

JIS

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS G 1318 : 1998

Method for chemical analysis of ferrovanadium

ICS 77.100

Descriptors : ferrovanadium, ferro-alloys, vanadium-containing alloys, chemical analysis and testing, testing, determination of content, vanadium, carbon, silicon, sulphur, phosphorus, aluminium

Reference number : JIS G 1318 : 1998 (E)

G 1318 : 1998

Foreword

This translation has been based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently, **JIS G 1318:1982** has been replaced with **JIS G 1318:1998**.

In this revision, the relevant provisions of ISO Standard are described as Annex 2, and also the determination of each element is shown in the form of Annex, for the purpose of conformity with the corresponding International Standard.

Date of Establishment: 1953-03-28

Date of Revision: 1998-02-20

Date of Public Notice in Official Gazette: 1998-02-20

Investigated by: Japanese Industrial Standards Committee

Divisional Council on Iron and Steel

JIS G 1318:1998, First English edition published in 1999-03

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 1999

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

Method for chemical analysis of ferrovanadium

Introduction In the application of standard, normative references to the standard shall be simultaneously referred to. Since ferrovanadium has many chances to be used as the supplementary raw material in iron and steel making process, **JIS G 1318** which specifies the determination of vanadium as main element and other 5 elements including carbon which give rise to discussion in the iron and steel production, was established in 1953.

Annex 2 is the Japanese Industrial Standard prepared based on **ISO 6467, Ferrovanadium — Determination of vanadium content — Potentiometric method**, published in 1980 as the first edition, without modifying its technical content and structure.

The Informative references underlined with dots in Annex 2 show the matters not given in the original International Standard.

1 Scope This Japanese Industrial Standard specifies determination of vanadium, carbon, silicon, phosphorus, sulfur and aluminium in ferrovanadium.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS G 1301 *General rules for chemical analysis of ferroalloys*

JIS K 0121 *General rules for atomic absorption spectrochemical analysis*

JIS K 8001 *General rule for test methods of reagents*

JIS Z 2615 *General rules for determination of carbon in metallic materials*

JIS Z 2616 *General rules for determination of sulfur in metallic materials*

3 General matters General matters common to the methods for chemical analysis shall be in accordance with **JIS G 1301** and **JIS K 0121**. However, these shall not be applied to Annex 2.

4 Classification for determination of each component The methods for determination of each component in ferrovanadium shall be according to Table 1.

**Table 1 The methods for determination of
each component in ferrovanadium**

Component	Determination method	Range of percentage of components % (m/m)	Annex (normative) No.
Vanadium	Potassium permanganate oxidation ferrous (II) ammonium sulfate visual titrimetric method	30 or over up to and incl. 90	1
	Potassium permanganate oxidation ferrous (II) ammonium sulfate potentiometric titration method (ISO 6467)	not more than 85	2
Carbon	Combustion-gas volumetric method	0.05 or over up to and incl. 0.30	3
	Combustion-infrared absorption method	0.001 or over up to and incl. 0.30	4
Silicon	Silicon dioxide gravimetric method	0.1 or over up to and incl. 3.0	5
Phosphorus	Iron and vanadium separation Phosphomolybdenum blue spectrophotometric method	0.01 or over up to and incl. 0.10	6
Sulfur	Combustion-sodium hydroxide titrimetric method	0.005 or over up to and incl. 0.10	7
	Combustion-infrared absorption method	0.001 or over up to and incl. 0.10	8
Aluminium	Disodium dihydrogen ethylene- diamine tetraacetate titrimetric method	0.1 or over up to and incl. 5.0	9
	Atomic absorption method	0.005 or over up to and incl. 1.0	10

**Annex 1 (normative) Determination of vanadium —
Potassium permanganate oxidation
ferrous (II) ammonium sulfate visual
titrimetric method**

1 Summary Decompose a sample with nitric acid and hydrochloric acid. Add sulfuric acid and phosphoric acid, heat to generate white fumes for removing hydrochloric acid and nitric acid. Oxidize vanadium with potassium permanganate, decompose the excess amounts of permanganate with sodium nitrite and decompose the excess amounts of nitrous acid with urea. After suppressing the interferences of generated dichromic acid with arsenious acid, the sample solution is titrated with ferrous (II) ammonium sulfate standard solution by using diphenylamine as the indicator.

