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Iron and steel—Methods for determination of niobium content

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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 Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

In this revision, inductively coupled plasma atomic emission spectrometric method is newly specified, however, niobium pentoxide gravimetric method and pyrogallol spectrophotometric method were withdrawn as they had not been used.

JIS G 1237:1997 contains Annexes as under:

Annex 1 Sulfochlorophenol S spectrophotometric method

Annex 2 Sulfochlorophenol S 1-butanol extraction spectrophotometric method

Annex 3 Inductively coupled plasma atomic emission spectrometric method

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Iron and steel—Methods for determination of niobium content

1 Scope This Japanese Industrial Standard specifies the methods for determination of niobium in iron and steel.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. Normative references described on the most recent edition shall apply.

JIS G 1201 *General rules for chemical analysis of iron and steel*

JIS K 0116 *General rules for atomic emission spectrometry*

JIS Z 8402 *General rules for permissible tolerance of chemical analyses and physical tests*

3 General matters General matters common to the determination methods shall be in accordance with JIS G 1201.

4 Classification of determination methods The methods for determination of niobium shall be in accordance with any one of the following methods.

- (1) **Sulfochlorophenol S spectrophotometric method** This method is applied to the sample of 0.5 % (m/m) or over up to and including 2.5 % (m/m) niobium content, and the determination method shall be as described in Annex 1. However, this method can not be applied to such sample that 1 mg or over tungsten, 0.2 mg or over titanium, 0.1 mg or over molybdenum, 0.05 mg or over copper, 0.01 mg or over zirconium, or 0.005 mg or over tantalum coexists even independently in the sample solution partially taken for coloration.
- (2) **Sulfochlorophenol S 1-butanol extraction spectrophotometric method** This method is applied to the sample of 0.01 % (m/m) or over up to and including 0.5 % (m/m) niobium content, and the determination method shall be as described in Annex 2. However, this method can not be applied to such sample that each 5 mg or over of manganese, nickel, copper, aluminium, cobalt, or vanadium, each 1 mg or over of tin, arsenic, tungsten, or yttrium, 0.2 mg or over of titanium, 0.1 mg or over of molybdenum, 0.02 mg or over of zirconium, or 0.005 mg or over of tantalum coexists even independently in the sample solution partially taken for coloration.
- (3) **Inductively coupled plasma atomic emission spectrometric method** This method is applied to the sample of 0.001 % (m/m) or over up to and including 2.5 % (m/m) niobium content, and the determination method shall be as described in Annex 3.

Annex 1 Sulfochlorophenol S spectrophotometric method

1 Summary Decompose a sample with aqua regia, add sulfuric acid and phosphoric acid, generate white fume of sulfur trioxide by heating, and remove nitric acid. Dissolve salts by adding L(+)-tartaric acid and hydrochloric acid, and mask iron or the like with L(+)-ascorbic acid and disodium dihydrogen ethylenediaminetetraacetate (hereafter, referred to as "EDTA2Na"). Thereafter, generate sulfochlorophenol S niobium complex by adding sulfochlorophenol S, and measure the absorbance with a photometer.

2 Reagents The reagents shall be as follows:

- (1) **Hydrochloric acid**
- (2) **Hydrochloric acid (1 + 1)**
- (3) **Nitric acid**
- (4) **Aqua regia** (hydrochloric acid 3, nitric acid 1)
- (5) **Mixed acid** (sulfuric acid 2, phosphoric acid 1, water 7)
- (6) **Iron** The iron with high purity as possible, having 0.001 % (m/m) or under niobium content.
- (7) **L(+)-Tartaric acid solution (200 g/l)**
- (8) **L(+)-Ascorbic acid solution (10 g/l)** The solution is prepared at each service.
- (9) **EDTA2Na solution** Dissolve 1 g of disodium dihydrogen ethylenediaminetetraacetate dihydrate in water, and make 100 ml the amount of the solution with water.
- (10) **Sulfochlorophenol S solution** Dissolve 0.05 g of sulfochlorophenol S ($C_{22}H_{10}N_4O_{16}S_4Na_4Cl_2$) in water, and make 100 ml of the solution with water. Reserve it in a brown bottle, and filter it with dry filter paper (class 5A) at each service.
- (11) **Standard niobium solution (100 µg Nb/ml)** Weigh out 0.100 g of niobium [99.9 % (m/m) or over], transfer it onto a platinum dish (#100), and cover it with a platinum cover. Add 10 ml of hydrofluoric acid and several drops of nitric acid, and decompose the solution by heating. Wash the under surface of the platinum cover with water, remove the cover, add 5 ml of sulfuric acid (1 + 1), and generate white fume of sulfur trioxide by heating. After allowing the solution to stand for cooling add 20 ml of L(+)-tartaric acid solution (200 g/l) while cooling with water, and mix by stirring. Further, cool to ordinary temperature in running water, transfer the solution to a 1000 ml volumetric flask by use of tartaric acid solution (8 g/l), and dilute it to the mark with L(+)-tartaric acid solution (8 g/l).

