SHIMADZU APPLICATION NEWS

LAAN-A-AA-E009

● SPECTROPHOTOMETRIC ANALYSIS

No.A372

Determination of Zn in River Water

■ Introduction

In Notification No. 123 from Japanese Ministry of Environment (November 5, 2003), part of the environmental standards related to water pollution was amended. The amendment establishes total zinc as an additional environmental standard for maintenance of the environment, in particular, for aquatic life and its habitat and maintenance of a growth environment in public water regions.
The standard is applicable to specified water regions.
The criteria value differs depending on the type of water body, with 0.03mg/L or less for rivers and lakes, and 0.02 or 0.01mg/L or less for coastal waters.
For the applicable analytical method, the industrial wastewater test method (JIS K0102) is used, including the following methods.

1. Flame atomic absorption method
2. Furnace atomic absorption method
3. ICP emission spectrometric method
4. ICP mass spectrometric method

The present study examines the determination of zinc in river water standard samples JAC 0031 (non-spiked) and JAC 0032 (spiked), provided by the Japan Society for Analytical Chemistry, using flame atomic absorption (hereafter, flame method) and furnace atomic absorption (hereafter, furnace method).

■ Instrument and Analytical Conditions

Tables 1 and 2 show the instrument and analytical conditions used with each method, respectively. In the furnace method, analysis sensitivities at low concentration and high concentration was changed by changing the flow rate of the argon gas passing through the graphite tube during atomization.

Table 1 Analytical Conditions in Flame AA

Instrument	AA-6300
Analysis wavelength	213.8nm
Slit width	0.7nm
Current	10mA
Lamp mode	BGC-D2
Flame type	Air – acetylene
Burner height	7mm
Std. Sol. Conc.	Upper limit 0.04mg/L (ppm)

Table 2 Analytical Conditions in Furnace AA

Instrument	Main unit AA-6800, Atomization unit GFA-EX7
Analysis wavelength	213.8nm
Slit width	0.5nm
Current	10mA
Lamp mode	BGC-D2
Tube type	High density graphite tube
Smpl. Inj. Vol.	2 – 20μL (total injection volume: 20μL)
Temp. Program	Drying 120˚C Ashing 400˚C Atomization 2000˚C Cleaning 2500˚C
Std. Sol. Conc.	Low concentration: Upper limit 1.5μg/L (ppb) High concentration: Upper limit 15μg/L (ppb)

■ Results

The methods used for adjusting the sensitivity in atomic absorption analysis include changing the analysis wavelength, and in the flame method, adjusting the burner angle. However, in the furnace method, sensitivity adjustment is possible by adjusting the argon gas flow rate during atomization. Fig.1 shows the relation between the gas flow rate and the absorbance of zinc at 2ppb.
Fig.2 displays the calibration curve of the flame method. Fig.3 shows the low concentration range calibration curve of the furnace method in which the argon gas flow rate was 0.0L/min. Fig.4 shows the high concentration range calibration curve of the furnace method in which the argon gas flow rate was 0.5L/min.

