
**Ferrovandium — Determination of
vanadium content — Potentiometric
method**

*Ferrovandium — Dosage du vanadium — Méthode
potentiométrique*

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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, *Ferrous alloys*.

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.

Ferrovandium — Determination of vanadium content — Potentiometric method

1 Scope

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

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

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, *Ferrovandium — 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 ($FeSO_4(NH_4)_2SO_4$) \approx 0,2 mol/l.

5.8.1 Preparation

In a 1 000 ml volumetric flask, dissolve 78,4 g of ammonium iron(II) sulphate ($FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$) 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.

6.2 Precision potentiometer, equipped with the following electrodes.

Indicating electrode: platinum.

Reference electrode: Hg/Hg₂SO₄/K₂SO₄ (saturated) or Ag/AgCl/KCl (saturated).

6.3 Magnetic stirrer.

7 Sample

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

8 Procedure

8.1 Test portion

Take a test portion of 0,500 0 g ± 0,000 1 g.

8.2 Blank test

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

8.3 Determination

8.3.1 In a 400 ml beaker, dissolve the test portion with 10 ml of water, 10 ml of the nitric acid (5.2) and 50 ml of the sulfuric acid (5.4); evaporate to white sulfuric fumes then allow to cool.

8.3.2 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.

8.3.3 Place the beaker on the magnetic stirrer, immerse the electrodes, and stir. Add the potassium permanganate solution (5.6), using a 50 ml burette, until the colour changes from green to orange-red, then add another 0,5 ml permanganate solution (5.6), wait for 15 min.

8.3.4 Reduce the excess of potassium permanganate with the potassium nitrite solution (5.5), added dropwise every 30 s. Stop the addition when there is a drop of about 200 mv in the potential and the solution becomes orange-red in colour. Add another 0,5 ml potassium nitrite solution (5.5) in excess.

Add rapidly about 0,2 g of the urea (5.1), then 10 ml of the phosphoric acid (5.3). Wait for about 5 min for the potential to stabilize.

8.3.5 Titrate with the ammonium iron (II) sulphate solution (5.8), using a 50 ml burette, until the end-point of titration is observed by the change of electric potential.

9 Expression of results

The vanadium content (w_v), expressed as a percentage by mass of the sample, is given by [Formula \(2\)](#).

$$w_v = \frac{(V_4 - V_3) \times C_2 \times 50,94}{m \times 1000} \times 100 \quad (2)$$

where

50,94 is a multiple of the atomic mass of vanadium, in grams per mole;

V_4 is the volume, in millilitres, of the ammonium iron(II) sulphate solution (5.8) used for the determination (8.3.5);

V_3 is the volume, in millilitres, used for the blank test (8.2);

C_2 is the concentration, in moles per litre, of the ammonium iron(II) sulphate solution (5.8.2);

m is the mass, in grams, of the test portion (8.1).

10 Precision

10.1 Expression of precision

A planned trial of this method was carried out by eight laboratories at five levels of vanadium, each laboratory making three determinations for each level.

The results obtained were treated statistically in accordance with ISO 5725-2(1).

The data show a linear relationship between the vanadium content and the repeatability of the results and a logarithmic relationship between the vanadium content and the reproducibility, which is summarised in Table 1. The original data is given in Annex A and a graphical representation of the precision data in Annex B.

Table 1 — Repeatability limit and reproducibility limit

Vanadium content % (mass fraction)	Repeatability limit r	Reproducibility limit R
47,20	0,175	0,478
50,28	0,196	0,490
53,92	0,206	0,484
78,04	0,305	0,624
81,08	0,313	0,520

The difference between two single and independent results found on identical test material by one analyst using the same apparatus within a short time interval in the normal and correct operation of the test method will exceed the repeatability, r , on average, in only one case in twenty.

The difference between two single and independent results found by two operators working in different laboratories on identical test material in the normal and correct operation of the test method will exceed the reproducibility, R , on average, in only one case in twenty.

10.2 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operation specified in Annex C.

11 Test report

This clause specifies which information is to be included in the test report:

- the sample;
- the document used (including its year of publication), i.e. ISO 6467:2018;
- the method used (if the standard includes several);

- d) the result(s), including a reference to the clause which explains how the results were calculated;
- e) any deviations from the procedure;
- f) any unusual features observed;
- g) the date of the test.

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Annex A (informative)

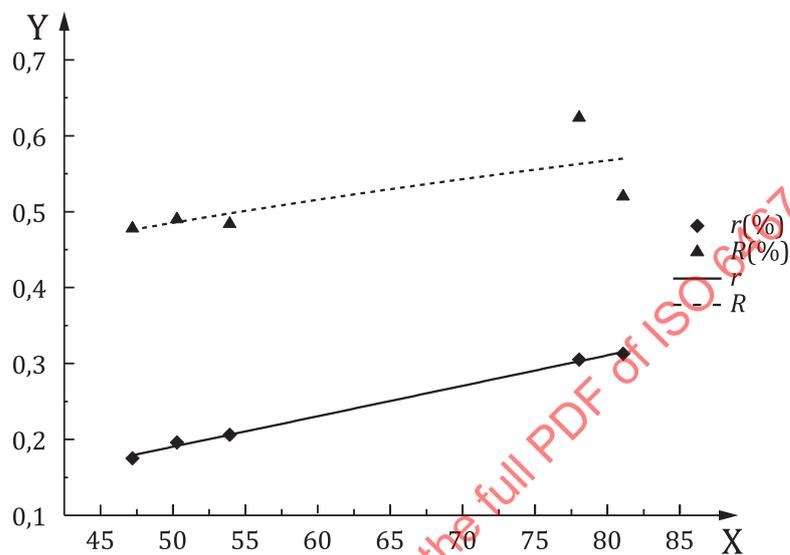
Original data from the verification test

Table A.1 — Validation data

Laboratory	Vanadium content, % (mass fraction)				
	V-1	V-2	V-3	V-4	V-5
Lab 1	47,384	50,339	54,102	77,928	81,257
	47,384	50,359	54,236	78,124	81,061
	47,467	50,516	54,334	78,222	81,110
Lab 2	47,26	50,15	53,82	77,68	81,06
	47,24	50,22	53,81	77,69	81,11
	47,23	50,40	53,95	78,05	81,18
Lab 3	46,951	50,669	54,031	78,371	80,975
	46,927	50,432	53,936	78,418	81,093
	46,880	50,550	53,818	78,323	81,212
Lab 4	47,17	50,01	53,67	77,93	80,76
	47,12	50,09	53,73	77,88	80,93
	46,96	50,19	53,74	77,78	80,92
Lab 5	47,149	50,165	53,905	78,206	81,106
	47,149	50,199	53,793	78,120	81,217
	47,162	50,176	53,889	77,907	81,136
Lab 6	47,34	50,30	53,99	78,20	81,19
	47,30	50,30	54,03	78,21	81,39
	47,27	50,32	54,07	78,22	81,14
Lab 7	47,34	50,11	53,78	77,99	81,48
	47,31	50,04	53,70	77,83	81,15
	47,44	50,02	53,82	77,59	81,10
Lab 8	47,112	50,452	54,103	78,124	80,745
	47,015	50,334	53,956	78,132	80,812
	47,213	50,324	53,942	78,143	80,856

Annex B (informative)

Graphical representation of the precision data



Key

X vanadium content, % (mass fraction)

Y precision, % (mass fraction)

r $0,004\ 00\ m - 0,009\ 8$

$\lg R$ $0,332\ 6\ \lg m - 0,879$

Figure B.1 — Relationship between vanadium content and repeatability limit and reproducibility limit