

JIS

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS G 1319 : 2000

Methods for chemical analysis of ferrotitanium

ICS 77.100

Descriptors : ferrotitanium, ferro-alloys, titanium, refractory metals, transition metals,
chemical analysis and testing, testing

Reference number : JIS G 1319 : 2000 (E)

G 1319 : 2000

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS G 1319:1982** is replaced with **JIS G 1319:2000**.

In this revision, Annex 2 has been prepared based on the ISO Standard in order to conform to the International Standard.

Date of Establishment: 1953-03-28

Date of Revision: 2000-03-20

Date of Public Notice in Official Gazette: 2000-03-21

Investigated by: Japanese Industrial Standards Committee
Divisional Council on Iron and Steel

JIS G 1319:2000 First English edition published in 2001-05

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

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Printed in Japan

Methods for chemical analysis of ferrotitanium

Introduction This Japanese Industrial Standard has been prepared based on **ISO 7692 Ferrotitanium — Determination of titanium content — Titrimetric method** published in 1983 as Annex 2 (normative), without any modification in technical contents and by supplementing the determination methods, which are not specified in the corresponding International Standard, as Annexes 1, 3, 4, 5, 6, 7 and 8.

1 Scope This Japanese Industrial Standard specifies determination methods of titanium, carbon, silicon, manganese, phosphorus and sulfur, which are contained in ferrotitanium.

Remarks: The International Standard corresponding to this Standard is as follows.

ISO 7692 : 1983 *Ferrotitanium — Determination of titanium content — Titrimetric method*

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 G 2309 *Ferrotitanium*

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

JIS K 8005 *Reference materials for volumetric analysis*

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 matter The general matter commonly used in determination methods is specified in **JIS G 1301**. Provided that **JIS G 1301** shall not be applicable to Annex 2 (normative).

4 Classification of determination methods The classification of determination methods shall be specified in Table 1.

Table 1 Classification of determination methods

Component to be determined	Determination method	Applicable range of contents % (m/m)	Annex (normative) No.
Titanium	Aluminium reduction ammonium iron(III) sulfate titrimetric method (1)	20 or over up to and incl. 75	1
	Aluminium reduction ammonium iron(III) sulfate titrimetric method (2) (ISO 7692)	20 or over up to and incl. 75	2
Carbon	Combustion-infrared ray absorption method	0.001 or over up to and incl. 0.40	3
Silicon	Silicon dioxide gravimetric method	0.10 or over up to and incl. 10	4
	Titanium separation molybdosilicic acid blue absorptiometry	0.01 or over up to and incl. 0.10	5
Manganese	Permanganic acid absorptiometry	0.05 or over up to and incl. 2.0	6
Phosphorus	Titanium separation molybdophosphoric acid blue absorptiometry	0.005 or over up to and incl. 0.05	7
Sulfur	Combustion-infrared ray absorption method	0.001 or over up to and incl. 0.10	8

**Annex 1 (normative) Determination of titanium content —
Aluminium reduction ammonium iron(III)
sulfate titrimetric method (1)**

1 Summary Decompose sample with sulfuric acid and hydrochloric acid, oxidize with nitric acid, and heat it until white fume of sulfuric acid emits. Reduce titanium by aluminium, and titrate it with ammonium iron(III) sulfate standard solution using ammonium thiocyanate as indicator.

2 Reagents Reagents shall be as follows.

- a) **Hydrochloric acid**
- b) **Hydrochloric acid (1+1, 1+4)**
- c) **Nitric acid**
- d) **Hydrofluoric acid**
- e) **Sulfuric acid**
- f) **Sulfuric acid (1+1)**
- g) **Sodium hydroxide solution (200 g/l)**
- h) **Ammonium thiocyanate solution (100 g/l)**
- i) **Sodium hydrogencarbonate solution (saturated, approx. 100 g/l)**
- j) **Sodium carbonate solution (20 g/l)**
- k) **Potassium disulfate**
- l) **Aluminium** Purity is 99 % (m/m) or more, and content of titanium and copper is 0.01 % (m/m) or less. A cube, bar, or wire type aluminium, about 1 g, shall be washed before being used with hydrochloric acid (1+5).
- m) **1/30 mol/l ammonium iron(III) sulfate standard solution** Dissolve 16 g of ammonium iron(III) sulfate dodecahydrate in about 300 ml of water which contains 10 ml of hydrochloric acid (1+1), transfer it into a 1 000 ml volumetric flask with rinsing it with water, add water up to the marked line. The factor of this solution shall be obtained by the following procedures.
 - 1) **Preparation and standardization of 1/30 mol/l sodium thiosulfate standard solution**
 - 1.1) **Preparation** Weigh out 8.3 g of sodium thiosulfate pentahydrate and 0.2 g of sodium carbonate, dissolve them in 1 000 ml of dissolved oxygen-free water [follow 3.6 (4) of JIS K 8001], store it in an air-tight vessel, and allow it to stand for 2 days.
 - 1.2) **Method for standardization**
 - 1.2.1) Dry the potassium iodate, specified in 2 (11) of JIS K 8005, under the drying condition specified in 4 Table 1 of JIS K 8005, allow it to cool in a desiccator, weigh out its 1.0 to 1.3 g to the nearest 0.1 mg, transfer it into a 1 000 ml volumetric flask, add water to dissolve it, and add water up to the marked line. Take accurately 25 ml of this solution into an Erlenmeyer flask with an interchangeable ground joint (200 ml).

