Cobalt, atomic absorption spectrometric, direct

Parameters and Codes:
Cobalt, dissolved, I-1239-85 (µg/L as Co): 01035
Cobalt, total recoverable, I-3239-85 (µg/L as Co): 01037
Cobalt, suspended recoverable, I-7239-85 (µg/L as Co): 01036
Cobalt, recoverable-from-bottom-material, dry wt, I-5239-85, (µg/g as Co): 01038

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 50 µg/L of cobalt. Samples solutions containing more than 1,000 µg/L need either to be diluted or to be read on a less expanded scale. Sample solutions containing less than 50 µg/L need to be analyzed by the atomic absorption spectrometric chelation-extraction method, providing the interference limits discussed in that method are not exceeded.

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

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

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

2. Summary of method

Cobalt is determined by atomic absorption spectrometry by direct aspiration of the sample into an air-acetylene flame without additional treatment of the sample other than the addition of ammonium chloride to mask certain interferences.

3. Interferences

3.1 Nitrate at 1 mg/L interferes by suppressing the absorption of the cobalt. This interference is eliminated in solutions containing about 18,000 mg/L of ammonium chloride. Samples adjusted to this concentration of ammonium chloride show no interference from 800 mg/L of nitrate.

3.2 Individual concentrations of sodium (9,000 mg/L), potassium (9,000 mg/L), calcium (4,500 mg/L), magnesium (4,500 mg/L), sulfate (9,000 mg/L), chloride (15,000 mg/L), iron (4 x 10^6 µg/L), and cadmium, nickel, copper, zinc, lead, and chromium (10,000 µg/L) do not interfere. Higher concentrations of each constituent were not investigated.

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

4.3 The Perkin-Elmer, flathead, single-slot burner allows a working range of 50 to 1,000 µg/L. Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Ammonium chloride solution,* 200 g/L: Dissolve 200 g NH_4Cl in demineralized water and dilute to 1 L with demineralized water.

5.2 *Cobalt standard solution I,* 1.00 mL= 100 µg Co: Dissolve 0.1407 g Co_2O_3 in a minimum amount of dilute HNO_3. Add 10 mL concentrated HNO_3 (sp gr 1.41) and dilute to 1000 mL with demineralized water.

5.3 *Cobalt standard solution II,* 1.00 mL= 10 µg Co: Dilute 100.0 mL cobalt standard solution I and 1 mL concentrated HNO_3 (sp gr 1.41) to 1,000 mL with demineralized water.

5.4 *Cobalt standard working solutions:* Prepare a series of at least six standard working solutions containing from 50 to 1,000 µg/L cobalt by appropriate dilution of cobalt standard solution II with acidified water. Add 1.0 mL NH_4Cl solution for each 10 mL standard working solution. Similarly, prepare an acidified water blank. Prepare fresh daily.

5.5 *Water, acidified*: Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to a liter of demineralized water.

6. Procedure

6.1 Add 1.0 mL NH_4Cl 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 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 cobalt in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing cobalt 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 cobalt, subtract dissolved-cobalt concentration from total-recoverable-cobalt concentration.

7.3 To determine micrograms per gram of cobalt in bottom-material samples, first

determine the micrograms per liter of cobalt in each sample as in paragraph 7.1; then

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

8. Report

8.1 Report cobalt, dissolved (01035), total-recoverable (01037), and suspended-recoverable (01036), concentrations to the nearest 50 µg/L.

8.2 Report cobalt, recoverable-from-bottom-material (01038), concentrations as follows: 50 to 100 µg/g, the nearest 10 µg/g; 100 µg/g and above, two significant figures.

9. Precision

9.1 The standard deviation for dissolved cobalt within the range of 6.2 to 19.3 µg/L (NOTE 1) for six samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 4.4 µg/L ranged from 3.4 to 6.4 µg/L.

NOTE 1. Precision data for cobalt are below the reporting level of 50 µg/L. Samples that contained greater cobalt concentrations were not available; however, precision should improve at greater concentrations.

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

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
5	6.2	57
4	15.8	27
3	19.3	11

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