Copper, atomic absorption spectrometric, direct

Parameters and Codes:
Copper, dissolved, I-1270-85 (µg/L as Cu): 01040
Copper, total recoverable, I-3270-85 (µg/L as Cu): 01042
Copper, suspended recoverable, I-7270-85 (µg/L as Cu): 01041
Copper, recoverable-from-bottom-material, dry wt, I-5270-85 (µg/g as Cu): 01043

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 10 µg/L of copper. Sample solutions containing more than 1,000 µg/L need either to be diluted or to be read on a less expanded scale. Brines need to be analyzed by the atomic absorption spectrometric chelation-extraction method, providing that the interference limits discussed in that method are not exceeded.

1.2 Suspended recoverable copper is calculated by subtracting dissolved copper from total recoverable copper.

1.3 This method may be used to analyze bottom material containing at least 1 µg/g of copper. Prepared sample solutions containing more than 1,000 µg/L need either to be diluted or to be read on a less expanded scale.

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

2. Summary of method

2.1 Copper is determined by atomic absorption spectrometry by direct aspiration of the sample solution into an air-acetylene flame (Fishman and Downs, 1966).

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

Individual concentrations of sodium (9,000 mg/L), potassium (9,000 mg/L), calcium (4,000 mg/L), magnesium (4,000 mg/L), sulfate (9,000 mg/L), chloride (9,000 mg/L), nitrate (2,000 mg/L), iron (4 X 10^6µg/L), lead, cadmium, zinc, and chromium (10,000 µg/L each) do not interfere. Higher concentrations of each constituent were not investigated. Nickel and cobalt concentrations greater than 8,000 µg/L suppress the copper absorption.

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 -------------------- 324.7 nn
Source (hollow-cathode
                lamp) ----------- Copper
Oxidant ------------------------- Air
Fuel ----------------------------- Acetylene
Type of flame ------------------ Oxidizing

4.3 The 100-mm (4-in.), flathead, single-slot burner allows a working range of 10 to 1,000 µg/L. Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Copper standard solution I,* 1.00 mL= 1000 µg Cu: Dissolve 1.252 g CuO in a minimum amount of dilute HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 (sp gr 1.41) and dilute to 1000 mL with demineralized water.

5.2 *Copper standard solution II,* 1.00 mL= 5.00 µg Cu: Dilute 5.0 mL copper standard solution I and 1 mL concentrated HNO_3 (sp gr

1.41) to 1,000 mL with demineralized water. This standard is used to prepare working standards at time of analysis.

5.3 *Copper working standards:* Prepare a series of at least six working standards containing from 10 to 1,000 µg/L of copper by appropriate dilution of copper standard solution II with acidified water. Prepare fresh daily.

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 copper in each sample solution from the digital display or printer while aspirating each sample. Dilute those samples containing copper concentrations that exceed the working range of the method and multiply by proper dilution factors.

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

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

$$Cu\ (\mu g/g) = \frac{\mu g/L\ Cu \times \dfrac{mL\ of\ original\ digest}{1,000}}{wt\ of\ sample\ (g)}$$

8. Report

8.1 Report copper, dissolved (01040), total-recoverable (01042), and suspended-recoverable (01041), concentrations as follows:

less than 100 µg/L, nearest 10 µg/L; 100 µg/L and above, two significant figures.

8.2 Report copper, recoverable-from-bottom-material (01043), 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 copper for 28 samples within the range of 10 to 595 µg/L may be expressed as follows:

$$S_T = 0.057X + 7.13$$

Where

S_T = overall precision, micrograms per liter, and

X = concentration of copper, micrograms per liter.

The correlation coefficient is 0.8007.

9.2 Precision for dissolved copper for five of the 28 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
9	10.0	90
30	60.3	14
22	100	16
28	245	11
23	595	9

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

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

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
17	21.1	39
23	131	16

References

Fishman, M. J., and Downs, S. C., 1966, Methods for analysis of selected metals in water by atomic absorption: U.S. Geological Survey Water-Supply Paper 1540-C, p. 28-30.