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● SPECTROPHOTOMETRIC ANALYSIS
No.A371

Mn Analysis by Atomic Absorption

■ Introduction

Mn is present in the earth's crust and in sea water at levels of about 0.09% and 0.002mg/L, respectively. Metallic Mn is obtained by reducing manganese ore using Al. The allotropes of Mn include α, β, γ and δ, among which the α and β allotropes are hard and fragile, the γ allotrope are ductile and the δ allotrope is stable only at high temperature. In the field of iron and steel, since manganese binds with sulfur in steel to alter the hardness of steel, it is an essential element in the production of steel withstanding strong impact and wear resistance. Since iron that contains Mn possesses great tensile strength, it is used in such parts as dredging buckets and caterpillars. Quadrivalent manganese dioxide, a typical Mn compound, is used as a desiccant and catalyst in the dye industry and as an iron decolorant as well as colorant in the glass industry. The powerful oxidant septivalent potassium permanganate is used for decoloration of bleach and oils and fats.
Mn which is ingested by humans through food and drinking water is converted to Mn^{2+} by the action of gastric acid in the stomach, and is then oxidized to form Mn^{3+} in the intestinal tract where it enters the blood. In the blood, Mn^{3+}, with characteristics similar to those of Fe^{3+}, combines with the iron transport protein transferring to circulate and be carried to the various internal organs. More than 95% of the manganese is excreted in the bile via the liver. However, Mn acts to promote enzyme activity by combining with proteins and enzymes, playing a role in regulating their catalytic action. In addition, Mn can substitute for Ca and Mg, etc., thereby suppressing the action of these metals. Moreover, Mn has been found to lower blood sugar, and accordingly, is thought to promote activation of the insulin receptor. In general, there are few cases of manganese deficiency diseases, however, it is known that excessive consumption can result in its accumulation in the thyroid gland, causing enlargement of the gland.
We introduce here the relationship between slit width and sensitivity in an analysis of manganese using flame analysis.

Table 1 Basic Data of Mn

Atomic weight	:	54.938	
Melting point	:	1244˚C ($MnCl_2$ 650˚C)	
Boiling point	:	1962˚C ($MnCl_2$ 1190˚C)	
Oxidation number	:	0	$Mn_2(CO)_{10}$
		+1	$[Mn(CN)_6]^-$
		+2	MnO, Mn_3O_4, $MnCl_2$
		+3	Mn_2O_3, MnF_3
		+4	MnO_2, MnF_4
		+5	MnO_4^{3-}
		+6	MnO_4^{2-}
		+7	Mn_2O_7, MnO_4^{2-}
Solubility	:	$MnCl_2$ 73.6g/100g Water (20˚C)	

Table 2 Wavelength of Mn

(nm)	Sensitivity Ratio
279.5	1.0
280.1	0.47
403.1	0.11

■ Flame Analysis of Mn

In setting of the spectrophotometer analytical conditions, it is necessary to select a slit width that is suitable for the measurement element. When measuring an element with many resonant lines near the analysis line, a narrow slit width is usually set to accurately capture the analysis line. Elements with resonant lines in the vicinity of the measurement wavelength include Co, Fe, Mn, Na, and Ni, etc. For example, in the case of Mn, 279.48nm is the analysis line with the highest sensitivity, however, 279.83nm and 280.11nm with low sensitivities are nearby. When a narrow slit width is selected as compared with a wide slit width, the light intensity reaching the detector is weaker, and there is a tendency for increasing baseline noise. The methods used to decrease the noise include increasing the lamp current value, making the slit width wider, etc., however, for elements that have nearby lines, selecting a wide slit width can cause such problems as decreased sensitivity and curved calibration curve. Therefore, it is necessary to carefully consider the objective and measurement accuracy in deciding whether or not to change the slit width.

Fig.1 and Fig.2 show the profile and calibration curve, respectively, obtained in flame analysis of Mn when using the recommended slit width of 0.2nm, while Fig.3 and Fig.4 show these using a slit width of 0.5nm.

