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**Methods for determination of silicon
in aluminium and aluminium alloys**

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In the event of any doubts arising as to the contents,
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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Aluminium Association (JAA) / Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS H 1352:1997** is replaced with this Standard.

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Methods for determination of silicon in aluminium and aluminium alloys

Introduction

This Japanese Industrial Standard was initially prepared based on the first editions of **ISO 797** and **ISO 808** in 1973. Since the International Standards have long been left unrevised after its publishment and therefore, are obsolete in the light of the new technology, at this time, this Japanese Industrial Standard has been revised with some modifications of the technical contents. The following are the clauses uniquely given in **JIS** that have been added to the contents of the original International Standards: silicomolybdic acid spectrophotometric method (clause 7), silicomolybdic acid blue spectrophotometric method (acid decomposition method) (clause 9), ICP atomic emission spectrometry (method A) (clause 10), ICP atomic emission spectrometry (method B)(clause 11) and ICP atomic emission spectrometry (method C)(clause 12).

A list of modifications with explanations is given in Annex JA.

1 Scope

This Standard specifies the methods for determination of silicon in aluminium and aluminium alloys.

NOTE : The International Standards corresponding to this Standard and the symbols which denote the degree of correspondence in the contents between the International Standards and JIS are as follows :

ISO 797 : 1973 *Aluminium and aluminium alloys—Determination of silicon—Gravimetric method*

ISO 808 : 1973 *Aluminium and aluminium alloys—Determination of silicon—Spectrophotometric method with the reduced silicomolybdic complex* (total : MOD)

The symbols, (MOD), as given in **ISO/IEC Guide 21**, indicate that the original International Standards have been modified.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS H 1351 *General rules for chemical analysis of aluminium and aluminium alloy*

JIS K 0116 *General rules for atomic emission spectrometry*

3 General requirements

General requirements common to methods for determination shall be in accordance with JIS H 1351 and JIS K 0116.

In this Standard, the ordinary temperature shall be $20\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ and the room temperature shall be $20\text{ }^{\circ}\text{C} \pm 15\text{ }^{\circ}\text{C}$.

4 Division of determination methods

The method for determination of silicon contents shall be in accordance with any one of the following.

- a) **Determination of silicon by gravimetric method (method A)** This method is applicable to the sample of silicon contents 0.2 % (mass fraction) or over up to and including 25.0 % (mass fraction) and total contents of tin and antimony is less than 1.0 % (mass fraction).
- b) **Determination of silicon by gravimetric method (method B)** This method is applicable to the sample of silicon contents 0.2 % (mass fraction) or over up to and including 25.0 % (mass fraction) and total contents of tin and antimony is not less than 1.0 % (mass fraction).
- c) **Silicomolybdic acid spectrophotometric method** This method is applicable to the sample of silicon contents 0.03 % (mass fraction) or over up to and including 0.5 % (mass fraction), but not applicable to the alloys contained with tin and/or bismuth.
- d) **Silicomolybdic acid blue spectrophotometric method (alkali decomposition method)** This method is applicable to the sample of silicon contents 0.005 % (mass fraction) or over up to and including 0.05 % (mass fraction).
- e) **Silicomolybdic acid blue spectrophotometric method (acid decomposition method)** This method is applicable to the sample of silicon contents 0.001 % (mass fraction) or over up to and including 0.01 % (mass fraction).
- f) **ICP atomic emission spectrometry (method A)** This method is applicable to the sample of silicon contents 0.01 % (mass fraction) or over to and excluding 0.2 % (mass fraction).
- g) **ICP atomic emission spectrometry (method B)** This method is applicable to the sample of silicon contents 0.2 % (mass fraction) or over to and excluding 2.0 % (mass fraction).
- h) **ICP atomic emission spectrometry (method C)** This method is applicable to the sample of silicon contents 2.0 % (mass fraction) or over up to and including 12.0 % (mass fraction).

5 Determination of silicon by gravimetric method (method A)

5.1 Summary

Decompose the sample with sodium hydroxide and hydrogen peroxide. Add nitric acid and perchloric acid and heat to vaporize and generate white fume of perchloric acid for converting silicon to insoluble silicon dioxide. After allowing to cool, add warm water to dissolve the solute salts. Separate the filtrate by filtering. After heating the precipitate heavily, measure the weight. Then add sulfuric acid and hydrofluoric acid thereto to evaporate the silicon dioxide as silicon fluoride by heating and measure the loss in weight after heating heavily.

