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Steel and iron — Determination of titanium content — Diantipyrylmethane spectrophotometric method

Aciers et fontes — Détermination du titane — Méthode spectrophotométrique au diantipyrylméthane

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Foreword

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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 10280:1991), which has been technically revised.

The main changes are as follows:

— Clause 2, Normative references, has updated;

— the mandatory Clause 3, Terms and definitions has been added and subsequent clauses have been renumbered;

— precision data has been re-evaluated.

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Steel and iron — Determination of titanium content — Diantipyrylmethane spectrophotometric method

1 Scope

This document specifies a diantipyrylmethane spectrophotometric method for the determination of titanium in steel and iron.

The method is applicable to titanium contents between 0,002 % (mass fraction) and 0,80 % (mass fraction).

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 648, *Laboratory glassware — Single-volume pipettes*

ISO 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 terminological 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 in hydrochloric, nitric and sulphuric acids.

Fusion of the residue with potassium hydrogen sulphate.

Formation of a yellow complex with 4,4'-diantipyrylmethane.

Spectrophotometric measurement of the coloured complex at a wavelength of about 385 nm.

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 Iron, of high purity containing less than 2 µg/g titanium.

5.2 Potassium hydrogen sulphate ($KHSO_4$).

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5.3 Sodium carbonate (Na_2CO_3), anhydrous.

5.4 Hydrochloric acid, ρ about 1,19 g/ml.

5.5 Nitric acid, ρ about 1,40 g/ml.

5.6 Hydrofluoric acid, ρ about 1,15 g/ml.

5.7 Hydrochloric acid, solution 1 + 1.

Carefully add 100 ml of hydrochloric acid (5.4) to 100 ml of water and mix.

5.8 Hydrochloric acid, solution 1 + 3.

Carefully add 50 ml of hydrochloric acid (5.4) to 150 ml of water and mix.

5.9 Sulphuric acid, solution 1 + 1.

Carefully add 100 ml of sulphuric acid (ρ about 1,84 g/ml) to 100 ml of water.

Allow to cool and mix

5.10 Tartaric acid, 100 g/l.

5.11 Ascorbic acid, 100 g/l.

Prepare this solution immediately before use.

5.12 Ammonium oxalate, 30 g/l.

Dissolve 6 g of ammonium oxalate monohydrate [$(COONH_4)_2 \cdot H_2O$] in water, dilute to 200 ml with water and mix.

5.13 Iron solution, 12,5 g/l.

Dissolve 1,25 g of iron (5.1) in 10 ml of hydrochloric acid (5.7) while heating gently. Add 5 ml of nitric acid (5.5) and boil until the volume of the solution is approximately 10 ml. Cool, transfer into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.14 Reagent blank solution.

Prepare a reagent blank solution in parallel with the determination, using the same quantities of reagents as those used for the determination but without iron. Follow exactly the procedure described in 8.3.1 and 8.3.2, dilute to 100 ml with water and mix.

5.15 Diantipyrylmethane, 40 g/l**.**

Dissolve 4 g of 4,4'-methylenebis(2,3-dimethyl-1-phenyl-5-pyrazolone) monohydrate, $C_{23}H_{24}O_2N_4.H_2O$, (diantipyrylmethane) in 20 ml of hydrochloric acid (5.7), dilute to 100 ml with water and mix.

5.16 Titanium standard solution, 1 g/l.

Weigh, to the nearest 0,001 g, 0,500 g of high purity titanium [purity > 99,9 % (mass fraction)] and transfer into a 300 ml beaker. Add 180 ml of sulfuric acid, ρ about 1,84 g/ml, diluted 1+3, cover with a watch glass, heat gently until the metal is dissolved and oxidize with nitric acid (5.5) added drop by drop. Allow to cool, transfer quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1,0 mg of titanium.

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5.17 Titanium standard solution, 50 mg/l.

Transfer 10,0 ml of the titanium standard solution (5.16) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 50 µg of titanium.

6 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 648 or ISO 1042 as appropriate.

Ordinary laboratory apparatus, and the following.

6.1 Platinum or platinum-gold alloy crucibles, of capacity 30 ml.

6.2 Spectrophotometer

The spectrophotometer shall be equipped to measure absorbances at a wavelength of 385 nm.

Wavelength adjustment shall be accurate to ±2 nm or less.

In the absorbance range 0,05 to 0,85, absorbance measurements shall be repeatable to ±0,003 or better.

7 Sampling and preparation of the test samples

Sampling and preparation of the test samples shall be carried out in accordance with ISO 14284 or appropriate national standards for steel and cast iron.

8 Procedure

8.1 Test portion

Weigh, to the nearest 1 mg, a test portion according to the presumed titanium content as follows:

a) for titanium contents in the range 0,002 % (mass fraction) to 0,125 % (mass fraction): 1,00 g;

b) for titanium contents in the range 0,125 % (mass fraction) to 0,80 % (mass fraction): 0,50 g.

8.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents and the same cell as for the determination, substituting an equivalent amount of iron (5.1) for the test portion.

8.3 Determination

8.3.1 Dissolution of the test portion

Place the test portion (8.1) in a 250 ml beaker. Add 20 ml of hydrochloric acid (5.4), cover the beaker with a watch glass and heat at 70 °C to 90 °C until the acid action ceases. Add 5 ml of nitric acid (5.5) and boil until the volume of the solution is approximately 10 ml.

Allow the solution to cool, add 20 ml of sulfuric acid (5.9) and evaporate until fumes of sulfur trioxide appear.

Just before fuming occurs, solid particles will begin to form and a gentle heating is required to avoid spurting.

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