Compared to analysis using the 0.2nm slit width, that using the 0.5nm value shows slightly decreased sensitivity, although it is clear that analysis with this slit width is possible.

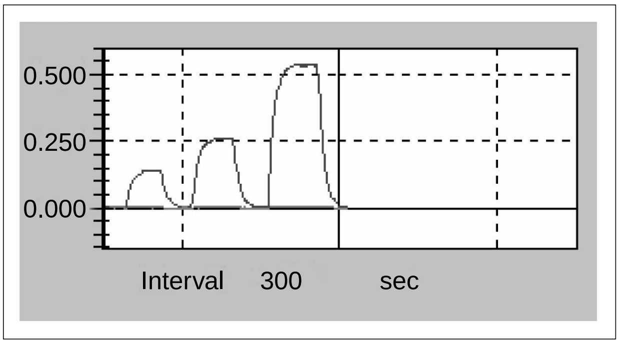

Fig.1 Profile (Slit Width 0.2nm)

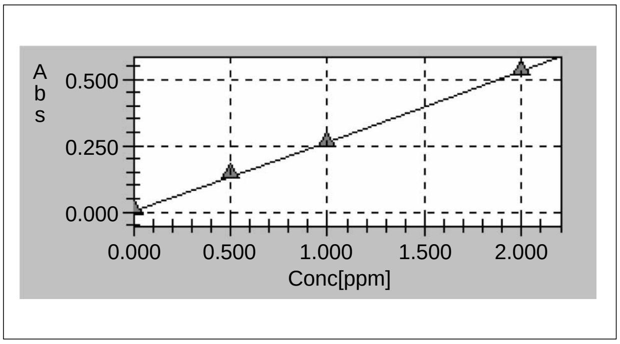

Fig.2 Calibration Curve (Slit Width 0.2nm)

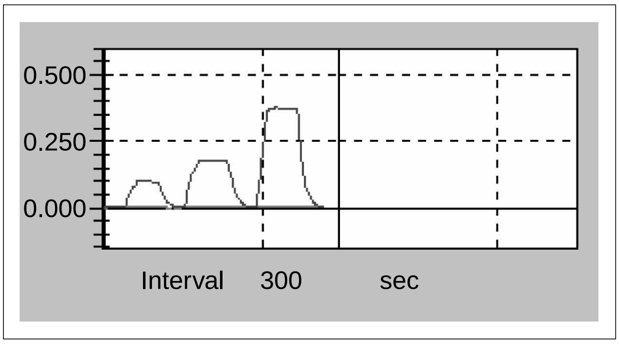

Fig.3 Profile (Slit Width 0.5nm)

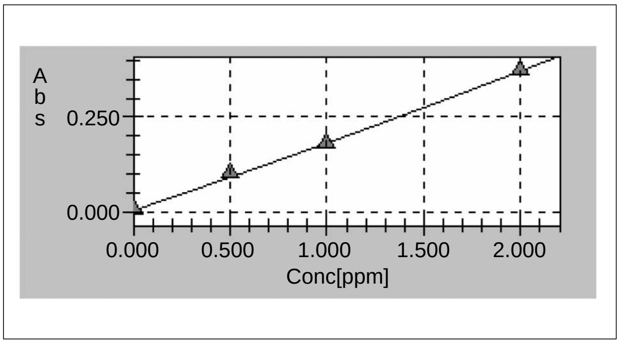

Fig.4 Calibration Curve (Slit Width 0.5nm)

■ Conclusion

A benefit of widening the slit width, aside from decreasing the noise, is improved background correction accuracy when the inherent wavelengths of coexisting components in the sample are near the analysis line of the measurement element. For example, in trace analysis of Mg in the sample which contains Na at a high concentration, the phenomenon of inaccurate background correction is seen when using the D2 method, since the Na wavelength is near the Mg analysis line. In this case, the slit width is widened with the aim of reducing the influence of the nearby Na line and improving background accuracy.

In atomic absorption analysis, matrix modifiers are widely used to suppress chemical interference, however, this technique is often used especially in furnace measurement, where interference is common. For example, magnesium nitrate is used as a Mn modifier.

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