Manganese, atomic absorption spectrometric, direct

Parameters and Codes:
Manganese, dissolved, I-1454-85 (µg/L as Mn): 01056
Manganese, total recoverable, I-3454-85 (µg/L as Mn): 01055
Manganese, suspended recoverable, I-7454-85 (µg/L as Mn): 01054
Manganese, recoverable-from-bottom-material, dry wt, I-5454-85 (µg/g as Mn): 01053

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 10 µg/L of manganese. Sample solutions containing more than 1,000 µg/L need either to be diluted or to be read on a less expanded scale. Brines need to be analyzed by the atomic absorption spectrometric, chelation extraction method, providing that the interferences discussed in that method are not exceeded.

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

1.3 This method may be used to analyze bottom material containing at least 1 µg/g of manganese. Prepared sample solutions containing more than 1,000 µg/L need either to be diluted or to be read on a less expanded scale.

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

2. Summary of method

2.1 Manganese is determined by atomic absorption spectrometry by direct aspiration of the sample into an air-acetylene flame without preconcentration or pretreatment.

2.2 The procedure may be automated by the addition of a sampler and either a strip-chart recorder or a printer or both.

3. Interferences

Magnesium (100 mg/L) and silica (100 mg/L) do not interfere. Magnesium in excess of 100 mg/L may present some interference, especially when the manganese concentration exceeds 500 µg/L. Silica interferes above 100 mg/L. Iron concentration to 4 X 10^6 µg/L does not interfere.

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

4.3 The 100-mm (4-in.), flathead, single-slot burner allows a working range from 10 to 1,000 µg/L. Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Manganese standard solution I,* 1.00 mL = 100 µg Mn: Dissolve 0.1000 g manganese flakes in a minimum of dilute HNO_3. Heat to increase rate of dissolution. Add 10.0 mL of concentrated HNO_3 (sp gr 1.41) and dilute to 1,000 mL with demineralized water.

5.2 *Manganese standard solution II,* 1.00 mL = 10.0 µg Mn: Immediately before use,

dilute 10.0 mL manganese standard solution I to 100 mL with demineralized water.

5.3 Manganese working standards: Prepare at least six working standards containing from 10 to 1,000 µg/L manganese by appropriate dilution of manganese standard solution II with acidified water. Prepare fresh daily.

5.4 Water, acidified: Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to 1 L 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 manganese in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing manganese concentrations 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 manganese, subtract dissolved-manganese concentration from total-recoverable-manganese concentration.

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

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

8. Report

8.1 Report manganese, dissolved (01056), total-recoverable (01055), and suspended-recoverable (01054), concentrations as follows: less than 100 µg/L, nearest 10 µg/L; 100 µg/L and above, two significant figures.

8.2 Report manganese, recoverable-from-bottom-material (01053), concentrations as follows: less than 10 µg/g, nearest microgram per gram; 10 µg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved manganese for 30 samples within the range of 3.0 to 568 µg/L may be expressed as follows:

$$S_T = 0.056X + 8.28$$

where

S_T = overall precision, micrograms per liter, and

X= concentration of manganese, micrograms per liter.

The correlation coefficient is 0.9003.

9.2 Precision for dissolved manganese for six of the 30 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
6	3.0	267
6	12	83
27	60	17
25	106	12
34	256	9
36	568	7

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

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

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
21	52	44
22	317	6