International Standard 439

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Steel and cast iron — Determination of total silicon — Gravimetric method

Aciers et fontes — Dosage du silicium total — Méthode gravimétrique

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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 439 was developed by Technical Committee ISO/TC 17, *Steel*.

It was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 439-1969, which had been approved by the member bodies of the following countries :

Australia	Greece	Norway
Austria	Hungary	Poland
Belgium	India	Romania
Canada	Iran	South Africa, Rep. of
Czechoslovakia	Israel	Sweden
Denmark	Italy	Switzerland
Egypt, Arab Rep. of	Japan	Thailand
Finland	Korea, Rep. of	Turkey
France	Netherlands	United Kingdom
Germany, F. R.	New Zealand	USSR

The member body of the following country had expressed disapproval of the document on technical grounds :

USA

Printed in Switzerland

Steel and cast iron — Determination of total silicon — Gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of total silicon in steel and cast iron.

The method is applicable to silicon contents between 0,10 and 8,0 % (m/m).

NOTE — For steels containing tungsten, tantalum, niobium, zirconium, titanium or molybdenum, the results are less accurate than for unalloyed steels.

2 Reference

ISO 377/2, *Selection and preparation of samples and test pieces of wrought steels — Part 2 : Samples and test pieces intended for the determination of the chemical composition.*[1)]

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids. Conversion of silica into hydrated silica by fuming with perchloric acid. Filtration of the hydrated silica, ignition of the impure silica and then weighing. Treatment of the ignited residue with hydrofluoric and sulphuric acids, followed by ignition and weighing.

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 about 1,19 g/ml.

4.2 Hydrochloric acid, diluted 1 + 1.

4.3 Hydrochloric acid, 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.

NOTE — It is also possible to use perchloric acid, ϱ 1,54 g/ml.

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

5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum crucibles, capacity approximately 30 ml.

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

6 Sampling

Sampling shall be carried out in accordance with ISO 377/2, or appropriate national standards for cast iron.

7 Procedure

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

7.1 Test portion

Millings or drillings of a maximum thickness of 0,2 mm.

According to the presumed silicon content, weigh, to the nearest 0,001 g, the following mass (m_0) of the test portion :

a) for Si contents between 0,10 and 0,50 % (m/m) : m_0 about 5 g;

b) for Si contents between 0,50 and 2,5 % (m/m) : m_0 about 2,5 g;

c) for Si contents between 2,5 and 8,0 % (m/m) : m_0 about 1 g.

7.2 Blank test

In parallel with the determination of the content and following the same procedure, carry out a blank test using the same quantities of all reagents.

1) At present at the stage of draft. (Partial revision of ISO/R 377-1964.)

7.3 Determination

7.3.1 Attack of the test portion and precipitation of the silica

Place the test portion (7.1) in a beaker of acid-resistant glass of suitable capacity.

Add 30 ml of the hydrochloric acid (4.1), and then gently heat the beaker covered with a watch-glass until the reaction ceases. Carefully oxidize by adding 15 ml of the nitric acid (4.4). When the fairly violent reaction ceases, rinse the watch-glass with a little hot water and collect the washings in the beaker. Add the perchloric acid (4.6) in accordance with the quantities shown in the table.

Table

Mass of test portion (7.1)	Volume of perchloric acid (4.6) ml	
g	ϱ = 1,67 g/ml	ϱ = 1,54 g/ml
5	60	75
2,5	40	50
1	25	35

Heat the uncovered beaker slightly until the attack is complete and then increase the rate of heating. As soon as the first white perchloric acid fumes appear, cover the beaker with the watch-glass and continue fuming for about 20 min. Allow to cool, carefully moisten with 5 ml of hydrochloric acid (4.1), heat slightly, dilute with 100 ml of water at 70 to 80 °C and heat again until the salts are dissolved (take care not to boil).

7.3.2 Filtration and washing

With a rubber-tipped glass rod, detach any clots of silica that may be adhering to the beaker and filter immediately through a medium-texture filter paper of known low ash content, containing a little filter paper pulp of the same quality.

Wash the beaker and the filter with the hot hydrochloric acid (4.3), transferring the silica on to the filter, and complete the washing, first with hot hydrochloric acid (4.2) and then with cold water until the iron salts are completely eliminated.

NOTE — Wash the filter thoroughly in order to avoid popping and loss of residue due to perchloric acid during ignition.

7.3.3 Recovery of silica in the filtrate

Transfer the filtrate and the washings into the beaker previously used for the attack, evaporate them by heating until dense white fumes of perchloric acid are evolved, and maintain a steady refluxing of acid on the walls of the beaker for about 20 min. Moisten and dilute according to the procedure specified in 7.3.1, then filter through a second medium-texture filter paper of known low ash content and wash according to the procedure specified in 7.3.2.

7.3.4 Ignition, volatilization of the silica and weighing

Put the two filters and their contents together in a platinum crucible (5.1). Heat at between 500 to 600 °C until the filters are completely incinerated, then cover partially the crucible with a platinum cover and ignite in the muffle furnace (5.2) at 1 100 °C for 30 to 45 min, depending on the quantity of silica or, for steel containing molybdenum, until constant mass is obtained.

Allow to cool, add approximately 2 ml of the sulphuric acid (4.7) to the crucible, heat carefully and continue heating until the sulphuric acid fumes are completely eliminated. Then ignite in the muffle furnace at 800 °C to constant mass.

Allow to cool in a desiccator and weigh the crucible and its contents (mass in grams : m_1).

Then moisten the ignited silica with a few drops of the sulphuric acid (4.7), add approximately 5 ml of the hydrofluoric acid (4.5), evaporate to dryness and continue heating until the sulphuric acid fumes are completely eliminated.

NOTE — If niobium, tantalum, titanium or zirconium are present, add 2 ml of the sulphuric acid (4.7) in order to avoid any partial volatilization of the fluorides of these elements.

Complete the ignition in the muffle furnace at 800 °C for 10 min.

Allow to cool in a desiccator, then weigh the crucible and its contents (mass in grams : m_2).

8 Expression of results

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

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

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

where

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

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

m_2 is the mass, in grams, of the crucible and residue after volatilization of the silica, in relation to the determination;

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

m_4 is the mass, in grams, of the crucible and residue after volatilization of the silica in the blank test;

0,467 4 is the Si/SiO_2 coefficient.

Express the results to the second decimal place.

9 Test report

The test report shall include the following particulars :

a) the method used, by reference to this International Standard;

b) the results, and the form in which they are expressed;

c) any unusual features noted during the determination;

d) any operation not specified in this International Standard, or any optional operation which may have influenced the result.