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Steel and irons — 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 %*

[Revision of first edition (ISO 4829-2:1988)]

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ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 4829-2 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

This second/third/... edition cancels and replaces the first/second/... edition (ISO 4829-2:1988), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

ISO 4829 consists of the following parts, under the general title *Steels — 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 %*

Steels — Determination of total silicon content - Reduced molybdosilicate spectrophotometric method — Part 2: Silicon contents between 0,01 and 0,05 %

1 Scope

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

The method is applicable to silicon contents between 0,01 and 0,05 % (mass fraction) in steels.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

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

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*.

3 Principle

Dissolution of a test portion in a hydrochloric/nitric acids 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 sulphuric 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 grade 2 water as specified in ISO 3696. Water demineralized by ion-exchange shall not be used as it may contain significant amounts of colloidal silica.

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.

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 Sulphuric acid, ρ approximately 1,84 g/ml, diluted 1 + 3

To 600 ml of water while stirring, add cautiously, 250 ml of sulphuric acid, ρ approximately 1,84 g/ml. Cool, dilute to 1 litre and mix.

4.4 Sulphuric acid, ρ approximately 1,84 g/ml, diluted 1 + 19

To 800 ml of water while stirring, add cautiously, 50 ml of sulphuric acid, ρ approximately 1,84 g/ml. Cool, dilute to 1 litre and mix.

4.5 Hydrochloric/nitric acids mixture

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

4.6 Ascorbic acid, 20 g/l solution

This solution shall be freshly prepared.

4.7 Oxalic acid, 50 g/l solution

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

4.8 Hydrogen peroxide, 60 g/l solution

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

4.9 Potassium permanganate, 22,5 g/l solution

This solution shall be filtered before use.

4.10 Sodium molybdate, 25 g/l solution

Dissolve 2,5 g of sodium molybdate di-hydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 50 ml of water and filter through a medium-texture filter paper.

Immediately before use, add 15 ml of sulphuric acid (4.4), dilute to 100 ml and mix.

4.11 Silicon standard solution, 1 g/l

Prepare a 1 g/l silicon standard solution by using one of the procedures described in 4.11.1 or in 4.11.2.

4.11.1 Preparation with silica

Weigh to the nearest 0,1 mg, 2,139 3 g of freshly calcined high-purity silica ($> 99,9\% \text{SiO}_2$) and transfer to a platinum crucible.

NOTE 1 The high-purity silica shall be calcined for 1 hour at $1\,100\text{ }^\circ\text{C}$ and cooled in a desiccator immediately before use.

Mix thoroughly with 16 g of anhydrous sodium carbonate and fuse at $1\,050\text{ }^\circ\text{C}$ for 30 minutes. Extract the fusion product with 100 ml of water in a polypropylene or polytetrafluoroethylene beaker

NOTE 2 Extraction of the fusion product may require gentle heating.

Transfer the extract, which should contain no trace of residue, into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix. Transfer immediately into a well-stoppered polytetrafluoroethylene bottle for storage.

1 ml of this standard solution contains 1 mg of silicon.

4.11.2 Preparation with ammonium hexafluorosilicate

Dry several grams of ammonium hexafluorosilicate $[(\text{NH}_4)_2\text{SiF}_6]$ in an oven at $105\text{ }^\circ\text{C}$ to $110\text{ }^\circ\text{C}$ for about 1 hour and cool to room temperature in a desiccator.

Using a suitable plastic beaker, dissolve 3,171 g of the dried product in hot water ($\sim 80\text{ }^\circ\text{C}$).

Allow to cool and transfer the solution quantitatively into a polyethylene 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this standard solution contains 1 mg of silicon.

4.12 Silicon standard solution containing, 20 mg/l

Transfer 10,0 ml of one of the silicon standard solutions (4.11.1 or 4.11.2) into 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 silicon.

5 Apparatus

Ordinary laboratory apparatus and:

5.1 Beakers and lids, of polypropylene or polytetrafluoroethylene

5.2 Zirconium crucibles, of 50 ml capacity

NOTE Vitreous carbon crucibles may be used as alternative to zirconium crucibles.

