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Iron and steel — Methods for determination of silicon 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. Consequently, this revision supersedes JIS G 1212:1981.

In this revision, the relevant ISO Standards are laid down as Annexes 2, 4 and 5 in order to harmonize with International Standards.

JIS G 1212:1997 contains Annexes as under;

- Annex 1 Gravimetric method as silicon dioxide (1)
- Annex 2 Gravimetric method as silicon dioxide (2)
(ISO 439)
- Annex 3 Reduced molybdosilicate spectrophotometric method (1)
- Annex 4 Reduced molybdosilicate spectrophotometric method (2)
(ISO 4829-1)
- Annex 5 Reduced molybdosilicate spectrophotometric method (3)
(ISO 4829-2)

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Iron and steel — Methods for determination of silicon content

Introduction This Japanese Industrial Standard has been prepared on the basis of the second edition of **ISO 439**, *Steel and iron—Determination of total silicon content—Gravimetric method* published in 1994, **ISO 4829-1**, *Steel and cast iron — Determination of total silicon content—Reduced molybdosilicate spectrophotometric method—Part 1: Silicon contents between 0.05 and 1.0 %* published in 1986 and **ISO 4829-2**, *Steel and iron — Determination of total silicon content — Reduced molybdosilicate spectrophotometric method — Part 2 : Silicon contents between 0.01 and 0.05%* published in 1988 which are edited in Annexes 2, 4 and 5, respectively without any modification in technical content and style thereof, but some additional requirements not specified in the corresponding International Standards are given as a part of the Japanese Industrial Standard.

Further, in this Standard, the “Informative reference” underlined with dotted line are the matters not specified in the original International Standards.

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

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. As to those normative references, the most recent editions thereof shall be applied.

- | | |
|------------|--|
| JIS G 1201 | <i>General rules for chemical analysis of iron and steel</i> |
| JIS Z 8402 | <i>General rules for permissible tolerance of chemical analyses and physical tests</i> |
| ISO 439 | <i>Steel and iron—Determination of total silicon content—Gravimetric method</i> |
| ISO 4829-1 | <i>Steel and cast iron—Determination of total silicon content—Reduced molybdosilicate spectrophotometric method—Part 1 : Silicon contents between 0.05 and 1.0 %</i> |
| ISO 4829-2 | <i>Steel and iron—Determination of total silicon content—Reduced molybdosilicate spectrophotometric method—Part 2 : Silicon contents between 0.01 and 0.05 %</i> |

3 General requirements General requirements common to methods for determination shall be in accordance with **JIS G 1201**, provided that this does not cover Annexes 2, 4 and 5.

4 Division of determination methods The method for determination of silicon content shall be in accordance with any one of the following:

- (1) **Gravimetric method as silicon dioxide** (1) This method is applicable to the sample of total silicon contents between 0.1 % (*m/m*) and 8.0 % (*m/m*), and the method for determination shall be in accordance with Annex 1.

- (2) **Gravimetric method as silicon dioxide (2) (ISO 439)** This method is applicable to the sample of total silicon contents between 0.10 % (*m/m*) and 5.0 % (*m/m*), and the method for determination shall be in accordance with Annex 2.
- (3) **Reduced molybdosilicate spectrophotometric method (1)** This method is applicable to the sample of silicon contents between 0.01 % (*m/m*) and 1.0 % (*m/m*), and the method for determination shall be in accordance with Annex 3.
- (4) **Reduced molybdosilicate spectrophotometric method (2) (ISO 4829-1)** This method is applicable to the sample of total silicon contents between 0.05 % (*m/m*) and 1.0 % (*m/m*), and the method for determination shall be in accordance with Annex 4.
- (5) **Reduced molybdosilicate spectrophotometric method (3) (ISO 4829-2)** This method is applicable to the sample of total silicon contents between 0.01 % (*m/m*) and 0.05 % (*m/m*), and the method for determination shall be in accordance with Annex 5.

Annex 1 Gravimetric method as silicon dioxide (1)

1 Purport Decompose the test portion with appropriate acids, heat to vaporize with addition of perchloric acid or sulfuric acid, and convert silicon to insoluble silicic acid. After separation of the precipitate by filtration, ignite it to make silicon dioxide, and measure the mass. Further, add sulfuric acid and hydrofluoric acid, heat to volatilize silicon dioxide, then measure the mass.