5.2 Reagents

The reagents shall be as follows :

5.2.1 Hydrochloric acid (1 + 1, 1 + 19)

5.2.2 Nitric acid

5.2.3 Perchloric acid

5.2.4 Hydrofluoric acid

5.2.5 Sulfuric acid (1 + 1)

5.2.6 Sodium hydroxide (granulated)

5.2.7 Sodium hydroxide solution, prepared by dissolving 250 g of sodium hydroxide with water, allowing to cool, and making up the volume to 1 000 ml with water.

This solution shall be stored in a polyethylene bottle and use its supernatant fraction.

5.2.8 Hydrogen peroxide (1 + 9)

5.2.9 Silver nitrate solution, prepared by dissolving 5 g of silver nitrate with 100 ml of water.

Stock the solution in brownish bottle.

5.3 Test portion

The test portion shall be as specified in table 1. Weigh out the sample to the nearest 1 mg.

Table 1 Test portion

Assumed silicon content % (mass fraction)	Test portion g
$0.2 \leq Si < 1.0$	3.0
$1.0 \leq Si < 3.0$	1.0
$3.0 \leq Si < 7.0$	0.50
$7.0 \leq Si < 25.0$	0.30

5.4 Procedure

5.4.1 Decomposition of the sample and dehydration treatment of silicon dioxide

Decomposition of the sample and dehydration treatment of silicon dioxide shall be in

accordance with any one of the following :

a) The sample of silicon contents 0.2 % (mass fraction) or over to and excluding 1.0 % (mass fraction)

- 1) Transfer the weighed out sample into a nickel beaker (300 ml), cover with a lid made of nickel, add 40 ml of sodium hydroxide solution (5.2.7) in small portions to decompose the sample.

When the reaction is violent, in order to make it moderate, the vessel can be cooled in running water.

- 2) When the reaction becomes moderate, add 10 ml of hydrogen peroxide (1 + 9) and heat to decompose it thoroughly.
- 3) After cooling, pour the solution into the beaker (500 ml) in which 5 ml of nitric acid and 90 ml of perchloric acid are introduced previously. Rinse the rear bottom of the nickel lid and inside wall of the beaker with small volume of water at first, and then rinse them with small volumes of hydrochloric acid (1 + 1) and combine the filtrate and the washing.
- 4) After heating the solution on a sand bath to generate white fume of perchloric acid, cover the beaker with a watch glass, heat heavily on the sand bath. Maintain fuming for 15 min to 20 min after the fumes of perchloric acid flow backward along the beaker wall. Allow to cool, rinse the rear bottom of the watch glass with small volumes of water and remove it.

b) The sample of silicon contents 1.0 % (mass fraction) or over to and excluding 3.0 % (mass fraction)

- 1) Transfer the weighed out sample into a nickel beaker (300 ml), cover with a Ni lid, add 6 g of sodium hydroxide (granulated) and add 10 ml of water in small portions to decompose the sample.

When the reaction is violent, in order to make it moderate, the vessel can be cooled in running water.

- 2) Carry out the procedure of **a) 2)**. After cooling, pour the solution into the beaker (500 ml) in which 5 ml of nitric acid, 50 ml of perchloric acid and 20 ml of water are introduced previously. Rinse rear bottom of the nickel lid and inside wall of the beaker with small volume of water at first, and then rinse them with small volumes of hydrochloric acid (1 + 1) and combine the filtrate and the washing.
- 3) Carry out the procedure specified in **a) 4)**.

c) The sample of silicon contents 3.0 % (mass fraction) or over to and excluding 7.0 % (mass fraction)

- 1) Transfer the weighed out sample into a nickel beaker (300 ml), cover with a Ni lid, add 5 g of sodium hydroxide (granulated) and add 10 ml of water in small portions to decompose the sample.

When the reaction is violent, in order to make it moderate, the vessel can be

cooled in running water.