5.3 Volumetric glassware

All volumetric glassware shall be class A, in accordance with ISO 385, 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 bandwidth of 10 nm or less, at a wavelength of 810 nm, with cell of 4 cm optical path length. Wavelength adjustment shall be accurate to ± 2 nm, as measured by the maximum absorption 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 14284 or appropriate national standards for iron.

7 Procedure

7.1 Test portion

Weigh, to the nearest 1 mg, $0,50 \pm 0,01$ g 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 does not exceed 0,050 as absorbance, which is equivalent to 0,008 % (mass fraction) silicon with a 4 cm optical path length.

7.3 Determination

7.3.1 Dissolution of the test portion

Introduce the test portion (7.1) into a 250 ml polypropylene or polytetrafluoroethylene beaker (5.1). Add 85 ml of hydrochloric/nitric acids mixture (4.5), cover with a lid (5.1) and heat gently to dissolve the test portion, without incurring significant loss of volume.

When effervescence has ceased, filter the solution through a hardened close-texture filter paper of known, low ash content and collect the filtrate in a 500 ml beaker. Rinse the beaker with 20 ml of hot water, remove adherent particles with a rubber tipped rod, and filter the rinsings through the same filter paper. Wash the filter paper several times with 20 ml quantities of hot water. Keep the filtrate for use in the procedure given in 7.3.2.

7.3.2 Treatment of insoluble residue

Transfer the paper and residue into a zirconium crucible (5.2) and ignite at low temperature until carbonaceous matter has been removed, and then ignite in a furnace at 600 °C. Cool and mix the residue with 0,25 g of sodium peroxide (4.2). Cover with an additional 0,25 g of sodium peroxide and heat in the furnace at 600 °C for 10 minutes. Cool, add 15 ml of water, cover the crucible with a lid and allow the reaction to subside. Add 15 ml of sulphuric acid (4.4), stir to dissolve any precipitate and add to the filtrate obtained by the procedure given in 7.3.1. Rinse the crucible and lid with water and add the rinsings to the filtrate.

7.3.3 Preparation of the test solution

Dilute the solution from 7.3.2 to approximately 300 ml and cool. Add 5 ml of potassium permanganate solution (4.9), followed, if necessary, by further dropwise additions until a definite pink colour is obtained which persists for at least 1 minute.

NOTE 1 Add the same amount of potassium permanganate solution (4.9) to the blank test solution (7.2) as is required to produce the pink colour in the test solution.

Heat to boiling and boil gently for 2 minutes. If precipitation of manganese dioxide occurs, add hydrogen peroxide (4.8) dropwise until the precipitate is just dissolved and boil gently for 5 minutes.

NOTE 2 Treat the blank test solution exactly as for the test solution, even though no precipitation of manganese dioxide occurs.

Cool, transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer immediately into a polypropylene or polytetrafluoroethylene container.

7.3.4 Development of the colour

Pipette two 20,0 ml aliquots from the test solution (7.3.3) and two 20 ml aliquots of the blank test solution (7.2) into separate 50 ml borosilicate one-mark volumetric flasks. In each case, one aliquot is for the test and the other is for the compensating solution.

Solutions of test samples containing niobium or tantalum will give finely divided precipitates on dilution. Allow the precipitate to settle and, immediately prior to taking aliquots, pour the supernatant liquid through a dry close-texture filter paper into a dry vessel. Discard the first few millilitres.

At a temperature range between 15 and 25 °C max., treat each test and compensating solution as stated below, using pipettes for all reagent solution additions.

a) Test solution

Add, in the following order, shaking after each addition:

- 10,0 ml of sodium molybdate solution (4.10) and allow to stand for 20 min;
- 5,0 ml of sulphuric acid (4.3);
- 5,0 ml of oxalic acid solution (4.7);
- immediately, 5,0 ml of ascorbic acid solution (4.6).