Magnesium, atomic absorption spectrometric, direct

Parameters and Codes:
Magnesium, dissolved, I-1447-85 (mg/L as Mg): 00925
Magnesium, total recoverable, I-3447-85 (mg/L as Mg): none assigned
Magnesium, suspended recoverable, I-7447-85 (mg/L as Mg): 00926
Magnesium, recoverable-from-bottom-material, dry wt, I-5447-85 (mg/kg as Mg): 00924

1. Application

1.1 This method maybe used to analyze atmospheric precipitation, water, brines, and water-suspended sediment.

1.2 Two analytical ranges for magnesium are included: from 0.01 to 5.0 mg/L and from 2.5 to 50 mg/L. Sample solutions containing magnesium concentrations greater than 50 mg/L need to be diluted.

1.3 Suspended recoverable magnesium is calculated by subtracting dissolved magnesium from total recoverable magnesium.

1.4 This method may be used to analyze bottom material containing at least 10 mg/kg of magnesium. Prepared sample solutions containing more than 50 mg/L to be diluted.

1.5 Total recoverable magnesium in water-suspended sediment needs to undergo preliminary digestion-solubilization by method I-3485, and recoverable magnesium in bottom material needs to undergo preliminary digestion-solubilization by method 1-5485 before being determined.

2. Summary of method

2.1 Magnesium is determined by atomic absorption spectrometry (Fishman and Downs, 1966). Lanthanum chloride is added to mask interferences.

2.2 This procedure may be automated by the addition of a sampler, a proportioning pump, and either a strip-chart recorder or a printer or both (fig. 1).

3. Interferences

3.1 The interference caused by aluminum concentrations greater than 2,000 µg/L is masked by addition of lanthanum. Because low magnesium values result if the pH of the sample is above 7, standards are prepared in hydrochloric acid solution and samples are preserved in the field with use of nitric acid solution.

3.2 Nitrate, sulfate, and silica interfere, but in the presence of lanthanum chloride hydrochloric acid solution at least 2,000 mg/L, 1,000 mg/L, and 200 mg/L, respectively, can be tolerated. The addition of nitric acid at the time of collection to preserve the samples causes no problem in the following procedure.

3.3 Sodium, potassium, and calcium cause no interference at concentrations less than 400 mg/L.

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to manufacturer's manual to optimize instrument for the following:

Grating --------------------- Ultraviolet
Wavelength ----------------- 285.2 nm
Source (hollow-cathode lamp) -------------------Magnesium
Oxidant ----------------------- Air
Fuel -------------------------- Acetylene
Type of flame --------------- Slightly reducing

4.3 The 50-mm (2-in.), flathead, single-slot burner allows a working range of 0.01 to 5.0 mg/L. This burner, rotated 90°, allows a working range of 2.5 to 50 mg/L. Different burners may be used according to manufacturers' instructions.