2 Reagents The reagents shall be as follows:

- (1) **Hydrochloric acid**
- (2) **Hydrochloric acid** (2+1, 1+4, 1+10)
- (3) **Nitric acid**
- (4) **Nitric acid** (1+1)
- (5) **Perchloric acid**
- (6) **Hydrofluoric acid**
- (7) **Sulfuric acid**
- (8) **Sulfuric acid** (1+1, 1+3)
- (9) **Aqua regia** (hydrochloric acid 3, nitric acid 1)
- (10) **Hydrogen peroxide**
- (11) **Sodium carbonate**
- (12) **Ammonium thiocyanate solution** (saturated)

3 Weigh-out mass of test portion The mass of test portion to be weighed out shall be as given in Annex 1 Table 1.

Annex 1 Table 1 Weigh-out mass of test portion

Silicon content % (m/m)	Weigh-out mass of test portion g
0.10 and over, to and excl. 1.0	3.0
1.0 and over, up to and incl. 8.0	1.0

4 Procedure

Informative reference: **Warning** — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material. All vaporations of perchloric acid must be carried out in fume cupboards suitable for use with perchloric acid.

4.1 Decomposition of test portion and dehydrating treatment of silicic acid

The decomposition of the test portion and the dehydrating treatment of silicic acid shall be carried out in accordance with any one of the following procedures:

(1) Sample easy for decomposition with perchloric acid and nitric acid

- (a) Transfer the test portion weighed out to a beaker (300 ml) and cover it with a watch glass.
- (b) Add 20 ml of perchloric acid for the weigh-out test portion of 1.0 g, or 40 ml for 3.0 g thereof. Further, add 15 ml to 20 ml of nitric acid (1+1) for every 1.0 g of weigh-out test portion, and heat gently to decompose.
- (c) Ignite successively, and continue heating for 15 min to 20 min after attaining the condition where incurred vapour of perchloric acid refluxes on the inner wall of the beaker.

(2) Sample difficult for decomposition with perchloric acid and nitric acid or sample containing niobium or tantalum

- (a) Transfer the test portion weighed out to a beaker (300 ml), and cover it with a watch glass.
- (b) Add 20 ml to 30 ml of aqua regia, and heat gently to decompose.
- (c) Add 20 ml of perchloric acid for the weigh-out test portion of 1.0 g, or 40 ml for 3.0 g thereof.
- (d) Carry out the operation in accordance with (1)(c).

(3) Sample containing chromium or tungsten

- (a) Transfer the test portion weighed out to a beaker (300 ml), and cover it with a watch glass.
- (b) Add 20 ml of hydrochloric acid (2+1) little by little for the weighed-out test portion of 1.0 g, or 40 ml for 3.0 g thereof, and heat it gently to decompose.
- (c) Add 20 ml of perchloric acid for the weighed-out test portion of 1.0 g, or 40 ml for 3.0 g thereof.
- (d) Ignite successively, and continue heating for 15 min to 20 min after attaining the condition where chromium is oxidized whereby it is converted to red dichromic acid and the vapour of perchloric acid refluxes on the inner wall of the beaker.

(4) Sample of high nickel, chromium, tungsten, cobalt, etc. content

- (a) Transfer the test portion⁽¹⁾ weighed out to a beaker (300 ml), and cover it with a watch glass.
- (b) Add 50 ml of water and 10 ml of hydrochloric acid, and add 30 ml of hydrogen peroxide little by little, then heat it gently to decompose after the completion of violent reaction.
- (c) Add 20 ml of perchloric acid.
- (d) Carry out the operation in accordance with (3)(d).

Note ⁽¹⁾ For the sample of high nickel, chromium, tungsten, cobalt, etc. content, the mass of test portion to be weighed out shall be 1.0 g, irrespective of the silicon contents.

(5) Sample containing molybdenum or titanium

- (a) Transfer the test portion⁽¹⁾ weighed out to a beaker (300 ml), and cover it with a watch glass.
- (b) Add 40 ml of sulfuric acid (1+3) for the weigh-out test portion of 1.0 g, or 60 ml for 3.0 g thereof, and heat to decompose.
- (c) Add 5 ml of nitric acid for every 1.0 g of weigh-out test portion, and boil it.
- (d) Add 5 ml to 10 ml of hydrochloric acid, remove the watch glass after washing its underneath surface with water, and continue heating on a sand bath to generate dense white fume of sulfuric acid for 15 min to 20 min.

