50% v/v ethanol and 0.15% v/v nitric acid. The calibration graph was linear from zero to 2.00 mg/L with a small degree of curvature from 2.00 to 8.00 mg/L. Samples were aspirated directly into an air-acetylene flame. Figure 1 shows the calibration curve.

Calibration Table		
Standard	**Absorbance**	**Concentration**
1	0.036	0.250
2	0.073	0.500
3	0.143	1.000
4	0.292	2.000
5	0.535	4.000
6	0.926	8.000

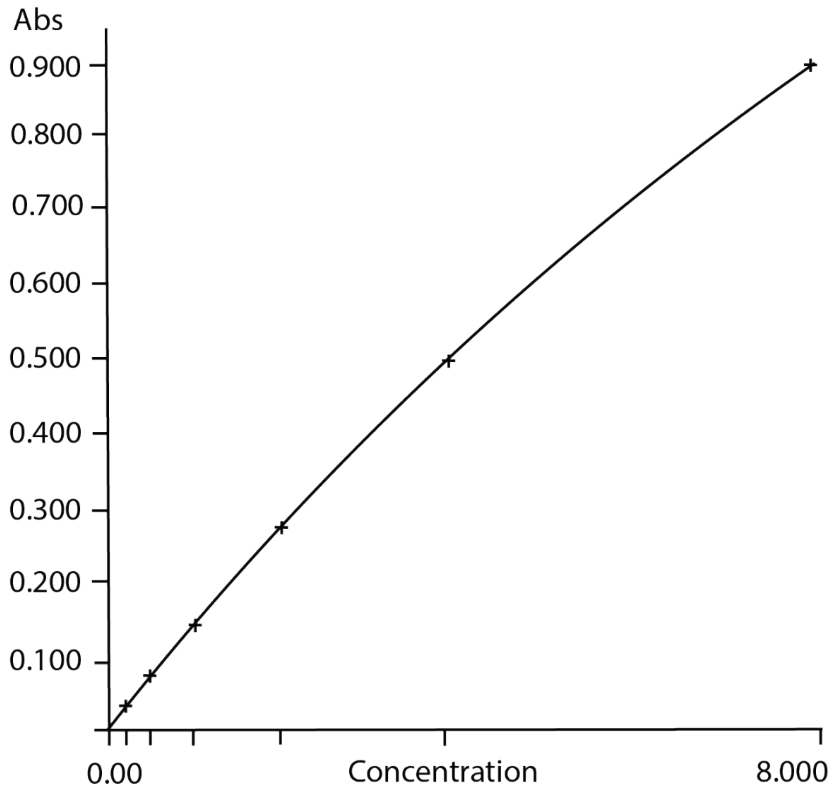


Figure 1: Calibration curve and calibration table for Mn in 50% v/v ethanol

Results and discussion

A major proportion of these beverages is ethanol (40-60%). At this concentration, ethanol will change the viscosity of the sample and hence affect the nebulizer efficiency. Ethanol was added to a solution containing 2 mg/L Mn in increasing concentrations (35% to 70%). The results graphed in Figure 2 indicated that absorbance did not change over the 35% to 60% v/v range for the added ethanol.

