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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 439

CHEMICAL ANALYSIS OF STEEL AND CAST IRON

DETERMINATION OF TOTAL SILICON

(Gravimetric method)

2nd EDITION
January 1969

This second edition supersedes the first edition

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 439, *Chemical analysis of steels – Determination of total silicon (Gravimetric method)*, was drawn up by Technical Committee ISO/TC 17, *Steel*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1957 and led, in 1962, to the adoption of a Draft ISO Recommendation.

In February 1963, this Draft ISO Recommendation (No. 529) was circulated to all the ISO Member Bodies for enquiry. It was approved by 29 Member Bodies. One Member Body opposed the approval of the Draft (U.S.A.).

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in June 1965, to accept it as an ISO RECOMMENDATION.

BRIEF HISTORY RELATING TO THE 2nd EDITION

In January 1965, the Secretariat of the Sub-Committee 1, *Methods of chemical and spectrographic analysis*, of Technical Committee ISO/TC 17 had prepared a first proposal for the revision of ISO Recommendation R 439–1965. The document was adopted in June 1967 as a Draft ISO Recommendation.

In October 1967, this Draft ISO Recommendation (No. 1348) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

One Member Body opposed the approval of the Draft :

U.S.A.

The title of the first edition was replaced by the following one : *Chemical analysis of steel and cast iron – Determination of total silicon (Gravimetric method).*

The Draft Revision was then submitted by correspondence to the ISO Council, which decided, in January 1969, to accept it.

The second edition supersedes the first edition of ISO Recommendation R 439–1965.

ISO Recommendation R 439 January 1969

CHEMICAL ANALYSIS OF STEEL AND CAST IRON

DETERMINATION OF TOTAL SILICON

(Gravimetric method)

1. SCOPE

1.1 This ISO Recommendation describes a gravimetric method of determining total silicon in steel and cast iron.

1.2 The method is applicable to silicon contents between 0.10 and 8.0 %.

NOTE. – For steels containing tungsten, tantalum, niobium, zirconium, titanium or molybdenum, the results are less precise than for non-alloy steels.

2. PRINCIPLE OF THE METHOD

2.1 The silicon present in steel and cast iron, after treatment with hydrochloric and nitric acids, is converted into hydrated silica which is rendered insoluble by fuming with perchloric acid.

2.2 The impure silica is then separated by filtration, calcined and determined by the loss in weight after volatilization with hydrofluoric acid in the presence of sulphuric acid.

3. REAGENTS

Demineralized or distilled water should be used for the preparation of reagents and throughout the procedure.

3.1 *Hydrochloric acid* (d = 1.19 approximately).

3.2 *Hydrochloric acid* (d = 1.10 approximately).
Dilute 500 ml of hydrochloric acid (3.1) to 1000 ml with water.

3.3 *Hydrochloric acid* (d = 1.01 approximately).
Dilute 50 ml of hydrochloric acid (3.1) to 1000 ml with water.

3.4 *Nitric acid* (d = 1.33 approximately).
Dilute 750 ml of nitric acid (d = 1.42 approximately) to 1000 ml with water.

3.5 *Perchloric acid*, 70 % (d = 1.67 approximately).

NOTE. – It is also possible to use perchloric acid, 60 % (d = 1.54 approximately). (1000 ml of perchloric acid, 70 %, is equivalent to 1270 ml of perchloric acid, 60 %).

3.6 *Sulphuric acid* (d = 1.48 approximately).
Carefully add 50 ml of sulphuric acid (d = 1.83 approximately) to 50 ml of water, cool and dilute to 100 ml with water.

3.7 *Hydrofluoric acid*, 40 % (d = 1.14 approximately).

4. APPARATUS

Ordinary laboratory equipment.

5. SAMPLING

In accordance with ISO Recommendation R 377, *Selection and preparation of samples and test pieces for wrought steel*, or the appropriate national standards for cast iron.

6. PROCEDURE

6.1 Test portion

Millings or drillings of a maximum thickness of 0.2 mm.

Weigh the test portion in accordance with the quantities shown in Table 1.

TABLE 1

Assumed Si content	Mass of test portion
%	g
0.10 to 0.50	5 ± 0.001
0.50 to 2.5	2.5 ± 0.001
2.5 to 8.0	1 ± 0.001

6.2 Blank test

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

6.3 Determination

6.3.1 *Attack of the test portion and precipitation of the silica.* Place the test portion in a beaker of suitable size made of acid resistant glass.

Add 30 ml of hydrochloric acid (3.1), and then gently heat the beaker covered with a watch-glass until the reaction ceases. Carefully oxidize by adding 15 ml of nitric acid (3.4). At the end of the fairly energetic reaction which occurs, rinse the watch-glass with a little hot water, collecting the washings in the beaker. Add perchloric acid (3.5) in accordance with the quantities shown in Table 2.

Heat the uncovered beaker slightly until the attack is complete and then increase the rate of heating. When the first white perchloric acid fumes appear, cover the beaker with the watch-glass and continue fuming for about 20 minutes (see Note 8.1). Allow to cool, carefully moisten with 5 ml of concentrated hydrochloric acid (3.1), heat slightly, dilute with 100 ml of water at 70 to 80 °C and heat again until the salts are dissolved (taking care not to boil).

TABLE 2

Mass of test portion	Volume of perchloric acid (3.5)	
	70 % solution	60 % solution
g	ml	ml
5	60	75
2.5	40	50
1	25	35