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Method for chemical analysis
of ferrosilicochromium

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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 the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently, **JIS G 1325 : 1989** is replaced with this Standard.

In this revision, the determination methods of each component are expressed using annexes system.

JIS G 1325 has the following Annexes.

- Annex 1 (normative) *Determination of silicon content—Silicon-dioxide gravimetric method*
- Annex 2 (normative) *Determination of silicon content—Sodium hydroxide titration method after separating precipitated potassium hexafluorosilicate*
- Annex 3 (normative) *Determination of chromium content—Potassium permanganate back-titration method of the solution treated with ammonium iron (II) sulfate after oxidation by ammonium peroxodisulfate*
- Annex 4 (normative) *Determination of chromium content—Ammonium iron (II) sulfate titration method of the solution oxidized by ammonium peroxodisulfate*
- Annex 5 (normative) *Determination of carbon content—Combustion-gas volumetric method*
- Annex 6 (normative) *Determination of carbon content—Combustion-infrared ray absorption method*
- Annex 7 (normative) *Determination of phosphorus content—Absorptiometry by molybdophosphoric acid blue*

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Method for chemical analysis of ferrosilicochromium

Introduction This Japanese Industrial Standard corresponds to the following two International Standards, that is, the first edition of **ISO 4158** *Ferrosilicon, ferrosilicomanganese and ferrosilicochromium—Determination of silicon content—Gravimetric method*, published in 1978, and the first edition of **ISO 4140** *Ferrochromium and ferrosilicochromium—Determination of chromium content—Potentiometric method*, published in 1979. This Standard, however, does not adopt the methods specified in them, because they both adopt the decomposition methods which use highly dangerous reagents. Instead, this Standard specifies the determination of silicon content in Annex 1 and Annex 2, and the determination of chromium content in Annex 3 and Annex 4, which are not specified in the corresponding International Standards, and furthermore supplementarily specifies the determination methods of carbon content and phosphorus content, which are not specified in the corresponding International Standards, in Annex 5, Annex 6 and Annex 7.

1 Scope This Standard specifies the determination of silicon, chromium, carbon, and phosphorus contained in ferrosilicochromium.

Remarks : The International Standards corresponding to this Standard are as follows.

ISO 4140 : 1979 *Ferrochromium and ferrosilicochromium—Determination of chromium content—Potentiometric method*

ISO 4158 : 1978 *Ferrosilicon, ferrosilicomanganese and ferrosilicochromium—Determination of silicon content—Gravimetric 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 K 0113 *General rules for methods of potentiometric, amperometric, coulometric, and Karl-Fischer titrations*

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

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

3 Generals The general matters applicable to determination methods are in conformity with **JIS G 1301** and **JIS K 0113**.

4 Classification of determination methods Determination methods are classified as shown in Table 1.

Table 1 Classification of determination methods

Component to be determined	Determination method	Applicable limit of content % (m/m)	No. of Annex (normative)
Silicon	Silicon-dioxide gravimetric method	10 or over up to and incl. 60	1
	Sodium hydroxide titration method after separating precipitated potassium hexafluorosilicate	10 or over up to and incl. 60	2
Chromium	Potassium permanganate back-titration method of the solution treated with ammonium iron (II) sulfate after oxidation by ammonium peroxodisulfate	20 or over up to and incl. 70	3
	Ammonium iron (II) sulfate titration method of the solution oxidized by ammonium peroxodisulfate	20 or over up to and incl. 70	4
Carbon	Combustion-gas volumetric method	0.05 or over up to and incl. 6.0	5
	Combustion-infrared ray absorption method	0.001 or over up to and incl. 6.0	6
Phosphorus	Absorptiometry by molybdophosphoric acid blue	0.005 or over up to and incl. 0.08	7

Annex 1 (normative)
Determination of silicon content—
Silicon-dioxide gravimetric method

1 Summary Fuse a sample with fusing mixture, dissolve it in hydrochloric acid, add perchloric acid, heat it until it emits white fumes, and make silicon undissolvable silicic acid. After dissolving dissolvable salts with water, filter the precipitate, ignite it to make it silicon dioxide, and weigh its mass. Then, add sulfuric acid and hydrofluoric acid, heat it to change silicon dioxide into silicon tetrafluoride, which vaporizes, and after igniting it weigh its mass.

2 Reagents Reagents shall be as follows.

- a) **Hydrochloric acid (1+2, 1+10)**
- b) **Perchloric acid**
- c) **Hydrofluoric acid**
- d) **Sulfuric acid (1+1, 1+3)**
- e) **Hydrogen peroxide**
- f) **Fusing mixture (sodium peroxide 7, sodium carbonate 3)**
- g) **Potassium thiocyanate solution (100 g/l)**

3 Quantity of sample The quantity of sample shall be 0.2 g weighed to the nearest 0.1 mg.

4 Operation

4.1 Decomposition of sample The decomposition of the sample shall be carried out as specified in the following procedures.

