Barium, atomic absorption spectrometric, direct

Parameters and Codes:
Barium, dissolved, I-1084-85 (µg/L as Ba): 01005
Barium, total recoverable, I-3084-85 (µg/L as Ba): 01007
Barium, suspended recoverable, I-7084-85 (µg/L as Ba): 01006
Barium, recoverable-from-bottom-material, dry wt, I-5084-85 (µg/g as Ba): 01008

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 100 µg/L of barium. Sample solutions containing more than 5000 µg/L need to be diluted.

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

1.3 This method may be used to analyze bottom material containing at least 5 µg/g of barium.

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

2. Summary of method

Barium is determined by atomic absorption spectrometry. Sodium chloride is added to control ionization of barium in the flame.

3. Interferences

The use of a nitrous oxide-acetylene flame virtually eliminates chemical interferences in the determination of barium. However, barium is easily ionized in the nitrous oxide-acetylene flame; to control this effect, sodium chloride solution must be added to each sample and standard.

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 ---------- Visible
Wavelength ---------- 553.6 nm
Source (hollow-cathode lamp) ---------- Barium
Burner ---------- Nitrous oxide
Oxidant ---------- Nitrous oxide
Fuel ---------- Acetylene
Type of flame ---------- Fuel-rich

5. Reagents

5.1 *Barium standard solution,* 1.00 mL= 100 µg Ba: Dissolve 0.1516 g $BaCl_2 \cdot 2H_2O$, dried at 180°C for 1 h, in demineralized water and dilute to 1,000 mL.

5.2 *Barium working standards:* Prepare a series of at least six working standards containing from 100 to 5,000 µg/L of barium by appropriate dilution of barium standard solution. Add 1.00 mL NaCl solution for each 10 mL of working standard. Similarly prepare a demineralized water blank. Prepare fresh daily.

5.3 *Sodium chloride solution,* 25.4 g/L: Dissolve 25.4 g NaCl in demineralized water and dilute to 1 L.

6. Procedure

6.1 Add 1.0 mL NaCl solution to 10.0 mL sample solution and mix thoroughly.

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

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

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

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

8. Report

8.1 Report barium, dissolved (01005), total-recoverable (01007), and suspended-recoverable (01006), concentrations as follows: less than 1,000 µg/L, nearest 100 µg/L; 1,000 µg/L and above, two significant figures.

8.2 Report barium, recoverable- from- bottom-material (01008), concentration as follows: less than 100 µg/g, nearest 10 µg/g; 100 µg/g and above, two significant figures.

9. Precision

9.1 The standard deviation for dissolved barium within the range of 43 to 800 µg/L for 17 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 93.5 µg/L ranged from 85.3 to 103.2 µg/L.

9.2 Precision for dissolved barium for five of the 17 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
13	43	70
17	112	53
18	294	32
9	756	7
6	800	0

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

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

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
12	91.7	86
10	172	40