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Ferrovanadium — Determination of vanadium content — Potentiometric method

Ferrovanadium — Dosage du vanadium — Méthode 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 132, *Ferroalloys*.

This second edition cancels and replaces the first edition (ISO 6467:1980), which has been technically revised. Changes have been made to the vanadium range, the procedure and the precision.

Ferrovanadium — Determination of vanadium content — Potentiometric method

1 Scope

This document specifies a potentiometric method for the determination of the vanadium content of ferrovanadium.

The method is applicable to vanadium contents between 35,0 % and 85,0 % (mass fraction) in ferrovanadium.

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 3713, *Ferroalloys — Sampling and preparation of samples — General rules*

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:

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

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

4 Principle

Dissolution of a test portion with nitric and sulfuric acids. Cold oxidation of the vanadium(IV) to vanadium(V) by a slight excess of potassium permanganate. Destruction of the excess of potassium permanganate by potassium nitrite, the excess of the latter being itself destroyed by urea. Reduction of the vanadium(V) to vanadium(IV) by iron(II) in a potentiometric titration.

5 Reagents

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

5.1 Urea.

5.2 Nitric acid, ρ 1,38 to 1,42 g/ml.

5.3 Phosphoric acid.

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

Add cautiously, while stirring, 500 ml of sulfuric acid, ρ approximately 1,84 g/ml to 400 ml of water. Cool, dilute to 1 000 ml with water and mix.

5.5 Potassium nitrite, 10 mg/ml solution.

Dissolve 10 g of potassium nitrite in water, dilute to 1 000 ml and mix.

5.6 Potassium permanganate, 6,3 mg/ml.

Dissolve 6,3 g of potassium permanganate in water, made up with water to a volume of 1 000 ml and mix.

5.7 Potassium dichromate, standard solution, C_1 ($1/6K_2Cr_2O_7$) = 0,2 mol/l.

Weigh, to the nearest 0,000 5 g, exactly 9,806 4 g of potassium dichromate previously oven-dried at 105 °C. Dissolve with water in a 1 000 ml volumetric flask. Dilute to the mark and mix.

5.8 Ammonium iron(II) sulphate, standard volumetric solution, C_2 ($FeSO_4(NH_4)_2SO_4$) ≈ 0,2 mol/l.

5.8.1 Preparation

In a 1 000 ml volumetric flask, dissolve 78,4 g of ammonium iron(II) sulphate ($FeSO_4(NH_4)_2SO_4.6H_2O$) with 500 ml of warm water. When the dissolution is complete, add 100 ml of the sulfuric acid (5.4), cool, dilute to the mark and mix.

5.8.2 Standardization

In a 600 ml beaker containing 270 ml of water, 20 ml of the sulfuric acid (5.4) and 10 ml of the phosphoric acid (5.3), introduce 40 ml of the potassium dichromate solution (5.7) using a burette. The potentiometric titration is carried out with the ammonium iron(II) sulphate solution. The end of the reaction is obtained when the maximum fall of potential is observed. The concentration of the ammonium iron(II) sulphate solution C_2 is given by Formula (1):

$$C_2 = \frac{C_1 \times V_1}{V_2} \quad (1)$$

where

C_1 is the concentration, in moles per litre, of the potassium dichromate standard solution (5.7);

V_1 is the volume, in millilitres, of the potassium dichromate standard solution (5.7);

V_2 is the volume, in millilitres, of the ammonium iron(II) sulphate solution (5.8) used.

The ammonium iron(II) solution is not stable and the actual concentration shall be determined at the time of use.

6 Apparatus

All volumetric glassware shall be Class A, in accordance with ISO 648 and ISO 1042. Ordinary laboratory apparatus and the following shall be used.

6.1 Beaker, capacity 400 ml.