- a) Weigh the sample, and transfer it into a nickel crucible (30 ml).
- b) Add 5 g of fusing mixture, mix them well, cover it with 1 g of fusing mixture, put a nickel cover on it, and heat it until the contents start to fuse.
- c) Raise its temperature, heat it at approximately 700 °C (dark red color) for about 5 min, swirling it several times, until it completely fuses.
- d) After allowing it to cool, place the crucible with its cover into a beaker (500 ml) in which 60 ml of hydrochloric acid (1+2) has been put, dissolve the fused, and remove the crucible and cover after being rinsed with water.

4.2 Separation of silicic acid The separation of silicic acid shall be carried out as specified in the following procedures.

- a) Add 40 ml of perchloric acid⁽¹⁾ into the solution obtained at 4.1 d), cover it with a watch glass, heat it on a sand bath, and heat it on for about 15 min under the condition that the perchloric acid vapor is refluxed along the inside wall of the beaker.

- b) After allowing it to cool, add about 150 ml of warm water to dissolve dissolvable salts.
- c) Add little by little 5 ml of hydrogen peroxide to reduce dichromic acid, and heat it to boil for about 1 min⁽²⁾.
- d) Wash the under surface of the watch glass with water, remove the watch glass, and immediately filter the precipitate through filter paper (5 grade A). Put the filtrate into another beaker (500 ml) for storing.
- e) Transfer the precipitate stuck on the inside wall of the beaker by rubbing it with a rubber-capped glass rod onto the filter paper, wash it with hydrochloric acid (1+10) warmed at 40 °C to 60 °C five times, and then wash it with warm water until no iron (III) ion can be detected⁽³⁾. Put the washings together with the filtrate stored at d). Preserve this filter paper and precipitate.

Notes (1) Instead of perchloric acid, 40 ml of sulfuric acid (1+1) may be used. In this case, at the same time of adding 40 ml sulfuric acid (1+1) add 10 ml of hydrogen peroxide, and heat it until the dense fumes of sulfuric acid emit. After allowing it to cool, add about 150 ml of warm water to dissolve dissolvable salts, and boil it for about 1 min⁽²⁾. Thereafter, follow the procedures stated in d) and downward.

(2) Boiling for a long time makes undissolvable silicic acid again dissolvable, so make the time for boiling as short as possible, and immediately carry out the procedures of filtration in d).

(3) Ensure that a portion of the filtrate does not give reddish orange color when adding potassium thiocyanate solution to it.

4.3 Recovery of silicic acid The recovery of silicic acid shall be carried out as specified in the following procedures.

- a) Add 10 ml of perchloric acid⁽⁴⁾ into the filtrate and washings obtained at 4.2 e), and cover it with a watch glass.
- b) Heat it on a sand bath, and heat it on for about 15 min under the condition that the perchloric acid vapor is refluxed along the inside wall of the beaker.
- c) After allowing it to cool, add about 150 ml of warm water to dissolve dissolvable salts.
- d) Add little by little 5 ml of hydrogen peroxide to reduce dichromic acid, and heat it to boil for about 1 min⁽²⁾.
- e) Wash the under surface of the watch glass with water, remove the watch glass, and immediately filter the precipitate through filter paper (5 grade A).
- f) Transfer the precipitate stuck on the inside wall of the beaker by rubbing it with a rubber-capped glass rod onto the filter paper and rinsing it with hydrochloric acid (1+10) warmed at 40 °C to 60 °C, wash it five times with hydrochloric acid (1+10) warmed at 40 °C to 60 °C, and then wash it with warm water until no iron (III) ion can be detected⁽³⁾. Preserve this filter paper and precipitate.

Note (4) If 40 ml of sulfuric acid (1+1) instead of perchloric acid is added in 4.2 a), as stated in Note (1), adding of 10 ml of perchloric acid is not necessary.

4.4 Weighing Weighing shall be carried out as specified in the following procedures.

- a) Put together the filter paper and precipitate obtained at 4.2 e) and 4.3 f), and place them in a platinum crucible (No. 30).
- b) After drying, heat it gently until filter paper is ashed.
- c) Ignite it at at least 1 100 °C for about 30 min then, allow it to cool in a desiccator to room temperature, and weigh its mass.
- d) Repeat the procedures in c) in order to get the constant mass of platinum crucible containing the residue.
- e) Moisten the residue in the platinum crucible with 2 to 3 drops of sulfuric acid (1+3), add 3 ml to 5 ml of hydrofluoric acid, and heat it with care not to cause spitting, so as to evaporate silicon dioxide and sulfuric acid.
- f) Again ignite the platinum crucible at at least 1 100 °C for about 30 min, allow it to cool in a desiccator to room temperature, and weigh its mass.
- g) Repeat the procedures in f) in order to get the constant mass of platinum crucible containing the residue.