(6) In the case of using sulfuric acid for dehydration of silicic acid in the sample which is easy for decomposition with nitric acid and hydrochloric acid

- (a) Transfer the test portion⁽¹⁾ weighed out to a beaker (300 ml), and cover it with a watch glass.
- (b) Add 15 ml to 20 ml of nitric acid (1+1) little by little for every 1.0 g of weigh-out test portion, and heat it gently to decompose.
- (c) Add 5 ml to 10 ml of hydrochloric acid.
- (d) Add 20 ml of sulfuric acid (1+1) for the weigh-out test portion of 1.0 g, or 30 ml for 3.0 g thereof.
- (e) Remove the watch glass after washing its underneath surface with water, continue heating on a sand bath to generate dense white fume of sulfuric acid for 15 min to 20 min.

4.2 Filtration and washing The filtration and washing shall be carried out in accordance with either of the following procedures:

(1) In the case of dehydration with perchloric acid

- (a) Let to cool the salts obtained in (1)(c), (2)(d), (3)(d), or (4)(d) of 4.1 to approximate room temperature.
- (b) Remove the watch glass after washing its underneath surface with water, add approximately 120 ml of warm water, and stir to dissolve the salts.
- (c) Immediately, separate the precipitation by filtration with a filter paper (Class 5B), remove the silicic acid adhered to the inner wall of the beaker with a policeman (rubber-tipped glass rod), and transfer it to the filter paper.
- (d) At the beginning, wash it about 5 times alternately with warm water and with hydrochloric acid (1+10) heated at between 40 °C and 60 °C and at last wash it with warm water until the ferric (iron III) ion vanishes in the filtrate⁽²⁾ ⁽³⁾ ⁽⁴⁾. Store the precipitate and discard the filtrate and the washings⁽⁵⁾.

Notes ⁽²⁾ In the case where small quantity of the washing is taken and ammonium thiocyanate solution (saturated) is added, wash it sufficiently until the coloration of (orange) red vanishes.

- (3) At the time of incineration according to 4.3 (1)(b), insufficient washing may cause popping, that is dangerous, and may incur loss of residue.
- (4) In the case where undecomposed residue of the test portion is appreciated in the precipitate after washing by filtration, transfer the precipitate together with the residue and the filter paper to a platinum crucible (No.30), dry, incinerate, add approximately 6 times quantity of sodium carbonate, and heat to fuse. After letting to cool, transfer the fusion products together with the platinum crucible to a beaker (300 ml), and 100 ml of warm water to extract the fusion products, and take out the platinum crucible after washing with water. Cover it with a watch glass, and add hydrochloric acid little by little until it is acidified to dissolve the salts. Further, add 30 ml of perchloric acid, then carry out the operations in accordance with 4.1 (1)(c) and 4.2 (1).
- (5) In the case of the sample of silicon contents of 1 % (*m/m*) or more, it is necessary to recover silicon from the filtrate and washings in accordance with the following procedure. Collect the filtrate and washings into a beaker (500 ml), cover with a watch glass, add 20 ml of perchloric acid, heat it, continue heating for 15 min to 20 min after attaining the condition where the vapour of perchloric acid being vaporized refluxes on the inner wall of the beaker, and let to cool to the room temperature. Thereafter, recover the silicon by the operation in accordance with (b) to (d), and incorporate with the precipitate having been stored. Discard the filtrate and washings.

(2) In the case of dehydration with sulfuric acid

- (a) Let to cool the salts obtained in (5) (d) or (6) (e) of 4.1 to approximate room temperature.
- (b) Add 50 ml of hydrochloric acid (1+4) little by little, and heat to dissolve the salts.
- (c) Carry out the operation in accordance with (1) (c).
- (d) At the beginning, wash it about 5 times alternately with warm water and with hydrochloric acid (1+10) heated at between 40 °C and 60 °C, and at last wash it with warm water until the ferric (iron III) ion vanishes in the filtrate⁽²⁾ (6) (7). Store the precipitation as main precipitation.
- (e) Collect the filtrate and washings into a beaker (500 ml), cover with a watch glass, and heat to vaporize. Remove the watch glass after washing its underneath surface with water, heat again on a sand bath to generate dense white fume of sulfuric acid for 15 min to 20 min, and then let to cool.
- (f) Carry out the operations in accordance with (b) and (c).
- (g) At the beginning, wash it about 5 times alternately with warm water and with hydrochloric acid (1+10) heated at between 40 °C and 60 °C, and at last wash it with warm water until the ferric (iron III) ion vanishes in the filtrate⁽²⁾. Discard the filtrate and washings. Incorporate precipitate with the main precipitate having been stored as specified in (d).