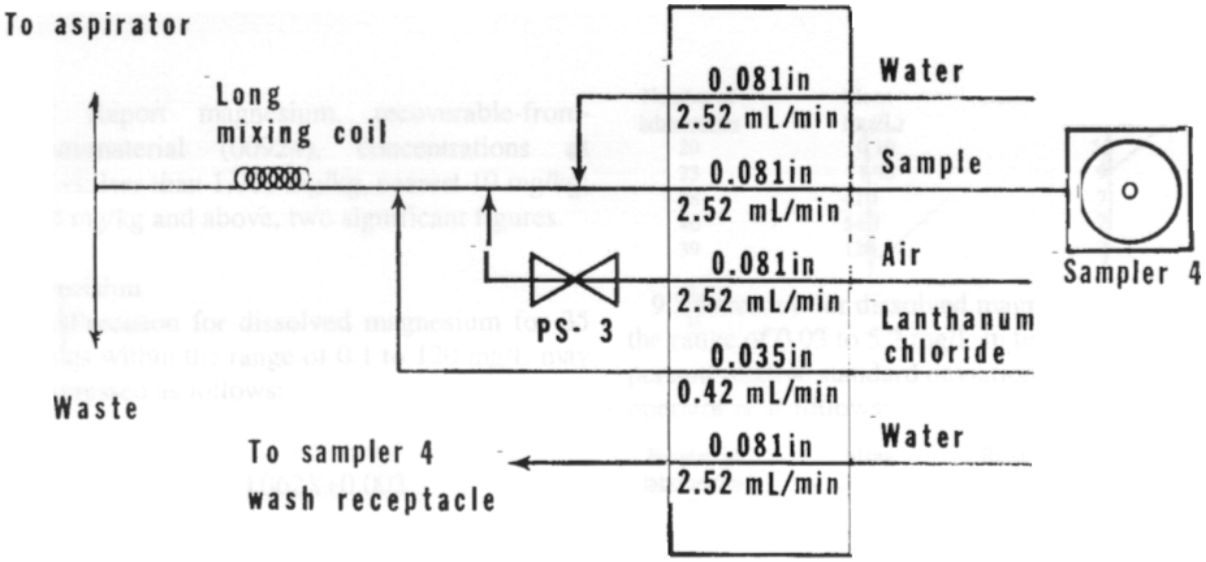

Figure 1. - Magnesium manifold.

5. Reagents

5.1 *Lanthanum chloride solution,* 87 g/L: Mix 29 g La_2O_3 with a few milliliters of demineralized water to form a slurry. Slowly add 250 mL concentrated HCl (sp gr 1.19) while stirring (reaction may be violent) to dissolve the La_2O_3. Dilute to 500 mL with demineralized water.

5.2 *Magnesium standard solution I,* 1.00 mL = 0.50 mg Mg: Dissolve 0.500 g pure metallic Mg in a minimum amount of dilute HCl, and dilute to 1,000 mL with demineralized water.

5.3 *Magnesium working standards:* Prepare a series of at least six working standards containing either from 0.01 to 5.0 mg/L or from 2.5 to 50 mg/L magnesium by appropriate dilution of magnesium standard solution I. Add 1.0 mL $LaCl_3$ solution for each 10 mL of working standard. Similarly, prepare a demineralized water blank.

6. Procedure

6.1 Add 1.0 mL lanthanum chloride solution to 10.0 mL of sample solution.

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 milligrams per liter of dissolved or total recoverable magnesium in each sample solution from the digital display or printer while aspirating each sample. Dilute those samples containing magnesium concentrations that exceed the working range of the method and multiply by the proper dilution factors.

7.2 To determine milligrams per liter suspended recoverable magnesium, subtract dissolved-magnesium concentration from total-recoverable-magnesium concentration.

7.3 To determine milligrams per kilogram of magnesium in bottom-material samples, first determine the milligrams per liter of magnesium in each sample as in paragraph 7.1; then

$$\text{Mg (mg/kg)} = \frac{\text{mg/L Mg} \times \dfrac{\text{mL of original digest}}{1000}}{\text{wt of sample (kg)}}$$

8. Report

8.1 Report magnesium, dissolved (00925), total-recoverable (none assigned), and suspended-recoverable (00926), concentrations as follows: less than 1.0 mg/L, two decimals; 1.0 to 10 mg/L, one decimal; 10 mg/L and above, two significant figures.

8.2 Report magnesium, recoverable-from-bottom-material (00924), concentrations as follows: less than 1,000 mg/kg, nearest 10 mg/kg; 1,000 mg/kg and above, two significant figures.

9. Precision

9.1 Precision for dissolved magnesium for 35 samples within the range of 0.1 to 120 mg/L may be expressed as follows:

$$S_T = 0.062X+0.003$$

where

S_T = overall precision, milligrams per liter, and

X = concentration magnesium, milligrams per liter.

The correlation coefficient is 0.9823.

9.2 Precision for dissolved magnesium for five of the 35 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (mg/L)	Relative standard deviation (percent)
20	0.10	50
23	1.98	9
38	11.1	7
40	54.7	7
39	120	7

9.3 Precision for dissolved magnesium within the range of 0.03 to 5.2 mg/L in terms of the percent relative standard deviation by a single operator is as follows:

Number of laboratories	Mean (mg/L)	Relative standard deviation (percent)
8	0.033	21.2
8	0.315	1.6
7	0.993	2.6
6	5.18	4.2

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

Reference

Fishman, M. J., and Downs, S C., 1966, Methods for analysis of selected metals in water by atomic absorption: U.S. Geological Survey Water-Supply Paper 1540-C, p. 30-5.