SLOVENSKI STANDARD
oSIST prEN ISO 4937:2024

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Jeklo in železo - Določevanje kroma - Potenciometrična ali vizualna titracijska metoda (ISO/DIS 4937:2024)

Steel and iron - Determination of chromium content - Potentiometric or visual titration method (ISO/DIS 4937:2024)

Stahl und Eisen - Bestimmung des Chromgehalts - Potentiometrische oder visuelle Titrationsmethode (ISO/DIS 4937:2024)

Aciers et fontes - Détermination du chrome - Méthode par titrage potentiométrique ou visuel (ISO/DIS 4937:2024)

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ISO/DIS 4937

Steel and iron — Determination of chromium content — Potentiometric or visual titration method

Aciers et fontes — Dosage du chrome — Méthode par titrage potentiométrique ou visuel

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Contents

Page

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459/SC 2, *Methods of chemical analysis for iron and steel*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 4937:1986), which has been revised. The main changes are as follows:

— re-assessment of the precision data.

— re-confirmation of upper limit of vanadium content in test portions by visual titration .

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Steel and iron — Determination of chromium content — Potentiometric or visual titration method

1 Scope

This document specifies a method for the determination of chromium in steel and iron by potentiometric or visual titration.

The method is applicable to chromium contents between 0,25 % (mass fraction) and 35 % (mass fraction).

If vanadium is present, the visual titration is applicable only to test portions containing less than 3 mg of vanadium.

NOTE The visual titration can be applicable to test portion containing between 3 mg and 6 mg of vanadium.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — One-mark pipettes.*

IS0 1042, *Laboratory glassware —One-mark volumetric flasks.*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Dissolution of a test portion with appropriate acids.

Oxidation of chromium in an acid medium to chromium(Vl) by ammonium peroxydisulfate in the presence of silver sulfate. Reduction of manganese(VII) by hydrochloric acid.

Reduction of chromium(Vl) by an ammonium iron(II) sulfate standard solution.

In the case of potentiometric detection, determination of the equivalence point by measurement of the potential variation when the ammonium iron(ll) sulfate standard solution is being added.

In the case of visual detection, titration the excess of ammonium iron(II) sulfate by a potassium permanganate standard solution which also acts as the indicator.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

5.1 Urea.

5.2 Perchloric acid, ρ approximately 1,67 g/ml.

5.3 Hydrofluoric acid, ρ approximately 1,15 g/ml.

5.4 Phosphoric acid, ρ approximately 1,70 g/ml.

5.5 Nitric acid, ρ approximately 1,40 g/ml.

5.6 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 1 + 1.

5.7 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 1 + 10.

5.8 Sulfuric acid, ρ approximately 1,84 g/ml, diluted 1 + 1.

5.9 Sulfuric acid, ρ approximately 1,84 g/ml, diluted 1 + 5.

5.10 Sulfuric acid, ρ approximately 1,84 g/ml, diluted 1 + 19.

5.11 Silver sulfate, 5 g/l.

5.12 Ammonium peroxydisulfate [$(NH_4)_2S_20_8$], 500 g/l.

Prepare this solution immediately before use.

5.13 Manganese sulfate [$MnS0_4 \cdot H_20$], 4 g/l.

5.14 Manganese sulfate [$MnS0_4 \cdot H_20$], 100 g/l.

5.15 Potassium permanganate, 5 g/l.

5.16 Sodium nitrite, 3 g/l.

Prepare this solution immediately before use.

5.17 Sulfamic acid (NH_2S0_3H), 100 g/l.

This solution remains stable for one week only.

5.18 Potassium permanganate, standard solution.

5.18.1 Preparation of the solution

Dissolve 3,2 g of potassium permanganate in 1 000 ml of water. After storage in complete darkness for 2 weeks, filter through a thick fritted filter without washing. Keep the solution in a coloured glass bottle and avoid contact with organic matter.

5.18.2 Standardization of the solution

Boil 250 ml of sulfuric acid (5.10) in a 600 ml beaker for 10 min and allow to cool. Weigh, to the nearest 0,000 1 g, 0,300 0 g of sodium oxalate [$Na_2(COO)_2$] previously dried at 105 °C and cooled in a desiccator.