2 Reagents The reagents shall be as follows :

- a) **Hydrochloric acid**
- b) **Nitric acid**
- c) **Mixed acid** (3 parts of sulfuric acid, 13 parts of phosphoric acid, and 14 parts of water)
- d) **Sodium nitrite solution** (30 g/l)
- e) **Potassium permanganate solution** (3 g/l) This solution shall be stored in the brown color bottle.
- f) **Arsenious acid solution** Dissolve 3 g of sodium carbonate in approx. 150 ml of water, dissolve 0.85 g of diarsenic trioxide and dilute to approx. 950 ml with water. After saturating the solution by passing carbon dioxide for approx. 3 min, dilute to 1 000 ml with water.

WARNING FOR SAFETY Because arsenious acid solution is a deadly poison, it is necessary to pay attention when its solution and a solution with arsenious acid added are treated.

- g) **Urea**
- h) **0.1 mol/l ferrous (II) ammonium sulfate standard solution** The method of preparation and standardization of this solution shall be in accordance with 4.5 (27) in JIS K 8001.
- i) **Diphenylamine solution** Dissolve 1.0 g of diphenylamine in 100 ml of phosphoric acid.

3 Test portion Weigh out 0.20 g of a sample, to the nearest 0.1 mg.

4 Operation

4.1 Preparation of sample solution Preparation of sample solution shall be according to the following procedures.

- a) Weigh out the sample, transfer it into an Erlenmeyer flask (500 ml) and insert a funnel into the inlet of the flask.
- b) Add 5 ml of nitric acid and 15 ml of hydrochloric acid. Decompose the sample by gently heating at approx. 60 °C.
- c) Wash the inner and outer sides of leg of the funnel with water and remove it.
- d) Add 30 ml of mixed acid [2 c)], heat it until white fumes start to be generated, and stand it to cool.
- e) Add approx. 150 ml of water, dissolve the soluble salts by heating, and cool it.

4.2 Oxidation of vanadium Oxidation of vanadium shall be according to the following procedures.

- a) Drop potassium permanganate solution [2 e)] while stirring the solution obtained in 4.1 e) and allow the pale red color of the solution to develop. Add further 0.5 ml of potassium permanganate solution in excess, mix them and allow to stand for approx. 10 min.
- b) Add 5 g of urea to this solution and dissolve it. Add drop by drop sodium nitrite solution, while stirring the solution, until the red color completely disappears.
- c) After stirring the solution until the fine bubbles caused by the decomposition of nitrous acid disappears, add 5 ml of arsenious acid solution [2 f)] ⁽¹⁾ and stir them for about 1 min.

Note ⁽¹⁾ When chromium does not coexist, arsenious acid solution may not be added.

4.3 Titration Titration shall be according to the following procedures.

- a) Dilute the solution obtained in 4.2 c) to approx. 200 ml with water.
- b) Add 0.05 ml of diphenylamine solution [2 i)] as the indicator and mix them by shaking. Thereafter, allow to stand for 2 min to 3 min to develop purple color.
- c) Titrate this purple solution with 0.1 mol/l ferrous (II) ammonium sulfate standard solution [2 h)], and allow the point where the purple color disappears to be the end point. Obtain the titration amount of 0.1 mol/l ferrous (II) ammonium sulfate standard solution used.

5 Blank Test Blank tests shall not be carried out.

6 Calculation The vanadium content in the sample shall be calculated with the following formula.

$$V = \frac{A \times F \times 0.005\,094}{m} \times 100$$

- where,
- V : vanadium contents in the sample [% (m/m)]
 - A : amount of 0.1 mol/l ferrous (II) ammonium sulfate standard solution used in 4.3 c) (ml)
 - F : factor of 0.1 mol/l ferrous (II) ammonium sulfate standard solution
 - m : test portion of the sample (g)

**Annex 2 (normative) Determination of vanadium —
Potassium permanganate oxidation
ferrous (II) ammonium sulfate
potentiometric method (ISO 6467)**

1 Scope

This Annex 2 specifies a potentiometric method for determination of vanadium in ferrovanadium.

This method shall be applicable to ferrovanadium having a vanadium contents not more than 85 % (m/m).

2 Normative references

ISO 3713 *Ferroalloys — Sampling and preparation of samples — General rules* [foot note (1)]

Informative reference : In the original Standard, "At present at the stage of draft" is described in foot note (1). However, this standard was published in 1987.

3 Principle

Decompose a sample with nitric and sulfuric acids. In the cold solution, oxidize vanadium (IV) into vanadium (V) with slight excess of potassium permanganate. Decompose the excess of potassium permanganate by potassium nitrite and decompose the excess of potassium nitrite by urea. Reduce vanadium (V) into vanadium (IV) with iron (II) and obtain vanadium contents with the 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 ρ 1.38 g/ml to 1.42 g/ml

4.3 Phosphoric acid

4.4 Sulfuric 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 sulfuric acid (ρ approximately 1.84 g/ml); mix, cool, dilute to 1 000 ml and mix.

