INTERNATIONAL STANDARD

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# Steel and cast iron — Determination of vanadium content — Potentiometric titration method

*Acier et fonte — Détermination des teneurs en vanadium — Méthode par titrage 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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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 4947:1986), which has been technically revised. The main changes compared to the previous edition are as follows:



— introduction of an optional electrode;

— re-assessment of precision data.

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 cast iron — Determination of vanadium content — Potentiometric titration method

## 1 Scope

This document specifies a potentiometric titration method for the determination of vanadium in steel and cast iron.

The method is applicable to vanadium contents between 0,04 % (mass fraction) and 2 % (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 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 http://www.iso.org/obp

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

## 4 Principle

Dissolution of a test portion with appropriate acids. Addition of hydrofluoric acid to keep tungsten in solution.

Oxidation of chromium and vanadium by potassium peroxydisulfate. Partial oxidation of chromium.

While checking the potential of the solution:

— reduction of chromium(VI) and vanadium(V) by ammonium iron(II) sulfate;

— oxidation of vanadium by a slight excess of potassium permanganate; reduction of the excess of permanganate by sodium nitrite, and reduction of the excess of sodium nitrite by sulfamic acid.

Potentiometric titration of vanadium with an ammonium iron(II) sulfate standard solution.

## 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, free from reducing or oxidizing activity.

**5.1 Potassium peroxydisulfate** ($K_2S_2O_8$).

**5.2 Hydrochloric acid,** $\rho$ approximately 1,19 g/ml.

**5.3 Nitric acid,** $\rho$ approximately 1,40 g/ml.

**5.4 Hydrofluoric acid,** $\rho$ approximately 1,15 g/ml.

**5.5 Sulfuric acid,** $\rho$ approximately 1,84 g/ml, diluted 1 + 4.

**5.6 Sulfuric acid,** $\rho$ approximately 1,84 g/ml, diluted 1 + 50.

**5.7 Orthophosphoric acid,** ρ approximately 1,70 g/ml.

**5.8 Ammonium iron(II) sulfate** [$Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$] solution in sulfuric acid medium.

Dissolve 40 g of ammonium iron(II) sulfate hexahydrate in approximately 500 ml of water, add 20 ml of sulfuric acid, $\rho$ approximately 1,84 g/ml, allow to cool, make up the volume to 1 000 ml with water and mix.



**5.9 Potassium permanganate,** 5 g/l.

**5.10 Sodium nitrite,** 3 g/l.

**5.11 Sulfamic acid** ($NH_2SO_3H$)**,** 100 g/l.



This solution is stable for only one week.

**5.12 Potassium dichromate,** standard solution.

Weigh, to the nearest 0,001 g, approximately 1 g of potassium dichromate (high purity grade) previously dried at 150 °C until a constant mass (the mass difference shall not exceed 0,3 mg) is obtained after cooling in a desiccator. Transfer the weighed mass into a 250 ml beaker, dissolve it in 20 ml of water and add 160 ml of sulfuric acid (5.5). Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, allow to cool, dilute to the mark with water and mix.

**5.13 Ammonium iron(II) sulfate** [$Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$], standard solution.

1 ml of this solution corresponds to approximately 1,299 mg of vanadium.

#### 5.13.1 Preparation of the solution

Dissolve 10 g of ammonium iron(II) sulfate hexahydrate in approximately 500 ml of water, add 25 ml of sulfuric acid, $\rho$ 1,84 g/ml approximately, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, allow to cool, dilute to the mark with water and mix.