- 1.2.2) Add 2 g of potassium iodide and 2 ml of sulfuric acid (1+1) in it, immediately put a stopper, and swirl gently it, allow it to stand in dark place for 5 min, titrate it with 1/30 mol/l sodium thiosulfate solution prepared at 1.1), when the solution gives faint yellow, add 0.5 ml of starch solution (prepared as shown in 4.4 Table 8 of JIS K 8001) as an indicator, successively titrate on it, make it an end point when the blue color of solution disappears, and obtain the consumed quantity of 1/30 mol/l sodium thiosulfate solution.
- 1.2.3) Take 25 ml of water in another Erlenmeyer flask with an interchangeable ground joint (200 ml), and thereafter carry out the procedures shown in 1.2.2).
- 1.2.4) Calculate the factor of 1/30 mol/l sodium thiosulfate standard solution in accordance with the following formula.

$$F_1 = \frac{m \times 25/1\,000}{0.001\,185 \times (V_1 - V_2)} \times \frac{P}{100}$$

where, F_1 : factor of 1/30 mol/l sodium thiosulfate standard solution

m : mass of potassium iodate weighed at 1.2.1) (g)

P : purity of potassium iodate [% (m/m)]

V_1 : consumed quantity, obtained at 1.2.2), of 1/30 mol/l sodium thiosulfate solution (ml)

V_2 : consumed quantity, obtained at 1.2.3), of 1/30 mol/l sodium thiosulfate solution (ml)

2) Standardization of 1/30 mol/l ammonium iron(III) sulfate standard solution

- 2.1) Take accurately 25 ml of 1/30 mol/l ammonium iron(III) sulfate standard solution in an Erlenmeyer flask with an interchangeable ground joint (300 ml), add 35 ml of water, 3 ml of hydrochloric acid (1+1), and 3 g of potassium iodide, immediately put a plug, swirl gently, and allow it to stand in dark place for 10 min.
- 2.2) Titrate isolated iodine with 1/30 mol/l sodium thiosulfate standard solution that was prepared and standardized at 1), when the solution gives faint yellow, add 10 ml of sodium acetate solution (250 g/l) and 3 ml of starch solution (prepare as shown in 4.4 Table 8 of JIS K 8001) as indicator, titrate on, make it an end point when the blue color of solution disappears, and obtain the consumed quantity of 1/30 mol/l sodium thiosulfate standard solution.
- 2.3) Take 60 ml of water into another Erlenmeyer flask with an interchangeable ground joint (300 ml), add 3 ml of hydrochloric acid (1+1) and 3 g of potassium iodide, immediately put a plug, swirl gently, allow it to stand in dark place for 10 min, thereafter carry out the procedures shown in 2.2).
- 2.4) Calculate the factor of 1/30 mol/l ammonium iron(III) sulfate standard solution in accordance with the following formula.

$$F_2 = \frac{(V_3 - V_4)}{25} \times F_1$$

where, F_2 : factor of 1/30 mol/l ammonium iron(III) sulfate standard solution

V_3 : consumed quantity, obtained at 2.2), of 1/30 mol/l sodium thiosulfate standard solution (ml)