Dissolve the salt in the boiled sulfuric acid (5.10). Add 39 ml to 40 ml of potassium permanganate solution (5.18.1) at a rate of 25 ml/min to 35 ml/min, stirring gently. The violet colour of the permanganate will disappear in approximately 45 s. Heat to 70 °C to 75 °C and complete the titration.

Towards the end, titrate very slowly and allow each drop to become colourless before adding the next.

To determine the blank test, titrate 250 ml of sulfuric acid (5.10), as described above, concurrently.

The concentration (ρ_2) of the potassium permanganate standard solution, expressed as milligrams of chromium per millilitre, is given by the Formula (1).

$$\rho_2 = \frac{300{,}0 \times 1{,}733}{6{,}700 \times (V_1 - V_0)} \qquad (1)$$

where

V_1 is the volume, in millilitres, of potassium permanganate solution (5.18.1) used for titrating sodium oxalate;

V_0 is the volume, in millilitres, of potassium permanganate solution (5.18.1) used for titrating the blank test of sulfuric acid (5.10);

6,700 is the molar mass of sodium oxalate divided by 20;

1,733 is the mass, in milligrams, of chromium(Vl) corresponding to 1 ml of the potassium dichromate standard reference solution (5.20);

300,0 is the mass, in milligrams, of sodium oxalate weighed.

5.19 Ammonium iron(II) sulfate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$], standard solution in sulfuric acid medium.

1 ml of this solution corresponds to about 2 mg of chromium.

5.19.1 Preparation of the solution

Dissolve 46 g of ammonium iron(II) sulfate hexahydrate in about 500 ml of water, add 110 ml of sulfuric acid (5.8), cool, dilute to 1 000 ml and mix.

5.19.2 Potentiometric standardization of the solution (to be carried out just before use)

Transfer 30,0 ml of the potassium dichromate standard reference solution (5.20), into a 600 ml beaker, add 45 ml of sulfuric acid (5.9) and make up to about 400 ml with water.

Titrate according to the conditions specified in 8.3.3.1.

The corresponding concentration (ρ_1) of ammonium iron(II) sulfate solution (5.19.1), expressed in milligrams of chromium per millilitre, is given by the Formula (2).

$$\rho_1 = \frac{30{,}0 \times 1{,}733}{V_2} \qquad (2)$$

where

V_2 is the volume, in millilitres, of ammonium iron(II) sulfate solution (5.19.1) used for the standardization ;

30,0 is the volume, in millilitres, of the potassium dichromate standard reference solution (5.20) taken for the standardization;

1,733 is the mass, in milligrams, of chromium corresponding to 1 ml of the potassium dichromate standard reference solution (5.20).

5.19.3 Visual standarization of the solution (to be carried out just before use)

Take 25,0 ml of ammonium iron(II) sulfate solution (5.19.1) and add 325 ml of sulfuric acid (5.10).

Titrate with the potassium permanganate standard solution (5.18) until a slight violet colour persists.

To determine the blank test, titrate a mixture of 25 ml of water and 325 ml of sulfuric acid (5.10) with the potassium permanganate standard solution (5.18).

The corresponding concentration (ρ'_1) of the ammonium iron (II) sulfate standard solution (5.19), expressed in milligrams of chromium per millilitre, is given by the Formula (3).

$$\rho'_1 = \rho_2 \times \frac{V_3 - V_0}{25{,}0} \qquad (3)$$

where

ρ_2 is the concentration of the potassium permanganate standard solution (5.18), expressed as milligrams of chromium per millilitre;

V_3 is the volume, in millilitres, of the potassium permanganate standard solution (5.18) used to oxidize 25 ml of ammonium iron(II) sulfate solution (5.19.1) ;

V_0 is the volume, in millilitres, of the potassium permanganate standard solution (5.18) used for titrating the blank test of sulfuric acid (5.10);

25,0 is the volume, in millilitres, of ammonium iron(II) sulfate solution (5.19.1) used for the standardization.

5.20 Potassium dichromate, standard reference solution.

Weigh, to the nearest 0,000 1 g, 4,903 1 g of potassium dichromate previously dried at 150 °C to constant mass and cooled in a desiccator.

Dissolve in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard reference solution contains 1,733 mg of Cr.