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Water quality — Determination of iron — Spectrometric method using 1,10-phenanthroline

Qualité de l'eau — Dosage du fer — Méthode spectrométrique à la phénanthroline-1,10

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6332 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 6332 : 1982), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Water quality — Determination of iron — Spectrometric method using 1,10-phenanthroline

1 Scope and field of application

This International Standard specifies a 1,10-phenanthroline spectrometric method for the determination of iron in water and waste water. Procedures are described for the determination of

- a) total iron (sum of dissolved and undissolved iron) :
 - 1) direct determination,
 - 2) determination after decomposition;
- b) total dissolved iron [sum of dissolved iron(II) and iron(III)];
- c) determination of dissolved iron(II).

The methods are applicable to the determination of iron concentrations between 0,01 and 5 mg/l. Iron concentrations above 5 mg/l may be determined after suitable dilution of the sample.

For interferences see clause 10.

2 Reference

ISO 5667-1, *Water quality — Sampling — Part 1 : Guidance on the design of sampling programmes*.

3 Principle

Addition of 1,10-phenanthroline solution to a test portion and photometric measurement of the orange-red complex at a wavelength of about 510 nm.

If determining total iron or total soluble iron, hydroxylammonium chloride is added to reduce iron(III) to iron(II). If undissolved iron, iron oxides or iron complexes are present, pretreatment is necessary to bring such compounds into solution (see 7.1.2).

The iron(II)-1,10-phenanthroline complex is stable in the pH range from 2,5 to 9 and the intensity of the colour is proportional to the amount of iron(II) present. The relationship between concentration and absorbance is linear up to a concentration of 5,0 mg of iron per litre. Maximum absorbance occurs at about 510 nm [molar absorption coefficient $11 \times 10^3 \text{ l}/(\text{mol}\cdot\text{cm})$].

4 Reagents

Use only reagents of recognized analytical grade.

The water used shall have as low an iron concentration as possible; a measurable iron concentration in the reagents is permissible provided that the lowest concentration to be determined is at least three times the standard deviation of the predetermined results of blank tests. Deionized water or water distilled from an all-glass apparatus has been found to be suitable.

4.1 Sulfuric acid, $\rho = 1,84 \text{ g/ml}$.

4.2 Sulfuric acid solution, $c(1/2 \text{ H}_2\text{SO}_4) \approx 4,5 \text{ mol/l}$.

Add slowly and with vigorous stirring 1 volume of concentrated sulfuric acid (4.1) to 3 volumes of water while cooling.

4.3 Nitric acid, concentrated, $\rho = 1,40 \text{ g/ml}$.

4.4 Hydrochloric acid solution, $\rho = 1,12 \text{ g/ml}$, $c(\text{HCl}) \approx 7,7 \text{ mol/l}$.

4.5 Acetate buffer.

Dissolve 40 g of ammonium acetate ($\text{CH}_3\text{COONH}_4$) and 50 ml of glacial acetic acid (CH_3COOH) ($\rho = 1,06 \text{ g/ml}$) in water and dilute to 100 ml with water.

4.6 Hydroxylammonium chloride, 100 g/l solution.

Dissolve 10 g of hydroxylammonium chloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in water and dilute to 100 ml.

This solution is stable for at least 1 week.

4.7 1,10-phenanthroline solution.

Dissolve 0,5 g of 1,10-phenanthroline chloride (monohydrate) ($\text{C}_{12}\text{H}_9\text{ClN}_2\cdot\text{H}_2\text{O}$) in water and dilute to 100 ml.

Alternatively, dissolve 0,42 g of 1,10-phenanthroline monohydrate ($\text{C}_{12}\text{H}_8\text{N}_2\cdot\text{H}_2\text{O}$) in 100 ml of water containing 2 drops of hydrochloric acid (4.4).

This solution is stable for 1 week if stored in the dark.