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Alloyed steels — Determination of manganese — Potentiometric or visual titration method

Aciers alliés — Détermination du manganèse — Méthodes par titration visuelle ou potentiométrique

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Foreword

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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 on 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 the following URL: 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*.

This second edition cancels and replaces the first edition (ISO 18632:2010), which has been technically revised. The following changes have been made:

— a procedure has been added for the removal of the oxidized layer when the manganese standard solution is prepared;

— superfluous figures have been deleted in Table A.2;

— Annex C has been added to explain the main redox reaction and the correction of vanadium and cerium content in the document.

Alloyed steels — Determination of manganese — Potentiometric or visual titration method

1 Scope

This document specifies a potentiometric or visual titration method for the determination of manganese content in alloyed steels.

The method is applicable to manganese mass fractions between 2 % and 25 %. Vanadium and cerium interfere with the determination. If the mass fraction of cerium in the sample is less than 0,01 %, or the mass fraction of vanadium in the sample is less than 0,005 %, the interference is negligible, otherwise theoretical corrections are necessary.

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 — 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 appropriate acids. Addition of phosphoric acid. Oxidation of manganese to manganese(III) in phosphoric acid medium by ammonium nitrate. Visual titration of manganese(III) with a ferroammoniumdisulfate standard solution with N-phenylanthranilic acid as indicator, or potentiometric titration with a ferroammoniumdisulfate standard solution. If the sample contains vanadium and/or cerium, the manganese content shall be corrected.

5 Reagents

During the analysis use only reagents of recognized analytical grade and only grade 2 water in accordance with ISO 3696.

5.1 Ammonium nitrate, NH_4NO_3.

5.2 Urea.

5.3 Phosphoric acid, ρ approximately 1,69 g/ml.

5.4 Nitric acid, ρ approximately 1,42 g/ml.

5.5 Hydrochloric acid, ρ approximately 1,19 g/ml.

5.6 Sulfuric acid, diluted 1 + 3.

5.7 Sulfuric acid, diluted 5 + 95.

5.8 N-phenylanthranilic acid solution, $C_6H_5NHC_6H_4COOH$, approximately 2 g/l.

Dissolve 0,20 g of N-phenylanthranilic acid and 0,20 g of sodium carbonate in 100 ml of water and filter.

5.9 Potassium dichromate solution, $K_2Cr_2O_7$, 0,002 5 mol/l.

Weigh 0,735 5 g of high purity potassium dichromate, previously dried at 150 °C for at least 2 h and cooled in a desiccator. Introduce it into a 250 ml beaker and dissolve in some water. Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.10 Manganese standard solution, corresponding to 1,00 g/l of manganese.

Weigh, to the nearest 0,1 mg, 1,000 g of pure manganese [purity ≥ 99,9 % (mass fraction)].

When the surface of manganese seems oxidized, the oxide layer should be removed before weighing as follows:

a) introduce several grams of manganese into a beaker containing sulfuric acid (5.7) and stir;

b) decant, discard the sulphuric acid solution and immediately rinse the metal several times, firstly with water and then with ethanol or acetone;

c) dry the metal for about 2 min at 100 °C and cool in a desiccator.

Introduce it in a 250 ml beaker and add 40 ml of hydrochloric acid (5.5). Cover with a watch glass and heat gently to complete dissolution. Cool and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1,00 mg of manganese.

5.11 Ferroammoniumdisulfate standard solution, $[(NH_4)_2Fe(SO_4)_2{\cdot}6H_20]$ 0,015 mol/l.

5.11.1 Preparation of the solution

Dissolve 5,9 g of ferroammoniumdisulfate in 1 000 ml sulfuric acid (5.7) and mix.