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Stål och gjutjärn — Bestämning av vanadin- halt — Potentiometrisk titreringsmetod

Orientering

Denna standard utgörs av den engelska versionen av den europeiska standarden EN 24 947:1989, vilken ikraftsätter ISO 4947:1986. Som svensk bilaga NA återges ISO 4947.

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

Introduction

This Swedish standard consists of the European standard EN 24 947:1989, which endorses ISO 4947:1986. ISO 4947 is reprinted in the national Swedish annex NA.

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English version

**Steel and cast iron — Determination of vanadium content —
Potentiometric titration method
(ISO 4947:1986)**

Acier et fonte — Détermination de
la teneur en vanadium — Méthode
de titration potentiométrique (ISO
4947:1986)

Stahl- und Gusseisen — Bestimmung
des Vanadium-Gehaltes — Potentio-
metrisches Titrierverfahren (ISO
4947:1986)

This European Standard was approved by CEN on 1991-01-30 and is identical to the ISO standard as referred to.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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FOREWORD

On the proposal of the Technical Committee ECISS/TC 20 "Chemical Analysis" the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization (ECISS) decided in January 1991 to submit the International Standard

ISO 4947:1986 Steel and cast iron – Determination of vanadium content – Potentiometric titration method

to Formal Vote.

This European Standard was adopted by CEN on 1991-01-30.

According to the Common CEN/CENELEC Rules, being part of the Internal Regulation of CEN, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

STATEMENT

The text of the International Standard ISO 4947:1986, edition 1, was approved by CEN as a European Standard without any modification.

BILAGA NA

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

1 Scope and field of application

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 and 2 % (*m/m*).

2 Reference

ISO 377, *Wrought steel — Selection and preparation of samples and test pieces*.

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) sulfate;

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

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

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from reducing or oxidizing activity.

4.1 Potassium peroxydisulfate ($K_2S_2O_8$).

4.2 Hydrochloric acid, ρ about 1,19 g/ml.

4.3 Nitric acid, ρ about 1,40 g/ml.

4.4 Hydrofluoric acid, ρ about 1,15 g/ml.

4.5 Sulfuric acid, ρ about 1,84 g/ml, diluted 1 + 4.

4.6 Sulfuric acid, ρ about 1,84 g/ml, diluted 1 + 50.

4.7 Orthophosphoric acid, ρ about 1,70 g/ml.

4.8 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, ρ about 1,84 g/ml, cool, make up the volume to 1 000 ml and mix.

4.9 Potassium permanganate, 5 g/l solution.

4.10 Sodium nitrite, 3 g/l solution.

4.11 Sulfamic acid (NH_2SO_3H), 100 g/l solution.

This solution is stable for only one week.

4.12 Potassium dichromate, standard reference solution.

Weigh, to the nearest 0,001 g, approximately 1 g of potassium dichromate (the highest purity grade) previously dried at 150 °C until a constant mass is obtained on cooling in the desiccator. Introduce 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 the mark with water and mix.

4.13 Ammonium iron(II) sulfate $[Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O]$, standard solution,

1 ml of this solution corresponds to approximately 1,275 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, ρ about 1,84 g/ml, make up the volume to 1 000 ml 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 reference solution (4.12) and titrate potentiometrically with the ammonium iron(II) sulfate solution (4.13.1). The corresponding concentration (c) of the ammonium iron(II) sulfate standard solution (4.13) is given by the formula:

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

$$= \frac{m_0}{V} \times 0,026$$

where

c 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 weighed potassium dichromate;

V is the volume, in millilitres, of the ammonium iron(II) sulfate standard solution (4.13) used for titration;

0,025 is the ratio between the volume taken and the total volume of the potassium dichromate standard reference solution;

49,03 is the relative molecular mass of potassium dichromate divided by 6;

50,94 is the relative atomic mass of vanadium.

5 Apparatus

Ordinary laboratory apparatus and

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

6 Sampling

Carry out sampling in accordance with ISO 377 or appropriate national standards for cast iron.

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:

- a) for vanadium contents between 0,04 and 0,25 % (m/m): approximately 5 g;

b) for vanadium contents between 0,25 and 1 % (m/m): approximately 2 g;

c) for vanadium contents between 1 and 2 % (m/m): approximately 1 g.

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

7.2 Blank test

Carry out a blank test in parallel with the determination, by the same procedure and using the same quantities of all reagents.

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 between 90 and 100 ml with water and proceed as in 7.3.2.

NOTES

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

2 The glassware used should not contain more than 0,05 % (m/m) of arsenic.

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 accomplished with a mixture of nitric acid (4.3) and hydrochloric acid (4.2).

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

Take up again with water, heating to dissolve the salts, then 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.