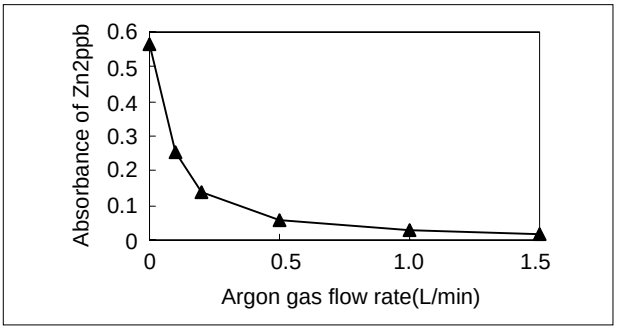

Fig.1 Relation between Ar Flow Rate and Sensitivity

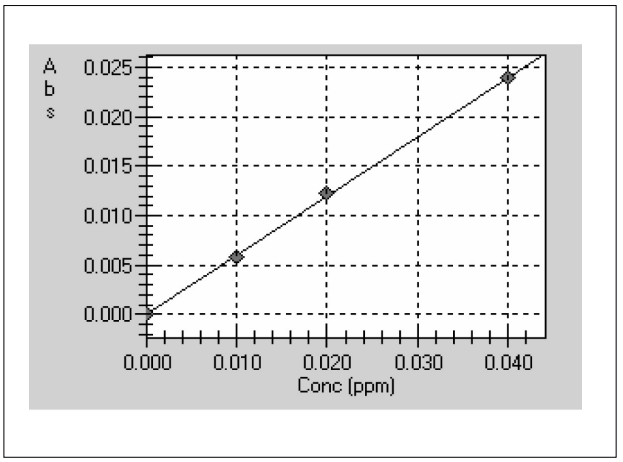

Fig.2 Calibration Curve in Flame Method

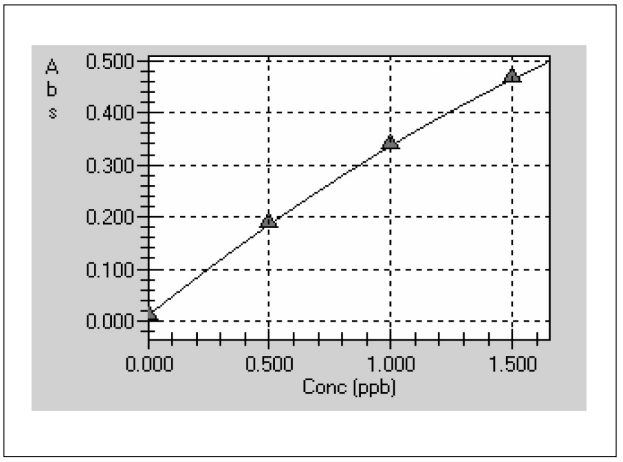

Fig.3 Calibration Curve in Furnace Method at Low Concentrations

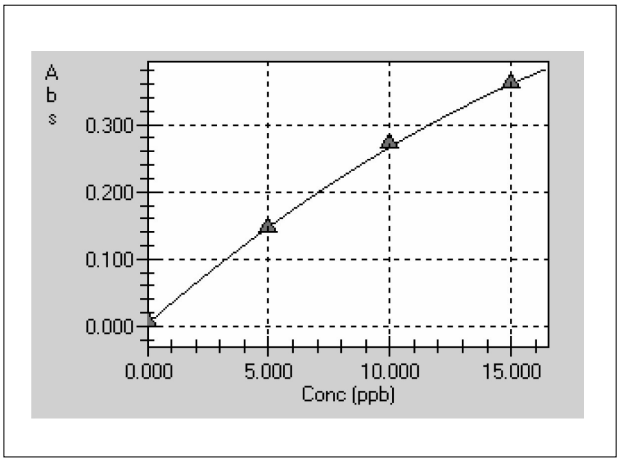

Fig.4 Calibration Curve in Furnace Method at High Concentrations

Fig.5 shows the peak profiles of the standard solutions at low concentrations in the furnace method.

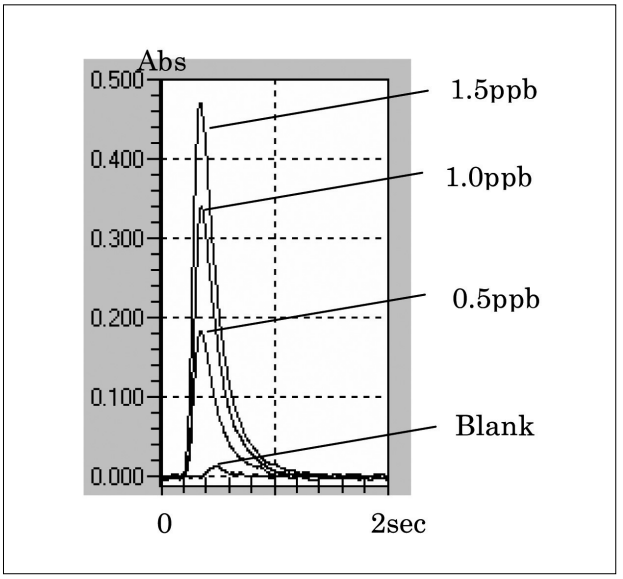

Fig.5 Peak Profiles in Furnace Method

Table 3 presents the results of analysis. Table 4 shows the expected lower quantification limits in the test solution in the flame method and furnace method, respectively, based on the results of the analysis. The lower quantification limit in the furnace method was predicted based on the analysis results in the low concentration range.

Table 3 Analysis Results

	Certified value	Flame AA	Furnace AA (Low Conc.)	Furnace AA (High Conc.)
JAC 0031 (non-spiked)	0.00079 (mg/L)	<0.007	0.00079	0.00074
JAC 0032 (spiked)	0.0113 (mg/L)	0.011	0.0105	0.0106

JAC 0032 was analyzed at low concentrations using the furnace method, with the injection volume decreased to 2µL.

Table 4 Quantification Limits

Flame Method	0.007mg/L
Furnace Method	0.0001mg/L

In the flame method, since direct quantification of zinc below 0.01mg/L is difficult, concentration processing is necessary. On the other hand, in the furnace method, quantification is possible down to 0.0001mg/L, 1/100 of the standard value, without boosting the concentration. However, with samples like seawater that have high salt concentrations, it is necessary to be careful of spectral and chemical interferences. In addition, with zinc concentrations lower than 0.01mg/L, it is necessary to take adequate precautions against the contamination from the containers or the acid used during processing.

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SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

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