



SLOVENSKI STANDARD
oSIST prEN ISO 4947:2019

01-oktober-2019

**Jeklo in lito železo - Določanje vanadija - Potenciometrijska metoda titracije
(ISO/DIS 4947:2019)**

Steel and cast iron - Determination of vanadium content - Potentiometric titration method
(ISO/DIS 4947:2019)

Stahl und Gusseisen - Bestimmung des Vanadium-Gehaltes - Potentiometrisches
Titrierverfahren (ISO/DIS 4947:2019)

Aciers et fontes - Dosage du vanadium - Méthode par titrage potentiométrique (ISO/DIS
4947:2019)

Ta slovenski standard je istoveten z: prEN ISO 4947

ICS:

77.080.01 Železne kovine na splošno Ferrous metals in general

oSIST prEN ISO 4947:2019

en,fr,de

DRAFT INTERNATIONAL STANDARD

ISO/DIS 4947

ISO/TC 17/SC 1

Secretariat: JISC

Voting begins on:
2019-08-16Voting terminates on:
2019-11-08

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

Aciers et fontes — Dosage du vanadium — Méthode par titrage potentiométrique

ICS: 77.080.01

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN ISO 4947:2020](https://standards.iteh.ai/catalog/standards/sist/0fb0c85c-5541-4c59-927d-192e9636a186/sist-en-iso-4947-2020)

<https://standards.iteh.ai/catalog/standards/sist/0fb0c85c-5541-4c59-927d-192e9636a186/sist-en-iso-4947-2020>

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING



Reference number
ISO/DIS 4947:2019(E)

© ISO 2019

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 4947:2020

<https://standards.iteh.ai/catalog/standards/sist/0fb0c85c-5541-4c59-927d-192e9636a186/sist-en-iso-4947-2020>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2019

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	1
5 Apparatus	3
6 Sampling	3
7 Procedure	3
7.1 Test Portion	3
7.2 Blank test	3
7.3 Determination	4
7.3.1 Preparation of the test solution	4
7.3.2 Oxidation of chromium and vanadium	4
7.3.3 Preparation of the solution for measurement	4
7.3.4 Oxidation of vanadium	5
7.4 Titration	5
8 Expression of results	5
8.1 Method of calculation	5
8.2 Precision	6
9 Test report	6
Annex A (informative) Additional information on the international interlaboratory test	7
Annex B (informative) Graphical representation of precision data	8
Bibliography	9

ISO/DIS 4947:2019(E)**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. 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. 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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

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 revised in order to re-assess the precision data.

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

1 Scope

This International Standard 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) to 2 % (mass fraction).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

SIST EN ISO 4947:2020

[https://standards.iteh.ai/catalog/standards/sist/0fb0c85c-5541-4c59-927d-](https://standards.iteh.ai/catalog/standards/sist/0fb0c85c-5541-4c59-927d-192e9636a186/sist-en-iso-4947-2020)

[192e9636a186/sist-en-iso-4947-2020](https://standards.iteh.ai/catalog/standards/sist/0fb0c85c-5541-4c59-927d-192e9636a186/sist-en-iso-4947-2020)

3 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) sulphate;
- 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.

4 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.

4.1 Potassium peroxydisulfate ($K_2S_2O_8$).

4.2 Hydrochloric acid, $\rho = 1,19$ g/ml approximately.

4.3 Nitrite acid, $\rho = 1,40$ g/ml approximately.

ISO/DIS 4947:2019(E)

4.4 Hydrofluoric acid, $\rho = 1,15$ g/ml approximately.

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

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

4.7 Orthophosphoric acid, $\rho = 1,70$ g/ml approximately.

4.8 Ammonium iron(II) sulfate $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ 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, $\rho 1,84$ g/ml approximately, cool, make up the volume to 1 000 ml with water and mix.

4.9 Potassium permanganate, 5 g/l.

4.10 Sodium nitrite, 3 g/l.

4.11 Sulfamic acid ($\text{NH}_2\text{SO}_3\text{H}$), 100 g/l.

This solution is stable for only one week.

4.12 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 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 (4.5). Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, cool, dilute to the mark with water and mix.

4.13 Ammonium iron(II) sulfate $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$, standard solution.

1 ml of this solution corresponds to approximately 1,275 mg of vanadium.

4.13.1 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 dilute to the mark with water and mix.

