Beryllium, atomic absorption spectrometric, direct

Parameters and Codes:
Beryllium, dissolved, I-1095-85 (µg/L as Be): 01010
Beryllium, total recoverable, I-3095-85 (µg/L as Be): 01012
Beryllium, suspended recoverable, I-7095-85 (µg/L as Be): 01011
Beryllium, recoverable-from-bottom-material, dry wt, I-5095-85 (µg/g as Be): 01013

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 10 µg/L of beryllium. Sample solutions containing more than 200 µg/L need to be diluted.

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

1.3 This method may be used to analyze bottom material containing at least 0.5 µg/g of beryllium.

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

2. Summary of method

Beryllium is determined by atomic absorption spectrometry. Calcium chloride is added to control ionization of beryllium in the flame.

3. Interferences

3.1 Beryllium is slightly ionized in the nitrous oxide-acetylene flame; to control this effect, calcium chloride solution must be added to each standard and sample.

3.2 Bicarbonate ion interferes; however, this interference is of no consequence if samples preserved by the addition of acid are used for the analysis.

3.3 Aluminum at concentrations greater than 500 µg/L has been reported to depress the beryllium absorbance.

3.4 Sodium and silicon at concentrations in excess of 1000 mg/L have been reported to severely depress the beryllium absorbance (Environmental Protection Agency, 1979).

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 -------------------- 234.9 nm
Source (hollow-cathode lamp) -------------------- Beryllium
Burner ------------------------ Nitrous oxide
Oxidant ----------------------- Nitrous oxide
Fuel --------------------------- Acetylene
Type of flame --------------- Fuel-rich

5. Reagents

CAUTION: Beryllium compounds and samples with high concentrations of beryllium should be handled with care; they may be carcinogenic.

5.1 *Beryllium standard solution I,* 1.00 mL= 100 µg Be: Dissolve 0.1000 g Be flakes in a minimum of aqua regia. Heat to increase rate of dissolution. Add 10.0 mL of concentrated HNO_3 (sp gr 1.41) and dilute to 1000 mL with demineralized water.

5.2 *Beryllium standard solution II,* 1.00 mL = 10.0 µg Be: Dilute 100.0 mL beryllium standard solution I to 1,000 mL with demineralized water.

5.3 *Beryllium working standards:* Prepare a series of at least six working standards containing from 10 to 200 µg/L beryllium by appropriate dilution of beryllium standard solution II. Add 1.0 mL $CaCl_2$ solution for each 10.0 mL of working standard. Similarly prepare a demineralized water blank. Prepare fresh daily.

5.4 *Calcium chloride solution,* 27.8 g/L: Suspend 25 g $CaCO_3$ in demineralized water and dissolve with a minimun of dilute HCl. Dilute to 1 L with demineralized water.

6. Procedure

6.1 Add 1.0 mL $CaCl_2$ 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 beryllium in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing concentrations of beryllium 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 beryllium, subtract dissolved-beryllium concentration from total-recoverable-beryllium concentration.

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

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

8. Report

8.1 Report beryllium, dissolved (01010), total-recoverable (01012), and suspended-recoverable (01011), concentrations as follows: less than 1,000 µg/L to the nearest 10 µg/L.

8.2 Report beryllium, recoverable-from-bottom-material (01013), as follows: less than 10 µg/g, nearest microgram per gram; 10 µg/g and above, two significant figures.

9. Precision

9.1 The standard deviation for dissolved beryllium within the range of 5.4 to 70 µg/L for 17 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 4.11 µg/L ranged from 3.6 to 4.9 µg/L.

9.2 Precision for dissolved beryllium for four 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)
8	5.4	54
8	20.8	19
11	47.7	9
3	70.0	0

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

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

Number of Laboratories	Mean (µg/L)	Relative standard deviation (percent)
4	22.5	27
5	64.2	7

Reference

U.S. Environmental Protection Agency, 1979, Methods for chemical analysis of water and wastes, Cincinnati, p. 210.1-1.