Zinc, atomic absorption spectrometric, direct

Parameters and Codes:
Zinc, dissolved, I-1900-85 (µg/L as Zn): 01090
Zinc, total recoverable, I-3900-85 (µg/L as Zn): 01092
Zinc, suspended recoverable, 1-7900-85 (µg/L as Zn): 01091
Zinc, recoverable-from-bottom-material, dry wt, I-5900-85 (µg/g as Zn): 01093

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing from 10 to 500 µg/L of zinc. Sample solutions containing more than 500 µg/L need to be diluted or to be read on a less expanded scale.

1.2 Suspended-recoverable zinc is calculated by subtracting dissolved zinc from total recoverable zinc.

1.3 This method may be used to analyze bottom material containing at least 1.0 µg/g of zinc. Sample solutions containing more than 500 µg/L of zinc need to be diluted or less scale expansion used.

1.4 Total recoverable zinc in water-suspended sediment needs to undergo preliminary digestion-solubilization by method I-3485, and recoverable zinc in bottom material needs to undergo preliminary digestion-solubilization by method I-5485 before being determined.

2. Summary of method

2.1 Zinc is determined by atomic absorption spectrometry by direct aspiration of the sample into an air-acetylene flame.

2.2 The procedure may be automated by the addition of a sampler and either a strip-chart recorder or a printer or both.

3. Interferences

3.1 Magnesium at concentrations greater than 100 mg/L interferes unless other cations, such as sodium, are present in the sample.

3.2 Individual concentrations of sodium, potassium, sulfate, chloride (9,000 mg/L of each), calcium (4,500 mg/L), nitrate (2,000 mg/L), iron (4 X 10^6 µg/L), and cadmium, nickel, copper, lead, cobalt, and chromium (10,000 µg/L each) do not interfere. Greater concentrations of each constituent were not investigated.

3.3 Samples containing 100 mg/L of silica cause no interference; however, zinc recovery is approx. 10 percent low in samples containing 200 mg/L of silica.

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating	Ultraviolet
Wavelength	213.8 nm
Source (hollow-cathode lamp)	Zinc
Oxidant	Air
Fuel	Acetylene
Type of flame	Oxidizing

4.3 The 50-mm (2-in.) and 100-mm (4-in.) flathead, single-slot burners allow a working range from 10 to 500 µg/L of zinc. Different burners may be used according to manufacturer's instructions.

5. Reagents

5.1 *Zinc standard solution I,* 1.00 mL = 100 µg Zn: Dissolve 0.100 g reagent grade zinc (30-mesh) in a slight excess of concentrated HCl (sp gr 1.19), and dilute to 1,000 mL with demineralized water.

5.2 *Zinc standard solution II,* 1.00 mL = 1.0 µg Zn: Dilute 10.0 mL zinc standard solution I to 1,000 mL with demineralized water containing 1 mL concentrated HNO_3 (sp gr 1.41).

5.3 *Zinc working standards*: Prepare a series of at least six working standards containing from 10 to 500 µg/L of zinc by appropriate dilutions of zinc standard solution II with acidified water.

5.4 *Water, acidified.* Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to 1 L of demineralized water.

6. Procedure

Aspirate the blank (acidified water) to set the automatic zero control. Use the automatic concentration control to set the concentrations of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

7. Calculations

7.1 Determine the micrograms per liter of dissolved or total recoverable zinc in each sample from the digital display or printer output while aspirating each sample. Dilute those samples whose zinc concentrations exceed the working range of the method and multiply by the proper dilution factors.

7.2 To determine micrograms per liter of suspended recoverable zinc, subtract dissolved-zinc concentration from total-recoverable-zinc concentration.

7.3 To determine micrograms per gram of zinc in bottom-material samples, first determine the micrograms per liter of zinc as in paragraph 7.1, then

$$\text{Zn (µg/g)} = \frac{\text{µg/L of Zn} \times \frac{\text{mL of original digest}}{1{,}000}}{\text{wt of sample (g)}}$$

8. Report

8.1 Report zinc, dissolved (01090), total-recoverable (01092), and suspended-recoverable (01091), concentrations as follows: less than 100 µg/L, nearest 10 µg/L; 100 µg/L and above, two significant figures.

8.2 Report zinc, recoverable-from-bottom-material (01093), concentrations as follows: less than 10 µg/g, nearest microgram per gram; 10 µg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved zinc for 30 samples within the range of 14 to 1110 µg/L may be expressed as follows:

$$S_T = 0.070X + 6.51$$

where

S_T = overall precision, micrograms per liter, and

X = concentration of zinc, micrograms per liter.

The correlation coefficient is 0.7967.

9.2 Precision for dissolved zinc for six of the 30 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
33	14	43
47	104	10
19	116	22
41	236	7
22	520	5
17	1110	9

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable zinc and recoverable zinc in bottom material will be greater than that reported for dissolved zinc.

9.4 Precision for total recoverable zinc expressed in terms of the percent relative standard deviation for two water-suspended sediment mixtures is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
23	78.4	27
26	172	31

Reference

Fishman, M.J., and Downs, S.C., 1966, Methods for analysis of selected metals in water by atomic absorption: United States Geological Survey Water-Supply Paper 1540-C., p. 43-5.