
INTERNATIONAL STANDARD



984

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Sodium hydroxide for industrial use — Determination of silica content — Reduced silico-molybdic complex photometric method

Hydroxyde de sodium à usage industriel — Dosage de la silice — Méthode photométrique au complexe silicomolybdique 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 984 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1973.

It has been approved by the Member Bodies of the following countries :

Austria	India	Spain
Belgium	Ireland	Switzerland
Bulgaria	Italy	Thailand
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
Egypt, Arab Rep. of	Poland	U.S.S.R.
France	Portugal	Yugoslavia
Germany	Romania	
Hungary	South Africa, Rep. of	

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

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 984-1969, of which it constitutes a technical revision.

Sodium hydroxide for industrial use – Determination of silica content – Reduced silico-molybdic complex photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reduced silico-molybdic complex photometric method for the determination of the silica content of sodium hydroxide for industrial use. The method is applicable to products having silica (SiO_2) contents exceeding 10 mg/kg.

2 REFERENCE

ISO 3195, *Sodium hydroxide for industrial use – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.*¹⁾

3 PRINCIPLE

Formation of the yellow oxidized silico-molybdic complex at pH $1,1 \pm 0,2$, in the presence of boric acid to suppress interference by fluorides.

Selective reduction of this complex with a mixture of amino-naphthol sulphonic acid (4-amino-3-hydroxynaphthalene-1-sulphonic acid), sodium metabisulphite and sodium sulphite, in the presence of oxalic acid and in a strongly acid medium so as to suppress interference by phosphates. Photometric measurement of the blue-coloured complex at a wavelength of about 795 nm.

4 REAGENTS

During the analysis use only reagents of recognized analytical reagent grade and only demineralized water. Store all the reagents in polyethylene bottles.

4.1 Sulphuric acid, approximately 9 N solution.

4.2 Hydrochloric acid, approximately 2 N solution.

4.3 Boric acid, saturated solution (about 48 g/l).

4.4 Oxalic acid, 100 g/l solution.

4.5 Sodium molybdate dihydrate [$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$], 140 g/l solution.

Dissolve 35 g of this reagent in 200 ml of water at about 50 °C in a polyethylene beaker. Cool to room temperature, transfer to a 250 ml one-mark volumetric flask, dilute to the mark and mix. Transfer to a polyethylene bottle.

If necessary, filter the solution before use.

4.6 Reducing solution

4.6.1 Dissolve 7 g of anhydrous sodium sulphite in 50 ml of water. Then add 1,5 g of 4-amino-3-hydroxynaphthalene-1-sulphonic acid and dissolve by trituration.

4.6.2 Dissolve 90 g of anhydrous sodium metabisulphite in 900 ml of water.

Mix the two solutions (4.6.1) and (4.6.2) and dilute to 1 000 ml. Filter, if necessary, store the solution in a cool place away from direct sunlight and renew it every 15 to 20 days.

4.7 Sodium chloride, 70 g/l solution.

4.8 Silica, standard solution corresponding to 0,500 g of SiO_2 per litre.

In a platinum crucible, weigh, to the nearest 0,001 g :

– either 0,500 g of silica (SiO_2) produced from silicic acid (H_2SiO_3) calcined at 1 000 °C to constant mass and cooled in a desiccator;

– or 0,500 g of pure quartz, finely ground and previously calcined for 1 h at 1 000 °C and cooled in a desiccator.

Add 5 g of anhydrous sodium carbonate to the crucible. Mix well, preferably with a platinum spatula, and fuse carefully. Allow to cool, add warm water, heat moderately until completely dissolved, cool, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Transfer immediately to a polyethylene bottle.

1 ml of this standard solution contains 0,500 mg of SiO_2 .

1) At present at the stage of draft.