

Iron, atomic absorption spectrometric, direct

Parameters and Codes:

Iron, dissolved, I-1381-85 ($\mu\text{g/L}$ as Fe): 01046

Iron, total recoverable, I-3381-85 ($\mu\text{g/L}$ as Fe): 01045

Iron, suspended recoverable, I-7381-85 ($\mu\text{g/L}$ as Fe): 01044

Iron, recoverable-from-bottom-material, dry wt I-5381-85 ($\mu\text{g/g}$ as Fe): 01170

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least $10 \mu\text{g/L}$ of iron. Sample solutions containing more than $1,000 \mu\text{g/L}$ need either to be diluted or to be read on a less expanded scale.

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

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

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

2. Summary of method

2.1 Iron is determined by atomic absorption spectrometry by direct aspiration of the sample solution into an air-acetylene flame.

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

Individual concentrations of sodium, potassium, chloride, and sulfate ($5,000 \text{ mg/L}$ each), calcium and magnesium ($1,000 \text{ mg/L}$ each), nitrate (100 mg/L), chromium, manganese, cobalt, nickel, copper, zinc, silver, cadmium, tin, lead, lithium, mercury, selenium, aluminum,

antimony, arsenic, vanadium, boron, and molybdenum ($1 \times 10^5 \mu\text{g/L}$ each) 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 ----- 248.3 nm

Source (hollow-cathode lamp) ----- Iron

Oxidant ----- Air

Fuel ----- Acetylene

Type of flame ----- Oxidizing

4.3 Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Iron standard solution I*, $1.00 \text{ mL} = 400 \mu\text{g Fe}$: Weigh 0.400 g analytical grade Fe wire that has been cleaned in dilute HCl, rinsed, and dried. Dissolve in 5 mL concentrated HNO_3 (SP gr 1.41), warming if necessary, and dilute to $1,000 \text{ mL}$ with demineralized water.

5.2 *Iron standard solution II*, $1.00 \text{ mL} = 4.00 \mu\text{g Fe}$: Dilute 10.0 mL iron standard solution I and 1 mL concentrated HNO_3 (sp gr 1.41) to $1,000 \text{ mL}$ with demineralized water. This standard is used to prepare working standards at time of analysis.

5.3 Iron working standards: Prepare a series of at least six working standards containing

from 10 to 1,000 µg/L of iron by appropriate dilution of iron standard solution II with acidified water. Prepare fresh daily.

5.4 Water, acidified: Add 1.5 mL concentrated HNO₃ (sp gr 1.41) to 1 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 iron in each sample solution from the digital display or printer while aspirating each sample. Dilute those samples containing iron 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 iron, subtract dissolved-iron concentration from total-recoverable-iron concentration.

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

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

8. Report

8.1 Report iron, dissolved (01046), total-recoverable (01045), and suspended-recoverable (01044), concentrations as follows: less than 100

µg/L, nearest 10 µg/L; 100 µg/L and above, two significant figures.

8.2 Report iron, recoverable-from-bottom-material (01170), concentrations as follows: less than 100 µg/g, nearest microgram per gram; 100 µg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved iron for 26 samples within the range of 38 to 996 µg/L may be expressed as follows:

$$S_T = 0.066X + 18.58$$

where

S_T = overall precision, micrograms per liter, and

X = concentration of iron, micrograms per liter.

The correlation coefficient is 0.8305.

9.2 Precision for dissolved iron for six of the 26 samples expressed in terms of the percent relative standard deviation is as follows:

<u>Number of laboratories</u>	<u>Mean (µg/L)</u>	<u>Relative standard deviation (percent)</u>
18	38	42
29	110	25
22	277	12
16	445	10
33	750	8
13	996	6

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

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

<u>Number of Laboratories</u>	<u>Mean (µg/L)</u>	<u>Relative standard deviation (percent)</u>
24	7376	49
19	7680	38