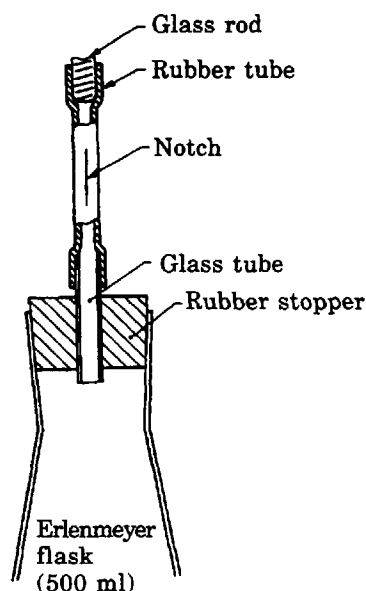
V_4 : consumed quantity, obtained at 2.3), of 1/30 mol/l sodium thiosulfate standard solution (ml)

F_1 : factor of 1/30 mol/l sodium thiosulfate standard solution

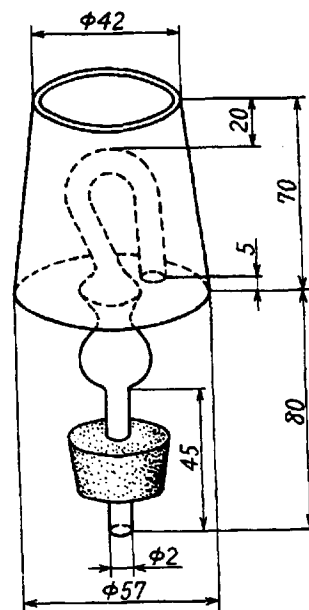
3 Implements for reduction The implements for reduction shall be either one of the following.

- Rubber stopper with valve** (see Annex 1 Fig.1) Pass a glass tube in a rubber stopper, and attach a rubber tube to it. Close the top of the rubber tube with a glass rod, and make a notch with 10 to 15 mm long between the end point and the glass tube using a razor edge.
- Rubber stopper with cap** (see Annex 1 Fig.2) Put sodium hydrogencarbonate solution (saturated, about 100 g/l) in the cap when it is used.

Unit: mm



Annex 1 Fig. 1 Rubber stopper with valve



Annex 1 Fig.2 Rubber stopper with cap

4 Quantity of sample Weigh out 1.0 g of sample to the nearest 0.1 mg.

5 Operation

5.1 Preparation of sample solution Carry out the preparation of sample solution in accordance with the following procedures.

- a) Weigh the sample into a beaker (500 ml), add 50 ml of water, cover it with a watch glass, and add little by little 30 ml of sulfuric acid then 50 ml of hydrochloric acid while making agitation. Heat gently the beaker to decompose the sample, add 10 ml of nitric acid, remove the watch glass after rinsing its lower surface with water, and continuously heat it until white fume of sulfuric acid emits. After allowing it to cool, wash the inside wall of the beaker with a little water, again heat it to generate a dense white fume of sulfuric acid for 5 min. After allowing it to cool, add 200 ml of water and agitate well, and heat gently it to dissolve the salts that are dissolvable⁽¹⁾.

Note ⁽¹⁾ In this solution if no undecomposed residue is recognized, the following procedures of b) and c) can be omitted.

- b) Filter the solution through a filter paper (5 grade C), wash the filter paper and undecomposed residue with warmed hydrochloric acid (1+4) and warmed water, and put the filtrate and washings in a beaker (500 ml) to store.
- c) Transfer the residue into a platinum crucible (No. 30) together with the filter paper, dry them, and heat gradually it to ash the filter paper. After allowing it to cool, moisten the residue with 2 to 3 drops of sulfuric acid (1+1), add 1 to 2 ml of hydrofluoric acid, heat it to vaporize silicon dioxide and sulfuric acid. After allowing it to cool, add 3 g of potassium disulfate in the platinum crucible, heat gradually it to raise its temperature finally up to about 700 °C, and fuse the residue. After allowing it to cool, place the platinum crucible into the filtrate and washings that was stored at b), heat it to dissolve the fused, and take out the crucible after washing it with water.
- d) After allowing it to cool to ordinary temperature, transfer the solution into a 500 ml volumetric flask with rinsing the beaker with water, and add water up to the marked line.

