
International Standard



6382

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General method for determination of silicon content — Reduced molybdosilicate spectrophotometric method

Méthode générale de dosage du silicium — Méthode spectrophotométrique au molybdosilicate réduit

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 6382 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1979.

It has been approved by the member bodies of the following countries :

Australia	Egypt, Arab Rep. of	Poland
Austria	France	Portugal
Belgium	Germany, F.R.	Romania
Brazil	Hungary	South Africa, Rep. of
Bulgaria	India	Switzerland
Canada	Italy	United Kingdom
China	Korea, Rep. of	USSR
Czechoslovakia	Netherlands	Yugoslavia

No member body expressed disapproval of the document.

This International Standard was also approved by the International Union of Pure and Applied Chemistry (IUPAC).

General method for determination of silicon content — Reduced molybdosilicate spectrophotometric method

WARNING — Attention is drawn to the dangers involved in the use of some of the reagents (see the corresponding notes).

1 Scope

This International Standard specifies a general method for the determination of silicon by spectrophotometric measurement of reduced molybdosilicate.

It describes a technique for the determination of the content of silicon in a solution. Reference should be made, for the preparation of the test solution, to the International Standard relating to the product to be analysed, which should indicate the modifications necessary to make the method applicable to that product.

2 Field of application

The method is applicable to the determination of quantities of silicon, expressed as SiO_2 , between 2 and 200 μg in the aliquot portion of the test solution taken for the determination.

The ions, elements and compounds given in the table in annex A do not interfere with the determination when they are present separately up to the limits indicated. In certain cases, this table gives guidance on the treatment to be applied to reduce or suppress interferences.

3 Principle

Depolymerization of any polymerized silica by treatment with sodium fluoride in the presence of hydrochloric acid.

Formation of the oxidized (yellow) molybdosilicate at a pH of $1,1 \pm 0,2$ in the presence of boric acid to suppress interference by fluoride ion.

Selective reduction of this complex in the presence of oxalic acid and in a sulphuric acid medium of acidity sufficiently high to eliminate interference by phosphates.

Spectrophotometric measurement of the blue-coloured complex at the wavelength corresponding to maximum absorption (about 800 nm).

4 Reagents

During the analysis, use only reagents of recognized analytical grade, as far as possible silica-free, and water which has been double-distilled from a quartz apparatus or water of equivalent purity.

NOTE — Demineralized water is not suitable.

Store all the reagents in flasks of material free from silica (for example, polyethylene).

4.1 Sulphuric acid, approximately 400 g/l solution.

Add, taking all necessary precautions, 240 ml of sulphuric acid, ρ 1,84 g/ml [98 % (*m/m*) solution], to 500 ml of water, dilute to 1 000 ml and mix.

4.2 Hydrochloric acid, approximately 150 g/l solution.

Dilute, taking all necessary precautions, 340 ml of hydrochloric acid, ρ 1,19 g/ml [38 % (*m/m*) solution], to 1 000 ml with water.

4.3 Boric acid, solution saturated at room temperature (approximately 48 g/l).

4.4 Oxalic acid dihydrate $[(\text{COOH})_2 \cdot 2\text{H}_2\text{O}]$, 100 g/l solution.

NOTE — Harmful in contact with skin and if swallowed. Avoid contact with skin and eyes.

4.5 Sodium fluoride, 20 g/l solution.

Store this solution in a flask of material free from silica.

NOTE — Toxic by inhalation, in contact with skin and if swallowed. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

In case of feeling unwell, seek medical advice (show the label where possible).

4.6 Disodium molybdate dihydrate, 140 g/l solution.

Dissolve, in a polyethylene beaker, 35 g of disodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 200 ml of water at about 50 °C. Allow to cool to ambient temperature, transfer to a 250 ml one-mark volumetric flask and dilute to the mark. Transfer the solution to a flask of material free from silica and, if necessary, filter before use.

Prepare this solution at the time of use.