4.13.2 Standardization of the solution (to be carried out just before use)

Take 25,0 ml of the potassium dichromate standard solution (4.12) and titrate potentiometrically with the ammonium iron(II) sulfate solution (4.13.1). The corresponding concentration (ρ) of the ammonium iron(II) sulfate standard solution (4.13) is given by the formula :

$$\rho = \frac{0,025 \times m_0}{49,03 \times V} \times 50,94$$

where

- ρ is the corresponding concentration of the ammonium iron(II) sulfate standard solution (4.13) expressed in milligrams of vanadium per millilitre;
- m_0 is the mass, in milligrams, of the weighted potassium dichromate;
- V is the volume, in millilitres, of the ammonium iron(II) sulphate standard solution (4.13) used for the titration;
- 0,025 is the ratio between the volume taken and the total volume of the potassium dichromate standard solution;
- 49,03 is the molar mass of potassium dichromate divided by 6;
- 50,94 is the molar mass of vanadium.

5 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385, ISO 648 or ISO 1042 as appropriate. Ordinary laboratory apparatus and

5.1 Potentiometric titration device which permits a difference in potential to be measured with platinum-saturated calomel electrodes or platinum-Ag/AgCl electrodes.

6 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steels and cast irons.

7 Procedure

7.1 Test Portion

According to the presumed vanadium content, weigh, to the nearest 0,001 g, the following mass of the test portion.

- for vanadium contents between 0,04 % (mass fraction) and 0,25 % (mass fraction): approximately 5 g;
- for vanadium contents between 0,25 % (mass fraction) and 1 % (mass fraction): approximately 2 g;
- for vanadium contents between 1 % (mass fraction) and 2 % (mass fraction): approximately 1 g;

The quantity of vanadium in the test portion may vary between 2 mg and 20 mg.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

ISO/DIS 4947:2019(E)

7.3 Determination

7.3.1 Preparation of the test solution

7.3.1.1 Samples soluble in sulfuric acid

Introduce the test portion (7.1) into a 400 ml beaker (see notes 1 and 2) and add 40 ml of sulfuric acid (4.5) for a 1 g or 2 g test portion. For a 5 g test portion, add 70 ml of sulfuric acid (4.5). Cover the beaker with a watch-glass and heat gently until effervescence ceases.

For samples with high silicon contents, add 5 to 10 drops of hydrofluoric acid (4.4). Bring to the boil to ensure complete cessation of effervescence.

If the test portion contains tungsten, add a sufficient quantity of hydrofluoric acid (4.4) to keep it in solution. In general 5 ml of hydrofluoric acid (4.4) will suffice for a 1 g test portion, 7 ml for a 2 g test portion, and 13 ml for a 5 g test portion.

Dilute the solution to a volume between 90 and 100 ml with water and proceed as described in 7.3.2.

NOTE 1 If hydrofluoric acid (4.4) is used, a fused silica beaker and a fused silica watch-glass should be employed.

NOTE 2 The glassware used should not contain more than 0,05 % (mass fraction) of arsenic.

NOTE 3 If the glass contains barium, a barium sulfate precipitate may form; however, this will not adversely affect the determination.

7.3.1.2 Samples not readily soluble in sulfuric acid

For test portions difficult to dissolve in the sulfuric acid medium, the initial dissolution may be carried out with a mixture of nitric acid (4.3) and hydrochloric acid (4.2).

After dissolution, add the quantity of sulfuric acid (4.5) specified in 7.3.1.1 and heat until white fumes are given off.

Add water and heat to dissolve the salts. Repeat the evaporation to white fumes to ensure that the nitric acid is completely eliminated.

If the test portion contains tungsten, add a sufficient quantity of hydrofluoric acid (4.4) to keep it in solution. In general 5 ml of hydrofluoric acid (4.4) will suffice for a 1 g test portion, 7 ml for a 2 g test portion, and 13 ml for a 5 g test portion.

Dilute the solution to a volume between 90 and 100 ml with water and proceed as described in 7.3.2.

7.3.2 Oxidation of chromium and vanadium

Cool the solution (7.3.1) to approximately 50 °C. Add 3 g of potassium peroxydisulfate (4.1) per gram of test portion, heat slowly to boiling and boil for at least 10 min.

7.3.3 Preparation of the solution for measurement

7.3.3.1 Control of the oxidation of chromium and vanadium

Cool the solution (7.3.2) to room temperature. If graphite is present in the solution, filter the solution through a cellulose pulp lined coarse filter paper and rinse with sulfuric acid (4.6), adding several drops of hydrofluoric acid (4.4) to facilitate filtering. Make up the final volume of the solution to approximately 150 ml.

Introduce the electrodes of the potentiometric device (5.1) into the beaker and agitate the solution, preferably with an electromagnetic stirrer. The potential should be above 770 mV.