- Notes (6) In the case where the sample contains molybdenum or titanium, at the beginning, wash the test portion 5 times or 6 times with hydrochloric acid (1+10) heated at between 40 °C and 60 °C, and then wash it with warm water until the ferric (iron III) ion vanishes in the filtrate.
- (7) In the case where undecomposed residue of the test portion is appreciated in the precipitate after washing by filtration, transfer the precipitate together with the residue and the filter paper to a platinum crucible (No.30), dry, incinerate, add approximately 6 times quantity of sodium carbonate, and heat to fuse. After letting to cool, transfer the fusion products together with the platinum crucible to a beaker (300 ml), add 100 ml of warm water to extract the fusion products, and take out the platinum crucible after washing with water. Cover it with a watch glass, and add hydrochloric acid little by little until acidity appears to dissolve the salts. Further, add 20 ml of sulfuric acid (1+1), then carry out the operations in accordance with 4.1 (6) (e) and 4.2 (2).

4.3 Incineration and weighing The incineration and weighing shall be carried out in accordance with either of the following procedures:

- (1) **Sample not containing niobium, tantalum, titanium, zirconium, tungsten and molybdenum**
 - (a) Transfer the precipitate of silicic acid obtained in (1) (d) or (2) (g) of 4.2 together with the filter paper to a platinum crucible (No.30), dry, and incinerate the filter paper at a lower temperature.
 - (b) Ignite at 1100 °C or higher for 30 min to 45 min, let to cool to the room temperature in a desiccator, and weigh the mass of the platinum crucible. Repeat this operation until a constant mass is obtained.
- (2) **Sample containing niobium, tantalum, titanium, zirconium, tungsten or molybdenum**
 - (a) Carry out the operations in accordance with (a) and (b) of (1).
 - (b) Add approximately 2 ml of sulfuric acid (1+1), and heat. Continue heating carefully until generation of white fume of sulfuric acid ceases.
 - (c) Ignite at 800 °C, let to cool to the room temperature in a desiccator, and weigh the mass of the platinum crucible. Repeat this operation until a constant mass is obtained.

4.4 Treatment with hydrofluoric acid and weighing The treatment with hydrofluoric acid and weighing shall be carried out in accordance with either of the following procedures:

- (1) **Sample not containing niobium, tantalum, titanium, zirconium, tungsten and molybdenum**
 - (a) Wet the silicon dioxide in the platinum crucible obtained in 4.3 (1) (b) with sulfuric acid (1+1), add approximately 5 ml of hydrofluoric acid, and heat carefully to volatilize the silicon dioxide.

- (b) Continue heating until generation of white fume of sulfuric acid ceases.
 - (c) Ignite at 1100 °C or higher, let to cool to the room temperature in a desiccator, and weigh the mass of the platinum crucible. Repeat this operation until a constant mass is obtained.
- (2) **Sample containing niobium, tantalum, titanium, zirconium, tungsten or molybdenum**
- (a) Add approximately 2 ml of sulfuric acid (1+1) and approximately 5 ml of hydrofluoric acid to the silicon dioxide in the platinum crucible obtained in 4.3 (2) (c), and heat carefully to volatilize the silicon dioxide.
 - (b) Continue heating until generation of white fume of sulfuric acid ceases.
 - (c) Ignite at 800 °C for 10 min, let to cool to the room temperature in a desiccator, and weigh the mass of the platinum crucible. Repeat this operation until a constant mass is obtained.

5 Blank test In parallel with the determination and following the same procedure applied for the sample, carry out a blank test without using the sample.

6 Calculation The silicon content 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 [% (m/m)]

m_1 : mass of platinum crucible obtained in (1)(b) or (2)(c) of 4.3 in the case of using the test portion (g)

m_2 : mass of platinum crucible obtained in (1)(c) or (2)(c) of 4.4 in the case of using the test portion (g)

m_3 : mass of platinum crucible obtained in (1)(b) or (2)(c) of 4.3 in the blank test (g)

m_4 : mass of platinum crucible obtained in (1)(c) or (2)(c) of 4.4 in the blank test (g)

m : mass of the test portion weighed out (g)

7 Precision The precision⁽⁸⁾ shall be as given in Annex 1 Table 2.

Annex 1 Table 2 Precision

Unit: % (m/m)

Silicon content	Within-laboratory reproducibility	Reproducibility
0.1 and over, up to and incl. 8.0	$D [0.0027 \times (Si) + 0.0029]$	$D [0.0067 \times (Si) + 0.0016]$

Note ⁽⁸⁾ The symbol D in the formula for calculation of precision stands for $D(n, 0.95)$ and the value shall be as given in Table 4 specified in **JIS Z 8402**. The value n stands for the number of analyses conducted in the same laboratory in the case of within-laboratory reproducibility, while number of laboratories concerned in the case of reproducibility.

