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**STEEL AND CAST IRON - DETERMINATION OF
TOTAL SILICON CONTENT - REDUCED
MOLYBDOSILICATE SPECTROPHOTOMETRIC
METHOD.
PART 2: SILICON CONTENT BETWEEN
0.01 AND 0.05%**

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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Steel and iron — Determination of total silicon content — Reduced molybdosilicate spectrophotometric method —

Part 2:

Silicon contents between 0,01 and 0,05 %

*Aciers et fontes — Dosage du silicium total — Méthode spectrophotométrique au
molybdosilicate réduit —*

Partie 2: Teneurs en silicium comprises entre 0,01 et 0,05 %

ISO 4829-2 : 1988 (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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization

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 4829-2 was prepared by Technical Committee ISO/TC 17, *Steel*.

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.

ISO 4829 consists of the following parts, under the general title *Steel and iron — Determination of total silicon content — Reduced molybdosilicate spectrophotometric method*:

- *Part 1: Silicon contents between 0,05 and 1,0 %*
- *Part 2: Silicon contents between 0,01 and 0,05 %*

Steel and iron — Determination of total silicon content — Reduced molybdosilicate spectrophotometric method —

Part 2: Silicon contents between 0,01 and 0,05 %

1 Scope and field of application

This International Standard specifies a spectrophotometric method using reduced molybdosilicate for the determination of total silicon in steel and iron.

The method is applicable to silicon contents between 0,01 and 0,05 % (*m/m*) in steel and iron.

2 References

ISO 377, *Wrought steel — Selection and preparation of samples and test pieces*.

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648, *Laboratory glassware — One-mark pipettes*.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

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

3 Principle

Dissolution of a test portion in a hydrochloric acid/nitric acid mixture.

Fusion of the acid-insoluble residue with sodium peroxide.

Formation of the oxidized molybdosilicate (yellow) complex in weak acid solution.

Selective reduction of the molybdosilicate complex to a blue complex with ascorbic acid, after increasing the sulfuric acid concentration and adding oxalic acid to prevent the interference of phosphorus, arsenic and vanadium.

Spectrophotometric measurement of the reduced blue complex at a wavelength of about 810 nm.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

Reagents supplied in glass bottles, once opened, may absorb moisture and become reactive to glassware. Alkaline reagents, e.g. sodium carbonate and sodium peroxide, are particularly susceptible. To avoid the risk of significant contamination arising from this source, it is recommended that only freshly opened bottles of all reagents be used for the preparation of reagent solutions.

In addition, only water prepared by distillation shall be used for the preparation of reagent solutions and throughout the procedure. Water demineralized by ion-exchange shall not be used as it may contain significant amounts of colloidal silica.

To avoid adventitious contamination, the water shall be prepared, as required, for the specific purpose, and collected in polypropylene containers for immediate use.

All solutions shall be freshly prepared and stored in polypropylene or polytetrafluoroethylene containers.

4.1 Pure iron, silicon content less than 2 µg/g.

4.2 Sodium peroxide, particle size of minus 500 µm.

4.3 Sulfuric acid, diluted 1 + 3.

To 600 ml of water add cautiously, with stirring, 250 ml of sulfuric acid, ρ about 1,84 g/ml. Cool, dilute to 1 000 ml and mix.

4.4 Sulfuric acid, diluted 1 + 19.

To 800 ml of water add cautiously, with stirring, 50 ml of sulfuric acid, ρ about 1,84 g/ml. Cool, dilute to 1 000 ml and mix.

ISO 4829-2 : 1988 (E)

4.5 Hydrochloric acid/nitric acid mixture.

Add 180 ml of hydrochloric acid, ρ about 1,19 g/ml, and 65 ml of nitric acid, ρ about 1,40 g/ml, to 500 ml of water. Cool, dilute to 1 000 ml and mix.

4.6 Ascorbic acid, 20 g/l solution.

Prepare this solution immediately before use.

4.7 Oxalic acid, solution.

Dissolve 5 g of oxalic acid dihydrate ($C_2H_2O_4 \cdot 2H_2O$) in water, dilute to 100 ml and mix.

4.8 Hydrogen peroxide, solution.

Dilute 200 ml of hydrogen peroxide, 300 g/l, to 1 000 ml and mix.

4.9 Potassium permanganate, 22,5 g/l solution.

Filter before use.

4.10 Sodium molybdate, solution.

Dissolve 2,5 g of sodium molybdate dihydrate ($Na_2MoO_4 \cdot 2H_2O$) in 50 ml of water and filter through a medium-texture filter paper. Immediately before use, add 15 ml of sulfuric acid (4.4), dilute to 100 ml and mix.

4.11 Silicon, standard solution.

4.11.1 Silicon stock solution, containing 1 g of Si per litre.

Weigh, to the nearest 0,1 mg, 2,139 3 g of freshly calcined high-purity silica ($> 99,9\% SiO_2$) and transfer to a platinum crucible. (The high-purity silica shall be calcined for 1 h at 1 100 °C and cooled in a desiccator immediately before use.) Mix thoroughly with 16 g of anhydrous sodium carbonate and fuse at 1 050 °C for 30 min. Extract the fusion product with 100 ml of water in a polypropylene or polytetrafluoroethylene beaker (see the note). Transfer the extract, which should contain no trace of residue, to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Transfer immediately to a well-stoppered polytetrafluoroethylene bottle for storage.

1 ml of this stock solution contains 1 mg of Si.

NOTE — Extraction of the fusion product may require gentle heating.

4.11.2 Silicon standard solution, containing 20 mg of Si per litre.

Transfer 10,0 ml of silicon stock solution (4.11.1) to a 500 ml one-mark volumetric flask. Dilute to the mark and mix. Transfer immediately to a well-stoppered polytetrafluoroethylene bottle for immediate use.

1 ml of this standard solution contains 20 µg of Si.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Beakers and lids, of polypropylene or polytetrafluoroethylene.

5.2 Crucibles, of zirconium metal, of 50 ml capacity.

NOTE — Vitreous carbon crucibles may be used as alternatives to zirconium metal crucibles.

5.3 Volumetric glassware.

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042, as appropriate.

The use of glassware shall be restricted to a minimum of contact time and borosilicate glass shall be used as far as possible.

5.4 Spectrophotometer.

The spectrophotometer shall be equipped to measure absorbance with a spectral band width of 10 nm or less at a wavelength of 810 nm. Wavelength measurement shall be accurate to ± 2 nm, as measured by the absorption maximum of a didymium filter at 803 nm, or other suitable calibration method. The absorption measurement for the solution of maximum absorbance shall have a repeatability, expressed as a relative deviation, of $\pm 0,3\%$ or better.

6 Sampling

Carry out sampling in accordance with ISO 377 or appropriate national standards for iron.

7 Procedure

7.1 Test portion

Weigh, to the nearest 1 mg, $0,50 \pm 0,01$ g (m) of test sample in the form of fine chips, turnings, millings or filings.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out two blank tests using the same quantities of all the reagents but using $0,50 \pm 0,01$ g of pure iron (4.1) instead of the test portion.

It is essential that blank values be controlled at consistently low and reproducible values. It is recommended that duplicate blanks be run with each batch of tests and the mean value be used as the basis for calculation. High or divergent blank values should be considered unacceptable and steps should be taken to trace the source of contamination by checking the quality of the water and of individual reagents before proceeding further. In particular, sodium peroxide and potassium permanganate require careful selection as some grades of these reagents give high blank values. It is recommended that the blank reading

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