G-25-21

International Standard 8343

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

Ferronickel — Determination of silicon content — Gravimetric method

Ferro-nickel — Dosage du silicium — Méthode gravimétrique

First edition — 1985-10-15

UDC 669.243.881 : 543.21 : 546.28 **Ref. No. ISO 8343-1985 (E)**

Descriptors: nickel, ferronickel, chemical analysis, determination of content, silicon, gravimetric analysis.

Price based on 2 pages

ISO 8343-1985 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8343 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated..

Printed in Switzerland

Ferronickel — Determination of silicon content — Gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of silicon in ferronickel in the range 0,2 to 4,0 % (*m/m*).

2 Reference

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

3 Principle

Dissolution of a test portion in nitric acid and addition of perchloric acid. Formation of insoluble silica by dehydration in perchloric acid, filtration, and weighing of the calcined precipitate. Volatilization of the silica with hydrofluoric and sulfuric acids, weighing of the residue, determination of the silica by difference and calculation of the silicon content.

4 Reagents

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

4.1 Hydrochloric acid, ϱ_{20} = 1,19 g/ml.

4.2 Hydrochloric acid, ϱ_{20} = 1,19 g/ml, diluted 1 + 9.

4.3 Hydrofluoric acid, ϱ_{20} = 1,14 g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes, producing severe skin burns which are slow to heal. In case of skin contact wash well with water and seek medical advice.

4.4 Nitric acid, ϱ_{20} = 1,41 g/ml, diluted 1 + 1.

4.5 Perchloric acid, ϱ_{20} = 1,61 g/ml (70 % (*m/m*)).

4.6 Sulfuric acid, ϱ_{20} = 1,83 g/ml, diluted 1 + 1.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Beaker, high form, of capacity 600 ml, unetched.

5.2 Platinum crucible, of capacity 40 ml.

5.3 Muffle furnace, capable of being maintained at 1 100 °C.

5.4 Desiccator.

6 Sampling and samples

6.1 Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by the relevant International Standard.

6.2 The laboratory sample normally is in the form of granules, millings or drillings and no further preparation of the sample is necessary.

6.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned by washing with high purity acetone and drying in air.

6.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test portion should be obtained by riffling.

7 Procedure

7.1 Test portion

7.1.1 For a silicon content greater than 1 % (*m/m*) weigh, to the nearest 0,001 g, 2,00 g of the laboratory sample.

7.1.2 For a silicon content between 0,25 and 1 % (*m/m*) weigh, to the nearest 0,002 g, 4,00 g of the laboratory sample.

7.1.3 For a silicon content less than 0,25 % (*m/m*) weigh, to the nearest 0,005 g, 10,00 g of the laboratory sample.

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

WARNING — Fuming perchloric acid is a powerful oxidant and can cause an explosive mixture when in contact with organic materials. All evaporations should be done in fume cupboards suitable for use with perchloric acid.

7.3.1 Transfer the test portion (7.1) to a beaker (5.1), add 50 ml of nitric acid (4.4) and cover with a watch-glass. Heat moderately and when dissolution is almost complete, add 50 ml of perchloric acid (4.5).

NOTE — If a 10 g test portion is used, add the nitric acid with care in small portions to prevent too great an effervescence. After dissolution add 70 ml of perchloric acid.

7.3.2 Heat gently and then progressively more strongly until the appearance of white fumes of perchloric acid. Continue heating until the residue reaches the point of crystallization. Remove from the hotplate and allow to cool. Add 100 ml of near boiling water to dissolve the salts, then add 15 ml of hydrochloric acid (4.1). Dilute to 250 ml with boiling water. Stir and heat for 2 min at just below boiling.

7.3.3 Filter on a 125 mm folded filter paper of medium porosity. Rinse the beaker using hot water and clean with a rubber policeman. Wash the filter and contents with hot hydrochloric acid diluted 1 + 9 (4.2) until the yellow colour of iron salts disappears. Finally wash with hot water until the filtrate is acid free. Discard the filtrate and washings.

WARNING — The filter shall be thoroughly washed to eliminate any trace of perchloric acid which could cause an explosion during incineration.

7.3.4 Place the filter containing the precipitate in a platinum crucible (5.2). Dry on a hotplate or in an oven and ignite in a muffle furnace (5.3) first at low temperature to char the paper. Calcine at 1 100 °C for at least 30 min. Allow to cool in a desiccator (5.4) and weigh the crucible containing the calcined precipitate to the nearest 0,1 mg. Repeat the calcination for 30 min intervals until a constant mass is obtained.

7.3.5 Wet the calcined precipitate with several drops of water. Add about 0,5 ml of sulfuric acid (4.6) followed by about 5 ml of hydrofluoric acid (4.3). Evaporate gently to dryness on a hotplate until sulfuric acid fumes are eliminated. Calcine in a muffle furnace at 1 100 °C for 10 min. Allow to cool in a desiccator and weigh the crucible containing the impurities to the nearest 0,1 mg. Repeat the calcination for 10 min intervals until a constant mass is obtained.

8 Expression of results

8.1 Calculation

The silicon content, expressed as a percentage by mass, in the test portion, is given by the formula

$$0{,}467 \times \frac{m_1 - m_2 - m_3}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the crucible and impure silica;

m_2 is the mass, in grams, of the crucible plus residual impurities;

NOTE — The difference $m_1 - m_2$ is the mass, in grams, of the pure silica volatilized.

m_3 is the mass, in grams, of the pure silica given by the blank test;

0,467 is the conversion factor for silica to silicon.

8.2 Precision

This International Standard was subjected to a limited interlaboratory test programme involving only five laboratories in four countries.

Repeatability and reproducibility were calculated according to the principles of ISO 5725 with the results given in the table.

Table

Silicon content (% (*m/m*))	0,26	1,01	2,56
Standard deviations			
— within laboratory, s_w	0,005	0,022	0,014
— between laboratories, s_b	0,001	0,012	0,027
Repeatability, r	0,013	0,062	0,039
Reproducibility, R	0,014	0,071	0,097

9 Test report

The test report shall include the following information:

a) the reference to the method used;

b) the results of the analysis;

c) the number of independent replications;

d) any unusual features noted during the analysis;

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