INTERNATIONAL STANDARD 4158

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Ferrosilicon, ferrosilicomanganese and ferrosilicochromium — Determination of silicon content — Gravimetric method

Ferrosilicium, ferro-silico-manganèse et ferro-silico-chrome — Dosage du silicium — Méthode gravimétrique

First edition — 1978-12-15

UDC 669.15-198 : 543.21 : 546.28

Ref. No. ISO 4158-1978 (E)

Descriptors : ferroalloys, ferrosilicon, chemical analysis, determination of content, silicon, gravimetric analysis.

Price based on 3 pages

FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4158 was developed by Technical Committee ISO/TC 132, *Ferroalloys*, and was circulated to the member bodies in October 1977.

It has been approved by the member bodies of the following countries :

Australia	Italy	Spain
Austria	Japan	Sweden
Bulgaria	Korea, Rep. of	Turkey
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No member body expressed disapproval of the document.

Printed in Switzerland

Ferrosilicon, ferrosilicomanganese and ferrosilicochromium — Determination of silicon content — Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the silicon content of ferrosilicon, ferrosilicomanganese and ferrosilicochromium.

The method is applicable to alloys containing from 8 to 95 % (*m/m*) of silicon.

2 REFERENCE

ISO 3713, *Ferroalloys — Sampling and preparation of samples — General rules.* [1]

3 PRINCIPLE

Transformation of the silicon in a test portion into silicate by oxidizing fusion with sodium peroxide and taking up with acid.

Double dehydration of the silicate by evaporation in a perchloric acid medium, and weighing of the impure silica.

Double hydrofluoric-sulphuric volatilization of the silica, weighing of the residue, and determination, by difference, of the pure silica.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

4.1 Sodium peroxide.

4.2 Sodium carbonate (anhydrous).

4.3 Ammonia solution, ρ 0,91 g/ml.

4.4 Perchloric acid[2], ρ 1,61 g/ml.

4.5 Hydrofluoric acid, ρ 1,14 g/ml.

4.6 Hydrochloric acid, ρ 1,19 g/ml.

4.7 Sulphuric acid, ρ 1,83 g/ml.

4.8 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 + 9.

4.9 Sulphuric acid, ρ 1,83 g/ml, diluted 1 + 1.

4.10 Silver nitrate, 10 g/l solution.

5 APPARATUS

Usual laboratory equipment, and in particular :

5.1 Crucible, capacity 40 ml, of silicon-free iron, vitreous carbon, nickel or zirconium.

5.2 Beakers, for melt dissolution, of polytetrafluoroethylene, stainless steel or high purity nickel.

5.3 Platinum dish, capacity 40 ml.

5.4 Fluted glass funnel, diameter 75 mm.

5.5 Glass beaker, capacity 600 ml or 800 ml.

5.6 Muffle furnace.

5.7 Desiccator.

6 SAMPLE

Use powder which will pass through a sieve with a mesh size of 160 µm, prepared in accordance with ISO 3713.

7 PROCEDURE

7.1 Test portion

For silicon contents less than or equal to 50 % (*m/m*), take a test portion of 0,50 ± 0,000 2 g.

NOTE — For silicon contents less than 25 % (*m/m*), it is possible to use a test portion of 1 g.

1) At present at the stage of draft.

2) Attention is drawn to the hazards associated with perchloric acid when heated to fuming.

For silicon contents more than 50 % (*m/m*), take a test portion of 0,25 ± 0,000 2 g.

7.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents.

7.3 Determination

7.3.1 Transfer the test portion (7.1) to the crucible (5.1) containing 10 to 12 g of the sodium peroxide (4.1). Mix carefully and then, to prevent any loss of the test portion, cover with 3 g of the sodium peroxide (4.1) or 3 g of the sodium carbonate (4.2).

Heat the crucible containing the test portion and flux on an electric hot-plate at 350 to 400 °C until the melt blackens. Holding the crucible in tongs, swirl it over a flame, heating gently at first to avoid deflagration, and then more strongly until the attack is complete. Maintain the product of fusion at red heat for approximately 5 min. Allow the crucible to cool.