3 Weighing out amount of sample The weighing out amount of a sample shall be as specified in Annex 1 Table 1.

Annex 1 Table 1 Weighing out amount of sample

Niobium content % (m/m)	Weighing out amount of sample g
0.5 or over up to and excluding 1.2	0.20
1.2 or over up to and including 2.5	0.10

4 Operation

4.1 Preparation of sample solution Prepare the sample solution as follows:

- (1) Weigh out a sample, transfer to a beaker (200 ml), cover it with a watch glass.
- (2) Add 15 ml of aqua regia. After decomposing by heating, add 25.0 ml of mixed acid.
- (3) Successively concentrate by heating⁽¹⁾, wash the bottom surface of the watch glass just before white fume of sulfur trioxide is generated, remove the watch glass, and generate white fume of sulfur trioxide for 4 min to 5 min by successively heating. Thereafter, cover it with a dry watch glass, and allow it to stand for cooling.
- (4) Add 40 ml of L (+)-tartaric acid solution (200 g/l) and 15 ml of hydrochloric acid, and dissolve salts by heating gently.
- (5) After cooling to ordinary temperature, wash the bottom surface of the watch glass with water, remove the watch glass, transfer the solution into a 250 ml volumetric flask by use of water, and dilute it to the mark with water⁽²⁾.

Notes (1) When carbides of niobium or the like exist in the sample, decompose them completely by raising temperature of the heat source to 500 °C or higher.

(2) When the solution is allowed to stand for a long time, niobium is sometimes hydrolyzed and the determination value decreases to a low value, therefore immediately carry out the operation of 4.2.

4.2 Coloration Partially take 5 ml from the solution obtained in 4.1 (5), transfer it into a 50 ml volumetric flask, add 15 ml of hydrochloric acid (1 + 1) and 5 ml of L (+)-ascorbic acid solution [2 (8)], and mix by shaking. After allowing to stand still for 3 min, add 5 ml of EDTA2Na solution [2 (9)], mix by shaking, add 3.0 ml or 4.0 ml⁽³⁾ of sulfochlorophenol S solution [2 (10)], mix by shaking, and dilute it to the mark with water. Then, dip the volumetric flask into the water bath preliminarily regulated at 60 °C to 65 °C for 5 min to heat, then cool it to ordinary temperature.

Note (3) As for the amount of sulfochlorophenol S solution [2 (10)] 3.0 ml is sometimes insufficient according to the lot of the reagent to be used, therefore add 4.0 ml if necessary.

4.3 Measurement of absorbance One min later from coloration, take a part of the solution obtained in 4.2 into the absorption cell (1 cm) of a photometer, and measure the absorbance at a wavelength close to 655 nm by using the blank test solution obtained in 5 as the contrast solution.

5 Blank test Weigh out the same amount of iron [2 (6)] instead of the sample, and carry out the same operation as that for the sample in parallel with the sample according to the procedure of 4.1 to 4.3.

6 Preparation of working curve Prepare six beakers (200 ml) for each range of niobium content, weigh out the same amounts of iron [2 (6)] as that of the weighed out sample and transfer it into each of the said beakers, add 0, 5.0, 10.0, 15.0, 20.0 and 25.0 ml of reference niobium solution [2 (11)] (0, 500, 1 000, 1 500, 2 000 and 2 500 µg as niobium) respectively, and cover with watch glasses. Then, carry out the same operations as those for the sample in parallel with the sample according to the procedure of 4.1 (2) to 4.3 (4), prepare the relation curve between the obtained absorbance and the amount of niobium added as reference niobium solution, and parallel transfer the relation curve so as to pass through the original point, and take it as the working curve.

Note (4) Measure the absorbance by using the solution to which reference niobium solution is not added as the contrast solution.

7 Calculation Obtain the amount of niobium from the absorbance obtained in 4.3 and the working curve prepared in 6, and calculate the content of niobium in the sample according to the following formula.

$$Nb = \frac{A}{m \times \frac{5}{250}} \times 100$$

where, Nb : niobium content in sample [% (m/m)]

A : detected amount of niobium in partially taken sample solution (g)

m : weighed out amount of sample (g)

8 Tolerance The tolerance⁽⁵⁾ shall be as specified in Annex 1 Table 2.

Annex 1 Table 2 Tolerances

Unit: % (m/m)

Niobium content	Within-laboratory-reproducibility limit	Reproducibility limit
0.5 or over up to and incl. 2.5	$D [0.007 2 \times (Nb) + 0.000 9]$	$D [0.010 0 \times (Nb) + 0.004 7]$

Note (5) The D in the calculation formula for tolerance means $D (n, 0.95)$, and the value shall be as described in Table 4 of JIS Z 8402. The value of (n) is the number of analyses in the same analytical laboratory for within-laboratory-reproducibility limit, and the number of analytical laboratories relating to the analysis for reproducibility limit.

The (Nb) means the niobium content [% (m/m)] on which the tolerance is obtained.

Informative reference: The tolerance is obtained from the result of cooperative experiments by use of the sample of 0.6 % (m/m) or over up to and including 2.5 % (m/m) niobium content.