- 2) Carry out the procedure specified in a) 2). After cooling, pour the solution into the beaker (500 ml) in which 5 ml of nitric acid, 40 ml of perchloric acid and 15 ml of water are introduced previously. Rinse the rear bottom of the nickel lid and inside wall of the beaker with small volume of water at first, and then rinse them with small volumes of hydrochloric acid (1 + 1) and combine the filtrate and the washing.
 - 3) Carry out the procedure specified in a) 4).
- d) **The sample of silicon contents 7.0 % (mass fraction) or over up to and including 25.0 % (mass fraction)**
- 1) Transfer the weighed out sample into a nickel beaker (300 ml), cover with a Ni lid, add 8 g of sodium hydroxide (granulated) and add 5 ml of water in small portions to decompose the sample becomes moderate.

When the reaction is violent, in order to make it moderate, the vessel can be cooled in running water.
 - 2) When the reaction becomes moderate, evaporate the sample solution to dehydrate and dry on a sand bath.
 - 3) After cooling, rinse the rear bottom of the nickel lid and inside wall of the beaker with a small volume of water, then add 10 ml of hydrogen peroxide (1 + 9) and heat the sample to decompose thoroughly.
 - 4) After cooling, pour the solution into the beaker (500 ml) in which 5 ml of nitric acid, 60 ml of perchloric acid and 20 ml of water are introduced previously. Rinse the rear bottom of the nickel lid and inside wall of the beaker with a small volume of water at first, then rinse them with small volumes of hydrochloric acid (1 + 1) and combine the filtrate and the washing.
 - 5) Carry out the procedure specified in a) 4).

5.4.2 Filtration and washing

Filtration and washing shall be in accordance with the following :

- a) Add warm water to the beaker containing the salts which are obtained at a) 4), b) 3), c) 3) or d) 5) in 5.4.1, to make about 400 ml, and mix the solution with glass stirring rod and heat to dissolve the salts.
- b) After standing the precipitate to settle, separate immediately by filtrating with a filter paper (class 5 B), rubbing out silicon dioxide adhered on inside wall of the beaker with policeman (glass rod attached with gum tube) and transfer onto a filter paper with small volumes of warm hydrochloric acid (1 + 19).
- c) Wash the filter paper and the precipitate about 10 times with warm hydrochloric acid (1 + 19), and then wash with warm water sufficiently until a small amount of washing, when silver nitrate solution (5.2.9) is added to it, no longer becomes turbid.

5.4.3 Weighing

Weighing shall be in accordance with the following.

- a) Transfer the precipitate of silicon dioxide obtained by 5.4.2 c) together with the filter paper into a platinum crucible (No. 30), then heat the filter paper to dry and ignite to ash.
- b) Heat heavily the precipitate and filter paper for about 1 h or so at 1 100 °C to 1 150 °C, allow to stand to cool to room temperature in a desiccator and then measure the mass. Repeat the procedures until it reaches the constant weight.
- c) Add 1 ml of sulfuric acid (1 + 1) and about 5 ml of hydrofluoric acid into a platinum crucible, heat gently to vaporize silicon dioxide and continue to heat until white fume of sulfuric acid does not generate.
- d) Heat heavily the precipitate and filter paper for half an hour or so at 1 100 °C to 1 150 °C, allow to stand to cool to room temperature in a desiccator and then measure the mass. Repeat the procedures until it reaches the constant weight.

5.5 Blank test

Procedure in 5.4, without using the sample, shall be conducted in parallel with that using the sample.

5.6 Calculation

The content of silicon in the sample shall be calculated according to the following formula :

$$Si = \frac{(m_1 - m_2 - m_3 + m_4) \times 0.4674}{m} \times 100 \%$$

where, Si : silicon content in the sample (mass fraction)

m_1 : mass (g) obtained in 5.4.3 b)

m_2 : mass (g) obtained in 5.4.3 d)

m_3 : mass (g) obtained in 5.4.3 b) of 5.5

m_4 : mass (g) obtained in 5.4.3 d) of 5.5

m : test portion (g)

6 Determination of silicon by gravimetric method (method B)

6.1 Summary

Decompose the sample with sodium hydroxide and hydrogen peroxide, add perchloric acid, bromine water and hydrobromic acid, allow to vaporize and generate white fume of perchloric acid. Vaporize to remove tin and antimony as bromides. Continue heat to vaporize for converting silicon to insoluble silicon dioxide. Allow to cool, add warm water to dissolve the soluble salts. Separate the filtrate by filtering. After heating the precipitate heavily, and measure it in weight. Then add sulfuric acid and hydrofluoric acid thereto to evaporate the silicon dioxide as silicon fluoride with heating heavily, and measure the loss in weight.