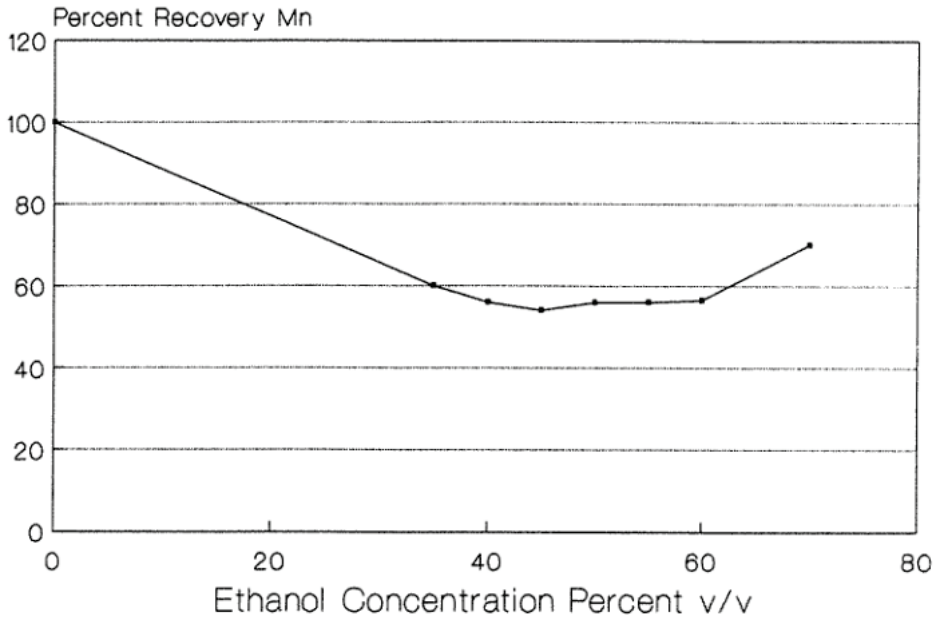
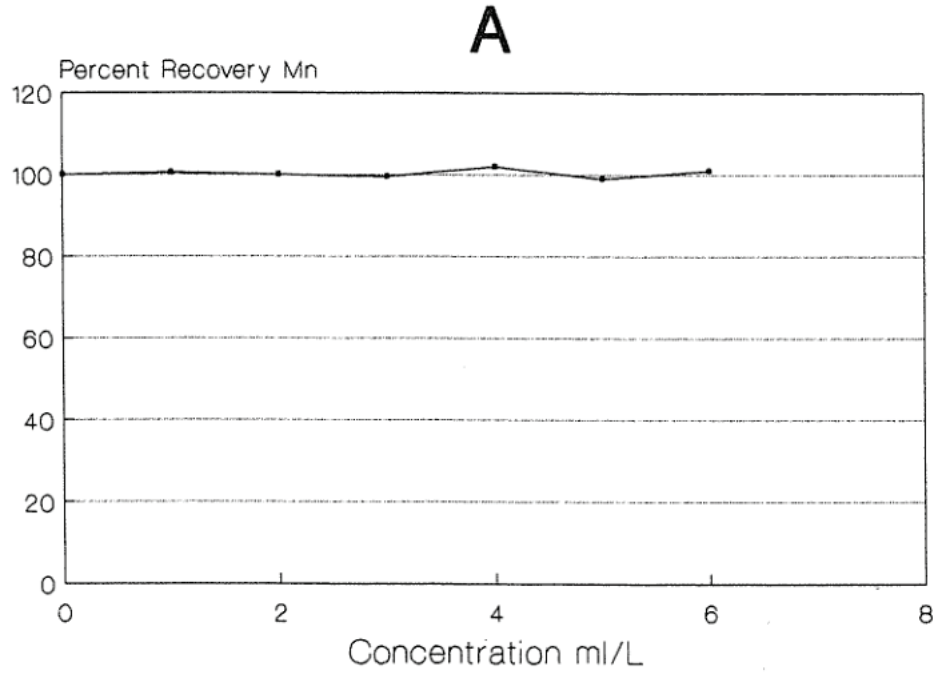


Figure 2: The effect of increasing concentrations of ethanol on the percent recovery of 2.0 mg/L manganese solution

The effect of acetic acid, acetic ester and sucrose on Mn determination was also assessed. Acetic acid and acetic ester are two major components of white spirit[4]. Both chemicals were added over a concentration range of 0–6 mL/Litre to a 2 mg/L aqueous Mn solution. The results graphed in Figure 3 indicated that both compounds did not affect the absorbance result at any concentration. Sucrose may also be added to white spirit, therefore the effect of sucrose on the recovery of 2 ppm Mn was also studied. The results shown in Figure 4 indicated that adding sucrose (0–2% w/v) did not produce any effect, however at concentrations above 2% (2–6% w/v) the absorbance of a 2 mg/L Mn solution decreased with increasing concentration of added sucrose.



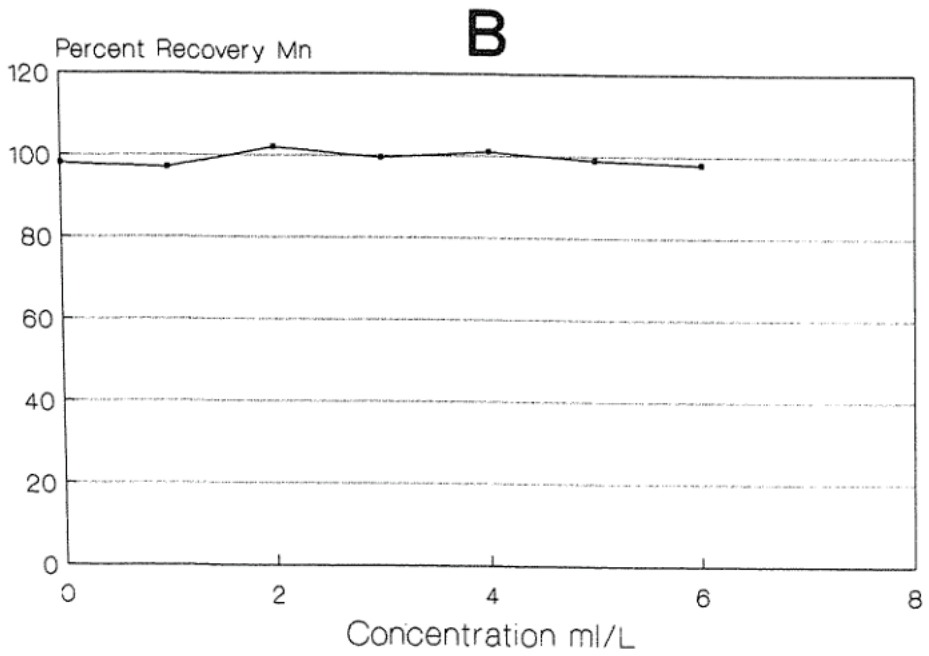


Figure 3: The effect of increasing concentrations of (A) acetic acid and (B) acetic ester on the percent recovery of a 2 mg/L Mn solution

