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International Standard



4829/1

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**Steel and cast iron — Determination of total silicon content — Reduced molybdsilicate spectrophotometric method —  
Part 1 : Silicon contents between 0,05 and 1,0 %**

*Aciers et fontes — Dosage du silicium total — Méthode spectrophotométrique au molybdsilicate réduit — Partie 1 : Teneurs en silicium entre 0,05 et 1,0 %*

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

International Standard ISO 4829/1 was prepared by Technical Committee ISO/TC 17, *Steel*.

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

## Part 1 : Silicon contents between 0,05 and 1,0 %

### 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 cast iron.

The method is applicable to silicon contents between 0,05 and 1,0 % (*m/m*) in steel and cast iron soluble in one or the other of the alternative acid mixtures.

### 2 Reference

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

### 3 Principle

Dissolution of a test portion in an acid mixture appropriate to the alloy composition.

Fusion of the acid-insoluble residue with sodium peroxide.

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

Selective reduction of the molybdosilicate complex to reduced 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 and only distilled water or water of equivalent purity.

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

**4.1 Pure iron**, silicon content less than 5 µg/g.

**4.2 Sodium peroxide**, particle size — 500 µm.

**4.3 Nitric acid.**

Slowly add 150 ml of nitric acid,  $\rho$  approximately 1,40 g/ml, to 600 ml of water. Cool, dilute to 1 000 ml and mix.

**4.4 Sulfuric acid.**

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

**4.5 Sulfuric acid.**

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

**4.6 Hydrochloric acid/nitric acid mixture.**

Add 180 ml of hydrochloric acid,  $\rho$  about 1,19 g/ml, and 65 ml of nitric acid,  $\rho$  about 1,40 g/ml, to 500 ml of water, cool, dilute to 1 000 ml and mix.

**4.7 Sulfuric acid/nitric acid mixture.**

To 500 ml of water add cautiously, with stirring, 35 ml of sulfuric acid,  $\rho$  about 1,84 g/ml, and 45 ml of nitric acid,  $\rho$  about 1,40 g/ml. Cool, dilute to 1 000 ml and mix.

**4.8 Ascorbic acid**, 20 g/l solution.

Prepare this solution immediately before use.

**4.9 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.

**4.10 Hydrogen peroxide.**

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