4.5 Potassium nitrite solution (10 g/l)

Dissolve 10 g of potassium nitrite in water and dilute the volume to 1 000 ml.

4.6 Potassium permanganate solution (6.3 g/l)

Dissolve 6.3 g of potassium permanganate in water and dilute the volume to 1 000 ml.

Informative reference : This solution shall be stored in a brown bottle.

4.7 Potassium dichromate standard solution $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0.2 \text{ mol/l}$

Weigh to the nearest 0.000 5 g, exactly 9.806 4 g of potassium dichromate dried at 105 °C previously, dissolve it in water, transfer the solution to a volumetric flask of 1 000 ml and dilute the volume to the mark with water.

4.8 Ammonium iron (II) sulfate standard solution $c[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4] = 0.2 \text{ mol/l}$ **4.8.1 Preparation**

Dissolve 78.4 g of ammonium iron (II) sulfate $[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ into 500 ml of warm water and transfer it into a volumetric flask of 1 000 ml. After dissolving it thoroughly, add 100 ml of sulfuric acid (4.4), cool, and dilute to the mark with water.

4.8.2 Standardization

Put 280 ml of water into a beaker (600 ml), add 10 ml of sulfuric acid (4.4) and 10 ml of phosphoric acid (4.3). Introduce 40 ml of potassium dichromate solution (4.7).

Titrate the solution potentiometrically with ammonium iron (II) sulfate solution.

The end of the reaction is obtained when the maximum fall in potential of at least 100 mV is observed. This is taken as the end point of the titration.

The correction factor C is given by the following expression :

$$C = 40 / V_1$$

where, V_1 : amounts of ammonium iron (II) sulfate solution used (ml)

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

5 Apparatus and equipments

The following equipments shall be also used as well as the apparatus and equipments in general use at laboratory.

5.1 Beaker (400 ml)**5.2 Precision potentiometer****5.2.1 Electrode**

Indicator electrode : platinum

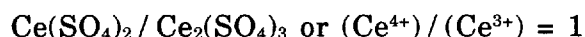
Reference electrode : $\text{Hg}/\text{Hg}_2\text{SO}_4/\text{K}_2\text{SO}_4$ (saturated)

5.2.2 Checking of measuring system

When the apparatus is not used, the reference electrode should be immersed in the saturated potassium sulfate solution.

The platinum electrode should be checked with stable oxidation-reduction buffer solution.

For example, Ce (III) and Ce (IV) salt solution is used.



This buffer solution is prepared by dissolving 0.330 g of $\text{Ce}(\text{SO}_4)_2$ and 0.280 g of $\text{Ce}_2(\text{SO}_4)_3$ into water. Transfer this solution into a volumetric flask of 1 000 ml, add sulfuric acid in order to obtain the equivalent mol concentration of sulfuric acid in practical operation. If the response of the platinum electrode is satisfactory, when it is coupled with the reference electrode, the millivoltmeter shows a reading of between 710 mV 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 $(0.5 \pm 0.000\ 2)$ g.

7.2 Blank test

Carry out a blank test without the sample, 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 Weigh out the sample and transfer into a beaker (400 ml), dissolve the test portion with 10 ml of water, 10 ml of the nitric acid (4.2) and 50 ml of the sulfuric acid (4.4), evaporate to generate white sulfuric fumes and 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 approx. 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 burette of 50 ml, until the maximum potential is reached. Then wait 15 min. (The potential is stabilized at approx. 700 mV $\pm$ 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 approx. 0.2 g of the urea (4.1), and then 10 ml of the phosphoric acid (4.3). Wait for approx. 5 min for the potential to stabilize.

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

8 Expression of results

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

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

- where,
- T : the constant 0.010 19, i.e. the mass of vanadium, equivalent to 1 ml of exactly 0.2 mol/l ammonium iron (II) sulfate solution (g).
 - V_0 : the volume of the ammonium iron (II) sulfate solution (4.8) used for the determination (7.4.5) (ml)
 - V_2 : the volume of the ammonium iron (II) sulfate solution (4.8) used for the blank test (7.2) (ml)
 - C : the correction factor of the ammonium iron (II) sulfate solution (4.8.2)
 - m : the mass of the test portion (7.1) (g)

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 Annex 2 or regarded as optional.