Aluminum, atomic absorption spectrometric, direct

Parameters and Codes:

Aluminum, dissolved, I-1051-85, (μg/L as Al): 01106
Aluminum, total recoverable, I-3051-85 (μg/L as Al): 01105
Aluminum, suspended recoverable I-7051-85 (μg/L as Al): 01107
Aluminum, recoverable-from-bottom-material, dry wt, I-5051-85 (μg/g as Al): 01108

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 100 μg/L of aluminum. Sample solutions containing more than 5,000 μg/L need either to be diluted or to be read on a less expanded scale. Sample solutions containing less than 100 μg/L and brines need to be analyzed by the atomic absorption spectrometric chelation-extraction method, provided that the interference limits discussed in that method are not exceeded.

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

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

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

2. Summary of method

Aluminum is determined by atomic absorption spectrometry by direct aspiration of the sample into a nitrous oxide-acetylene flame without preconcentration or pretreatment of the sample other than the addition of sodium chloride to control ionization of aluminum, and bis(2-ethoxyethyl)ether to enhance the analytical sensitivity (Ramakrishna and others, 1967).

3. Interferences

3.1 Aluminum ionizes slightly in the nitrous oxide-acetylene flame; to control this effect, adjust the sodium-ion concentration of each standard and sample to at least 850 mg/L.

3.2 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 (9,000 mg/L), and iron (9×10^6 μg/L) do not interfere. Greater 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 --------	309.3 nm
Source (hollow-cathode lamp) ----	Aluminum
Burner ------------	Nitrous oxide
Oxidant -----------	Nitrous oxide
Fuel --------------	Acetylene
Type of flame ------	Fuel-rich

5. Reagents

5.1 *Aluminum standard solution*, 1.00 mL= 100 μg Al: Dissolve 0.100 g Al powder in a minimum of 6*M* HCl. Heat to increase rate of dissolution. Add 10.0 mL 6*M* HCL and dilute to 1,000 mL with demineralized water.

5.2 *Aluminum working standards*, Prepare at least six working standards containing from 100 to 5,000 μg/L of Al by appropriate dilution of aluminum standard solution. Add 1.0 mL NaCl solution and 1.0 mL bis(2-ethoxyethyl)-ether for each 10 mL of working standard. Prepare fresh daily.

5.3 *Bis(2-ethoxyethyl)ether*: Eastman Kodak Co. Chemical No. 4738 or equivalent.

5.4 *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 and 1.0 mL bis(2-ethoxyethyl)ether to 10.0 mL of 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 aluminum in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing concentrations of aluminum 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 aluminum, subtract dissolved-aluminum concentration from total-recoverable-aluminum concentration.

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

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

8. Report

8.1 Report aluminum, dissolved (01106), total-recoverable (01105), and suspended-recoverable (01107), concentrations as follows: less than 10,000 µg/L, nearest 100 µg/L; 10,000 µg/L and above, two significant figures.

8.2 Report aluminum, recoverable from bottom material (01108), concentrations as follows: less than 1,000 µg/g, nearest 10 µg/g; 1,000 µg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved aluminum for 17 samples within the range of 40 to 811 µg/L, may be expressed as follows:

$$S_T = 0.253X + 9.84$$

where

S_T= overall precision, micrograms per liter, and

X= concentration of aluminum, micrograms per liter.

The correlation coefficient is 0.7779.

9.2 Precision for dissolved aluminum for six 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)
7	40	135
7	40	85
5	138	79
8	144	31
15	513	19
7	811	9

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

9.4 Precision for total recoverable aluminum 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)
12	4450	13
7	5530	33

References

Ramakrishna, T. V., West, P. W., and Robinson, J. W., 1967, The determination of aluminum and beryllium by atomic absorption spectroscopy: Analytica Chimica Acta, v. 39, p. 81-87.