5.2 Reduction of titanium Carry out the reduction of titanium in accordance with either procedure of the following.

a) Case of containing no vanadium, molybdenum, or tin in sample

- 1) Take partly 50 ml from the solution prepared at 5.1 d) into an Erlenmeyer flask (500 ml), add 60 ml of hydrochloric acid, and add water to make total about 200 ml.
- 2) Add about 3 g of aluminium [2.1], heat the apparatus for reduction (3) equipped with a rubber stopper, instantly aluminium begins to decompose let down it from the heat source, allow it to stand, complete the decomposition of aluminium, then heat it again to boil for 1 to 2 min, and cool it in running water to ordinary temperature.

b) Case of containing vanadium, molybdenum, or tin in sample

- 1) Take partly 50 ml from the solution prepared at 5.1 d) into a beaker (300 ml), neutralize it by adding drop by drop sodium hydroxide solution, then add excessively its 10 ml, heat it to boil for 1 to 2 min, and cool it to ordinary temperature.
- 2) Filter the precipitate with a filter paper (5 grade B or 6 grade), wash once the inside of the beaker and two or three times the filter paper and precipitate with sodium carbonate solution. Discard the filtrate and washings.

- 3) Collect the precipitate into the original beaker with jetting warm water on it, dissolve the precipitate adhering on the filter paper by pouring 20 ml of warmed hydrochloric acid (1+1), and put the solution into the original beaker. Wash the filter paper with warmed hydrochloric acid (1+4), and put the washings into the original beaker. Heat gently the beaker to dissolve the precipitate completely, transfer it into an Erlenmeyer flask (500 ml), add 50 ml of hydrochloric acid, and add water to make total about 200 ml.
- 4) Carry out the procedures shown in a) 2).

5.3 Titration Disassemble the apparatus for reduction from the Erlenmeyer flask, in which the solution obtained at a) 2) or b) 4) of 5.2 was kept, immediately add 10 ml of ammonium thiocyanate solution in the flask as indicator, titrate it with 1/30 mol/l ammonium iron(III) sulfate standard solution [2 m)], make it an end point when reddish brown color lasts for about 1 min, and obtain the consumed quantity of 1/30 mol/l ammonium iron(III) sulfate standard solution.

6 Blank test Employ reagents only, and carry out the procedures from 5.1 to 5.3 in parallel with the procedures for sample.

7 Calculation Calculate the titanium content in the sample in accordance with the following formula.

$$Ti = \frac{(V_5 - V_6) \times F_2 \times 0.001597}{m \times 1/10} \times 100$$

- where,
- Ti : titanium content in sample [% (m/m)]
 - V_5 : consumed quantity, obtained at 5.3, of 1/30 mol/l ammonium iron(III) sulfate standard solution (ml)
 - V_6 : consumed quantity, obtained at 6, of 1/30 mol/l ammonium iron(III) sulfate standard solution (ml)
 - F_2 : factor of 1/30 mol/l ammonium iron(III) sulfate standard solution
 - m : quantity of sample weighed out (g)

**Annex 2 (normative) Determination of titanium content —
Aluminium reduction ammonium iron(III)
sulfate titrimetric method (2)**

Introduction This Annex has been prepared based on the first edition of **ISO 7692 Ferrotitanium — Determination of titanium content — Titrimetric method** published in 1983, without any modification in technical contents.

In this Annex, portions underlined with dots are the items which are not specified in the corresponding International Standard.

1 Purpose and scope This Annex 2 (normative) specifies a titrimetric method for the determination of the titanium content of ferrotitanium.

The method is applicable to ferrotitanium having titanium contents between 20 and 75 % (m/m).

Informative reference: Though the original in ISO gives 80 % (m/m), this Annex adopts 75 % (m/m) which is the maximum limit in the products of ISO 5454 and JIS G 2309.

2 Normative references

ISO 3713:1987 *Ferroalloys — Sampling and preparation of samples — General rules*

Informative reference: In the original of ISO, Note ⁽¹⁾ informs that this ISO 3713 is at the stage of draft at present.

ISO 5454:1980 *Ferrotitanium — Specification and conditions of delivery*

JIS G 2309 *Ferrotitanium*

3 Principle Dissolution of a test portion using sulphuric, hydrofluoric, nitric and hydrochloric acids. Separation of the interfering elements (chromium, vanadium, molybdenum and tin), if present, by precipitation of titanium hydroxide in the presence of hydrogen peroxide.

Reduction of the titanium to Ti(III) by aluminium metal in an atmosphere of carbon dioxide or nitrogen. Titration of the Ti(III) with standard volumetric iron(III) ammonium sulphate solution in the presence of thiocyanate as indicator.

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

4.1 Aluminium Of minimum purity 99.5 % (m/m), in the form of foil 0.05 mm thick and free from titanium.

4.2 Sodium hydrogen carbonate (NaHCO₃).

4.3 Nitric acid Density 1.42 g/ml

4.4 Sulphuric acid Density 1.84 g/ml