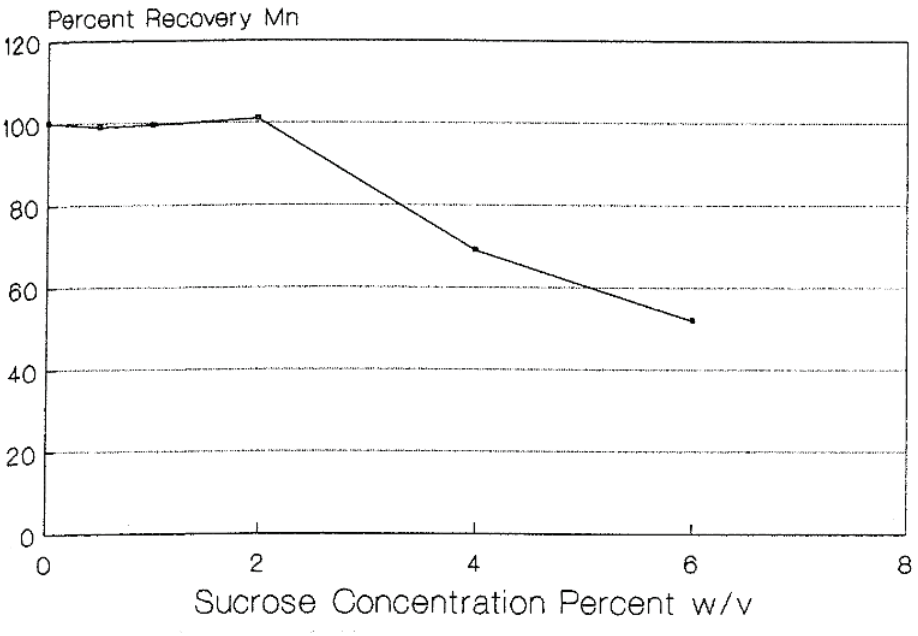


Figure 4: The effect of increasing sucrose concentration on the percent recovery of a 2 mg/L Mn solution

The characteristic concentration of Mn using this method was 0.03 mg/L. Accuracy of the method was determined by adding 3 known amounts of Mn to 5 separate white spirits and calculating percentage recovery. The amount of Mn added was equivalent to 1.0, 2.0 and 3.0 mg/L. The recovery values obtained are listed in Table 2, and show good recovery and accuracy. Interferences by metal ions in the method were also studied. A 1.00 mg/L Mn solution was taken and metal ions were added (Pb, 5.0 mg/L; Hg, 5.0 mg/L; Fe, 10.0 mg/L; Cu, 10.0 mg/L; Zn, 1.0 mg/L; Mg, 10.0 mg/L; Ag, 2.0 mg/L; K, 10.0 mg/L; Na, 10.0 mg/L; As, 5.0 mg/L). The results are shown in Table 3 and indicate that there was no change in the absorbance for 1 mg/L Mn with any added metal ion.

Sample No.	Mn (mg/L)	Added Mn (mg/L)	Total Mn (mg/L)	% Recovery
1	0.20	1.0	1.20	100.3
		2.0	2.26	102.8
		3.0	3.23	101.1
2	0.27	1.0	1.21	93.3
		2.0	2.30	101.4
		3.0	3.18	96.8
3	0.25	1.0	1.28	103.1
		2.0	2.27	101.4
		3.0	3.19	95.7
4	0.21	1.0	1.28	106.8
		2.0	2.34	106.4
		3.0	3.14	97.7
5	0.78	1.0	1.69	90.7
		2.0	2.57	88.2
		3.0	3.32	84.2

Table 2: Percentage recovery values for the addition of 1.0, 2.0 and 3.0 mg/L Mn to 5 different white spirits

Metals Added (mg/L)	Absorbance
None	0.144
Ag, 2.0	0.140
As, 5.0	0.144
Cu, 10.0	0.144
Fe, 10.0	0.149
Hg, 5.0	0.143
K, 10.0	0.142
Mg, 10.0	0.145
Na, 10.0	0.140
Pb, 5.0	0.145
Zn, 10.0	0.142

Table 3: The effect of adding metal ions to a 1 mg/L solution of Mn. The absorbance value is the mean of 7 measurements

A comparison was made between the proposed method and a digestion method for 15 commercial white spirits, whereby a volume of white spirt was dried and then ashed at 425°C for two hours, the cold ash being dissolved in concentrated nitric acid. Using the student's t-test, no significant difference was noted.

Conclusion

This paper has described an accurate method for determining manganese in white spirit by flame atomic absorption spectrophotometry. This method allows simple, direct aspiration of an undiluted sample, and a fast analysis is possible.

References

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