Further, (Si) is the silicon content [% (m/m)] in the determination of the precisions.

Informative reference: The precision is obtained from the round robin test results using samples of silicon contents between 0.1 % (m/m) and 3.3 % (m/m).

Annex 2 Gravimetric method as silicon dioxide (2)**Foreword for Annex 2**

This Annex 2 has been prepared on the basis of the second edition of ISO 439, *Steel and iron—Determination of total silicon content—Gravimetric method* published in 1994, without any modification in technical content and style for Japanese Industrial Standard.

Further, in this Standard the underlined portions with dotted line are the matters not specified in the original International Standard.

1 Scope This Annex 2 specifies a gravimetric method for the determination of the total silicon content in iron and steel. This method is applicable to the sample of silicon contents between 0.10 % (m/m) and 5.0 % (m/m)⁽¹⁾.

Note ⁽¹⁾ For steels containing molybdenum, niobium, tantalum, titanium, tungsten, zirconium or chromium, the results are less precise than for unalloyed steels.

2 Normative references The following standards contain provisions which, through reference in this test, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

- | | |
|----------------|---|
| ISO 377-2:1989 | <i>Selection and preparation of samples and test pieces of wrought steels—Part 2: Samples for the determination of the chemical composition</i> |
| ISO 385-1:1984 | <i>Laboratory glassware — Burettes — Part 1: General requirements</i> |
| ISO 648:1977 | <i>Laboratory glassware — One-mark pipettes</i> |
| ISO 1042:1983 | <i>Laboratory glassware — One-mark volumetric flasks</i> |
| ISO 3696:1987 | <i>Water for analytical laboratory use — Specification and test methods</i> |
| ISO 5725:1986 | <i>Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests</i> |

Informative reference: **ISO 377-2** was superseded by the following standard:

ISO 14284:1996 *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Principle Attack of a test portion by hydrochloric and nitric acids. Conversion of acid-soluble silicon compounds to hydrated silicon dioxide by evaporation with perchloric acid until white fumes appear. Filtration of the hydrated silicon dioxide and acid-insoluble silicon compounds, ignition to form impure silicon dioxide and then weighing. Treatment of the ignited residue with hydrofluoric and sulfuric acids, followed by ignition and weighing.

4 Reagents During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and grade 2 water as specified in **ISO 3696**.

4.1 Hydrochloric acid (ρ about 1.19 g/ml)

4.2 Hydrochloric acid (ρ about 1.19 g/ml) diluted 1+1

4.3 Hydrochloric acid (ρ about 1.19 g/ml) diluted 1+19

4.4 Nitric acid (ρ about 1.42 g/ml) diluted 3+1

4.5 Hydrofluoric acid (ρ about 1.14 g/ml)

4.6 Perchloric acid (ρ about 1.67 g/ml)⁽²⁾

4.7 Sulfuric acid (ρ about 1.83 g/ml) diluted 1+1

Note ⁽²⁾ Perchloric acid (ρ about 1.54 g/ml) may also be used.

5 Apparatus All volumetric glassware shall be class A, in accordance with **ISO 385-1**, **ISO 648** or **ISO 1042**, as appropriate.

Ordinary laboratory apparatus, and

5.1 Platinum crucibles, of capacity approximately 30 ml.

5.2 Muffle furnace, adjustable from 800 °C up to 1 100 °C.

6 Sampling Carry out sampling in accordance with **ISO 377-2** or appropriate national standards for steel and iron.

7 Procedure

Warning — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic matter in general. All evaporations must be carried out in fume cupboards suitable for use with perchloric acid.

7.1 Test portion Use millings or drillings of a maximum thickness of 0.2 mm.

According to the presumed silicon content, weigh out to the nearest 0.001 g, the following mass (m_0) of the test portion:

- a) for silicon contents between 0.10 % (m/m) and 0.50 % (m/m): $m_0 \approx 5$ g;
- b) for silicon contents between 0.50 % (m/m) and 2.5 % (m/m): $m_0 \approx 2.5$ g;