7.3.2 Dissolve the melt as specified in either 7.3.2.1 or 7.3.2.2.

7.3.2.1 *Alternative I*

Place the crucible in a beaker (5.2), containing 200 ml of water. Cover with a watch glass. When effervescence has ceased, remove the crucible and wash it with hot water, collecting the washings in the same beaker. Transfer the alkaline solution to a glass beaker (5.5), containing 30 ml of the hydrochloric acid (4.6). Stir to mix the solution, wait for the salts to dissolve and add 100 ml of the perchloric acid (4.4).

Proceed as specified in 7.3.3.

7.3.2.2 *Alternative II*

Cover the crucible and tap it on a hard surface to loosen the cake. Transfer the cake to a clean glass beaker (5.5), containing 100 ml of the perchloric acid (4.4). Fill the crucible with hot water and, after effervescence has ceased in the beaker, add the contents of the crucible to the beaker. Transfer any residue from the crucible to the beaker using a rubber-tipped stirring rod and a minimum of water. Add 30 ml of the hydrochloric acid (4.6).

Proceed as specified in 7.3.3.

7.3.3 Place the beaker on a strongly heated hot-plate until white perchloric fumes are evolved. Continue heating until the fuming residue begins to crystallize and acquires a pasty consistency. Remove from the hot-plate and allow to cool. Take up with 20 ml of the hydrochloric acid (4.6), added gently down the wall of the beaker, and a few millilitres of hot water. Stir and dilute with 250 ml of boiling water. Mix well and allow to settle.

Filter the silica on a 125 mm flat ashless filter paper placed on the fluted glass funnel (5.4). Retain the filtrate. Rinse the beaker with warm water, using a rubber-tipped stirrer. Wash the filter with warm hydrochloric acid (4.8) until the yellow colour due to iron salts disappears, then finally wash several times with hot water until chloride ions are no longer present [verify by means of a spot test with the silver nitrate solution (4.10)]. The filter shall be thoroughly washed, so as to remove any trace of perchloric acid, which would cause flaming up during incineration.

Transfer the filtrate and the washings to the beaker used for the initial dehydration. Evaporate to a volume of about 250 ml. Add 20 ml of the perchloric acid (4.4) and carry out a second dehydration as described in the first paragraph of 7.3.3. Take up, filter (using a fresh filter paper) and wash the precipitate as before, but carrying out the final washing with cold water instead of hot water.

7.3.4 Place the two filters containing the silica precipitates in the platinum dish (5.3). Add 4 drops of the ammonia solution (4.3) to the filters[1]. Dry to remove excess moisture and incinerate in the muffle furnace (5.6), initially at 400 °C maximum. Allow to cool. Add 1 ml of the sulphuric acid (4.9). Evaporate to dryness until sulphuric fumes cease to be evolved and then calcine at 1 100 °C to constant mass. Allow to cool in a desiccator and weigh the dish containing the impure silica.

7.3.5 Moisten the impure silica with a few drops of water. Add about 10 ml of the hydrofluoric acid (4.5) and 2 or 3 drops of the sulphuric acid (4.7). Evaporate to dryness until sulphuric fumes cease to be evolved. Carry out a second volatilization under the same conditions, but decreasing the quantity of hydrofluoric acid to 2 ml.

Calcine in the muffle furnace (5.6) at 1 100 °C to constant mass. Allow to cool in a desiccator and weigh the dish containing the impurities.

1) This reduces the hazard of volatile perchlorates spitting some of the silica from the crucible.

8 EXPRESSION OF RESULTS

The silicon content, expressed as a percentage by mass of the sample, is given by the formula

$$[(m_1 - m_2) - (m_3 - m_4)] \times 0{,}467\,4 \times \frac{100}{m_0}$$

$$= \frac{46{,}74\,[(m_1 - m_2) - (m_3 - m_4)]}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of the dish and impure silica obtained in the determination (7.3.4);

m_2 is the mass, in grams, of the dish and impurities obtained in the determination (7.3.5.);

m_3 is the mass, in grams, of the dish and impure silica obtained in the blank test;

m_4 is the mass, in grams, of the dish and impurities obtained in the blank test;

0,467 4 is the conversion factor from silica to silicon.

9 REPRODUCIBILITY

Experience has shown that the 95 % confidence limits for an experienced operator are :

± 0,30 % for silicon contents exceeding 50 % (*m/m*);

± 0,20 % for silicon contents less than 50 % (*m/m*).

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

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