Cadmium, atomic absorption spectrometric, direct

Parameters and Codes:
Cadmium, dissolved, I-1135-85 (µg/L as Cd): 01025
Cadmium, total recoverable, I-3135-85 (µg/L as Cd): 01027
Cadmium, suspended recoverable, I-7135-85 (µg/L as Cd): 01026
Cadmium, recoverable-from-bottom-material, I-5135-85 (µg/g as Cd): 01028

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 10 µg/L of cadmium. Sample solutions containing more than 250 µg/L need either to be diluted or to be read on a less expanded scale. Sample solutions containing less than 10 µg/L 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 cadmium is calculated by subtracting dissolved cadmium from total recoverable cadmium.

1.3 This method may be used to analyze bottom material containing at least 1 µg/g of cadmium.

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

2. Summary of method

Cadmium is determined by atomic absorption spectrometry by direct aspiration of the sample into an air-acetylene flame without precon-centration or pretreatment of the sample.

3. Interferences

3.1 Individual concentrations of sodium (9,000 mg/L), potassium (9,000 mg/L), magnesium (4,500 mg/L), sulfate (9,000 mg/L), chloride (9,000 mg/L), nitrate (100 mg/L), iron (4×10^6 µg/L), and cobalt, nickel, copper, zinc, lead, and chromium (10,000 µg/L each) do not interfere. Greater concentrations of each constituent were not investigated.

3.2 Calcium at concentrations greater than 1,000 mg/L suppresses the cadmium absorption. At 2,000 mg per liter of calcium, the suppression is approximately 19 percent.

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 ---------- 228.8 nm
Source (hollow-cathode lamp) ---------- Cadmium
Oxidant ---------- Air
Fuel ---------- Acetylene
Type of flame ---------- Oxidizing

4.3 The Perkin-Elmer, flathead, single-slot burner allows a working range of 10 to 250 µg/L. Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Cadmium standard solution I,* 1.00 mL= 100 µg Cd: Dissolve 0.1000 g Cd splatters in a minimun of dilute HNO_3. Heat to increase rate of dissolution. Add 10 mL concentrated HNO_3 (sp gr 1.41) and dilute to 1,000 mL with demin-eralized water.

5.2 *Cadmium standard solution II,* 1.00 mL= 1.0 µg Cd: Dilute 10.0 mL cadmium standard solution I and 1 mL concentrated HNO_3 (SP gr 1.41) to 1,000 mL with demineralized water.

5.3 *Cadmium working standards:* Prepare a series of at least six working standards containing from 10 to 250 µg/L of cadmium by appropriate dilution of cadmium standard solutions I and II with acidified water. Prepare fresh daily.

5.4 *Water, acidified:* Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to a liter 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 cadmium in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing cadmium concentrations that exceed the working range of the method and multiply by the proper dilution factors.

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

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

$$\text{Cd (µg/g)} = \frac{\text{µg/L Cd} \times \dfrac{\text{mL of original Digest}}{1{,}000}}{\text{wt of Sample (g)}}$$

8. Report

8.1 Report cadmium, dissolved (01025), total-recoverable (01027), and suspended-recoverable (01026), concentrations as follows: less than 1,000 µ/L, nearest 10 µg/L; 1,000 µg/L and above, two significant figures.

8.2 Report cadmium, recoverable-from-bottom-material (01028), concentrations 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 cadmium within the range of 2.8 to 18.4 µg/L for 21 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 3.57 µg/L ranged from 3.24 to 3.95 µg/L.

9.2 Precision for dissolved cadmium for five of the 21 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
12	2.8	124
17	4.8	50
15	10.7	19
15	15.7	24
5	18.4	71

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

9.4 Precision for total recoverable cadmium 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)
13	5.4	43
9	15.8	25