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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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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries :

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No member body expressed disapproval of the document.

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# Ferrovanadium — Determination of vanadium content — Potentiometric method

## 1 Scope and field of application

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

The method applies to alloys having a vanadium content less than or equal to 85 % (*m/m*).

## 2 Reference

ISO 3713, *Ferroalloys — Sampling and preparation of samples — General rules.*[1]

## 3 Principle

Dissolution of a test portion with nitric and sulphuric 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.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, except where otherwise specified.

**4.1 Urea.**

**4.2 Nitric acid,** $\varrho$ 1,38 to 1,42 g/ml.

**4.3 Phosphoric acid.**

**4.4 Sulphuric acid,** 50 % (*V/V*) solution, rendered inert to potassium permanganate by adding a slight excess of this reagent.

To 400 ml of water, add cautiously 500 ml of sulphuric acid ($\varrho$ approximately 1,84 g/ml); mix, cool, dilute to 1 000 ml and mix.

**4.5 Potassium nitrite,** 10 g/l solution.

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

**4.6 Potassium permanganate,** 6,3 g/l solution.

Dissolve 6,3 g of potassium permanganate in water, dilute to 1 000 ml and mix.

**4.7 Potassium dichromate,** standard solution, $c(K_2Cr_2O_7) = 0,2$ 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.

**4.8 Ammonium iron(II) sulphate,** standard volumetric solution, $c[FeSO_4(NH_4)_2SO_4] = 0,2$ mol/l.

### 4.8.1 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 sulphuric acid (4.4), cool, dilute to the mark and mix.

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[1] At present at the stage of draft.

#### 4.8.2 Standardization

In a 600 ml beaker containing 280 ml of water, 10 ml of the sulphuric acid (4.4) and 10 ml of the phosphoric acid (4.3), introduce 40 ml of the potassium dichromate solution (4.7) using a burette. Titrate potentiometrically with the ammonium iron(II) sulphate solution. The end of the reaction is obtained when the maximum fall of potential is observed. This is of the order of 100 mV.

The correction factor $C$ is given by the expression

$$C = \frac{40}{V_1}$$

where $V_1$ is the volume, in millilitres, of the ammonium iron(II) sulphate solution used.

The concentration of this ammonium iron(II) solution varies, and it shall be verified for each series of determinations.

## 5 Apparatus

Usual laboratory equipment, and in particular:

### 5.1 **Beaker**, capacity 400 ml.

### 5.2 Precision potentiometer.

#### 5.2.1 Electrodes

Indicating electrode : platinum.

Reference electrode : $Hg/Hg_2SO_4/K_2SO_4$ (saturated).

#### 5.2.2 Checking of measuring system

When it is not being used, the reference electrode should be immersed in a saturated potassium sulphate solution.

The platinum electrode should be periodically checked with a stable redox buffer. Use, for example, a solution equionic in cerium(III) and cerium(IV) salts :

$$\frac{Ce(SO_4)_2}{Ce_2(SO_4)_3} \text{ or } \frac{[Ce^{4+}]}{[Ce^{3+}]} = 1$$

Prepare this buffer by dissolving in distilled water 0,330 g of $Ce(SO_4)_2$ and 0,280 g of $Ce_2(SO_4)_3$. Add sulphuric acid in order to obtain, after introduction into a 1 000 ml volumetric flask, an acidity corresponding to that of a molar solution of sulphuric acid. The response of the platinum electrode is satisfactory if, when it is coupled with the reference electrode, the millivoltmeter shows a reading of between 710 and 740 mV.

### 5.3 Magnetic stirrer.

## 6 Sample

Use powder which will pass through a sieve with a mesh size of 250 µm, prepared in accordance with ISO 3713.

## 7 Procedure

### 7.1 Test portion

Take a test portion of 0,5 ± 0,000 2 g.

### 7.2 Blank test

Carry out a blank test, in parallel with the determination, following the same procedure and using the same reagents.

### 7.3 Check test

Check the validity of the execution of the method by carrying out, in parallel with the analysis and following the same procedure, the determination of the vanadium in one or several samples of the same type and having a known vanadium content.

### 7.4 Determination

**7.4.1** In a 400 ml beaker, dissolve the test portion with 10 ml of water, 10 ml of the nitric acid (4.2) and 50 ml of the sulphuric acid (4.4); evaporate to white sulphuric fumes then allow to cool.

**7.4.2** Add 100 ml of water, poured gently down the side of the beaker and boil to dissolve the salts. Allow to cool and dilute to about 200 ml.

**7.4.3** Place the beaker on the magnetic stirrer, immerse the electrodes, and stir. Add the potassium permanganate solution (4.6), using a 50 ml burette, until the maximum potential is reached. Then wait 15 min. (The potential is stabilized at about 700 ± 50 mV.)

**7.4.4** Destroy the excess of potassium permanganate with the potassium nitrite solution (4.5), added dropwise every 30 s under potentiometric checking. Stop the addition when the potential reaches a value of about 200 mV.

Add rapidly about 0,2 g of the urea (4.1), then 10 ml of the phosphoric acid (4.3). Wait for about 5 min for the potential to stabilize.

**7.4.5** Titrate with the ammonium iron(II) sulphate solution (4.8), using a 50 ml burette, until the maximum fall of the potential is observed (about 100 mV).

## 8 Expression of results

The vanadium content, expressed as a percentage by mass of the sample, is given by the formula

$$T(V_0 - V_2) \times C \times \frac{100}{m}$$

where

$T$ is the constant 0,010 19; i.e. the mass of vanadium, in grams, equivalent to 1 ml of exactly 0,2 mol/l ammonium iron(II) sulphate solution;

$V_0$ is the volume, in millilitres, of the ammonium iron(II) sulphate solution (4.8) used for the determination (7.4.5);

$V_2$ is the volume, in millilitres, used for the blank test (7.2);

$C$ is the correction factor of the ammonium iron(II) sulphate solution (4.8.2);

$m$ is the mass, in grams, of the test portion (7.1).

## 9 Repeatability

Experience has shown that the 95 % confidence limits for an experienced operator are ± 0,20 %.

## 10 Test report

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.