5 Blank test Employ the reagents only, and carry out the procedures in 4.1 to 4.4 in parallel with those for the sample.

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

$$Si = \frac{[(m_1 - m_2) - (m_3 - m_4)] \times 0.467}{m} \times 100$$

where, Si : silicon content in the sample [% (m/m)]
 m_1 : mass obtained at d) in 4.4 of sample test (g)
 m_2 : mass obtained at g) in 4.4 of sample test (g)
 m_3 : mass obtained at d) in 4.4 of blank test (g)
 m_4 : mass obtained at g) in 4.4 of blank test (g)
 m : mass of weighed sample (g)

7 Tolerance Tolerance shall follow Annex 1 Table 1.

Annex 1 Table 1 Tolerance of silicon determination in ferrosilicochromium

Unit : % (m/m)		
Silicon content	Reproducibility within laboratory	Reproducibility between laboratories
44.2	0.4	1.9

Informative reference : The silicon content in Annex 1 Table 1 is the silicon content [% (m/m)] found in the sample employed for cooperative experiments which were carried out to obtain tolerances in accordance with JIS Z 8402 : 1991.

Annex 2 (normative)

Determination of silicon content— Sodium hydroxide titration method after separating precipitated potassium hexafluorosilicate

1 Summary Decompose a sample with nitric acid and hydrofluoric acid with co-existing potassium fluoride, and precipitate potassium hexafluorosilicate; alternatively when it is hard to decompose with acid, carry out alkali fusing, dissolve it in nitric acid, add hydrofluoric acid and potassium nitrate, and precipitate potassium hexafluorosilicate. Filter this precipitate, dissolve it in warm water, and titrate it with sodium hydroxide standard solution using phenolphthalein as indicator.

2 Reagents Reagents shall be as follows.

- a) **Hydrochloric acid**
- b) **Nitric acid (1+1)**
- c) **Hydrofluoric acid**
- d) **Fusing mixture (sodium peroxide 2, sodium carbonate 1)**
- e) **Potassium nitrate**
- f) **Potassium nitrate solution (50 g/l)** If this solution shows alkaline property, neutralize it with nitric acid, and preserve in a polyethylene bottle.
- g) **Potassium fluoride solution** Dissolve 200 g of potassium fluoride in hydrofluoric acid, and add hydrofluoric acid to make 1 000 ml. Store this solution in a polyethylene bottle.
- h) **0.5 mol/l sodium hydroxide standard solution** Carry out the preparation, standardization, and calculation in accordance with (19.2) in 4.5 of JIS K 8001. Store this solution in a polyethylene bottle.
- i) **Phenolphthalein solution** Dissolve 0.5 g of phenolphthalein in 100 ml of ethanol (95).

3 Quantity of sample The quantity of sample shall be 0.2 g weighed to the nearest 0.1 mg.

4 Operation

4.1 Decomposition of sample and formation of precipitation The decomposition of sample and formation of precipitation shall be carried out as specified in either of the following procedures.

- a) **Decomposition by acid**
 - 1) Weigh the sample, and place it into a polyethylene beaker (300 ml).
 - 2) Add 30 ml of nitric acid (1+1), and add and shake little by little 10 ml of potassium fluoride solution [2 g] to decompose the sample.

- 3) Add 5 ml of hydrochloric acid and water to make about 80 ml, and heat it on a water bath for about 15 min.
- 4) Add a small amount of filter paper pulp in it, allow it to stand for about 30 min while being cooled to 15 °C or lower, and precipitate potassium hexafluorosilicate.

b) Fusion by alkali

- 1) Weigh the sample, and place it in a nickel crucible (30 ml).
- 2) Add 5 g of fusing mixture, mix them well, and cover it with 1 g of fusing mixture.
- 3) Put a nickel cover on it, heat it gently first until the contents start to fuse.
- 4) Raise its temperature, heat it at approximately 700 °C (dark red color) for about 5 min, swirling it several times, until it fuses completely.
- 5) After allowing it to cool, add a little amount of warm water, and heat gently to dissolve the fused.
- 6) Transfer the solution into a polyethylene beaker (300 ml) in which 50 ml of nitric acid (1+1) has been placed, rinsing the beaker with a little of water.
- 7) Pour 3 ml of hydrochloric acid into the crucible to dissolve the fused material stuck to its inside wall, and wash the crucible and cover with water sufficiently.
- 8) Heat the polyethylene beaker keeping the solution on a water bath to completely dissolve the fused.
- 9) Add water to make about 80 ml, add 10 ml of hydrofluoric acid and 3 g of potassium nitrate, agitate them, and heat it for about 15 min on a water bath.
- 10) Add a small amount of filter paper pulp in it, allow it to stand for about 30 min while being cooled to 15 °C or lower, and precipitate potassium hexafluorosilicate.

4.2 Separation of precipitate The separation of precipitate shall be carried out as specified in the following procedures.

- a) Filter the solution containing the precipitate obtained at **a) 4)** or **b) 10)** of **4.1** while being cooled to 15 °C or lower using a polyethylene funnel attached with a filter paper (5 grade A).
- b) Wash⁽¹⁾ sufficiently the precipitate on the filter paper with potassium nitrate solution [2 f)] cooled to 15 °C or lower.

Note ⁽¹⁾ Wash about 15 times until one drop of the washings on blue litmus paper gives no changing to red color.

4.3 Titration Titration shall be carried out as specified in the following procedures.