
INTERNATIONAL STANDARD



3198

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Sodium hydroxide for industrial use — Determination of sulphur compounds — Method by reduction and titrimetry

Hydroxyde de sodium à usage industriel — Dosage des composés soufrés — Méthode par réduction et titrimétrie

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Descriptors : sodium hydroxide, chemical analysis, determination of content, sulphates, sulphur, volumetric analysis.

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 3198 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in January 1974.

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

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

No Member Body expressed disapproval of the document.

Sodium hydroxide for industrial use – Determination of sulphur compounds – Method by reduction and titrimetry

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reduction and titrimetric method for the determination of sulphur compounds in sodium hydroxide for industrial use. The method is applicable to products of which the content of sulphur compounds, expressed as sulphate (SO₄), is equal to or greater than 5 mg/kg.

2 REFERENCE

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

3 PRINCIPLE

Reduction of the sulphur compounds to hydrogen sulphide by heating with a mixture of hydriodic acid and hypophosphorous acid.

Absorption of the hydrogen sulphide, entrained in a current of oxygen-free nitrogen, in a mixture of sodium hydroxide and acetone.

Titration of the sulphide with standard volumetric mercury(II) nitrate solution in the presence of dithizone as indicator.

4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

4.1 Acetone.

4.2 Nitrogen, oxygen-free.

4.3 Sodium hydroxide, approximately 1 N solution.

4.4 Potassium permanganate and mercury(II) chloride solution, to purify the nitrogen (4.2).

Dissolve first 2 g of potassium permanganate (KMnO₄) and then 7 g of mercury(II) chloride (HgCl₂), in 100 ml of water and filter the solution.

4.5 Pyrogallol solution, to purify the nitrogen (4.2).

Dissolve 15 g of pyrogallol in 25 ml of water and add, while cooling, 150 ml of a 30 % (m/m) potassium hydroxide solution.

4.6 Reduction solution

Place in the flask (J) of the apparatus (5.3), shaking after each addition :

– 50 ml of hypophosphorous acid (H₃PO₂), ρ approximately 1,21 g/ml, about 50 % (m/m) solution;

– 100 ml of hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (m/m) solution;

– 120 ml of hydriodic acid, ρ approximately 1,97 g/ml, about 67 % (m/m) solution.

Assemble the flask and the reflux condenser (K) then, while passing a slow flow of the nitrogen (4.2), boil for 4 h.

Cool to room temperature under a flow of the nitrogen (4.2) and store the reagent away from direct sunlight in an amber glass bottle previously flushed with the nitrogen (4.2) and fitted with a ground glass stopper.

4.7 Sodium sulphate, 0,001 M standard reference solution.

Weigh, to the nearest 0,000 1 g, 0,142 0 g of anhydrous sodium sulphate, previously dried at 110 °C and cooled in a desiccator. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

1 ml of this solution corresponds to 96 μ g of SO₄.

4.8 Mercury(II) nitrate, 0,05 M standard volumetric solution (= 0,1 N).

Dissolve 10,85 \pm 0,01 g of mercury(II) oxide (HgO) in 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (m/m) solution, dilute to 1 000 ml with water and mix.

NOTE — The strength of the solution thus prepared is sufficiently exact for the small quantities of sulphur compounds to be determined and standardization is therefore unnecessary.

In most laboratories, however, a precisely 0,1 N standard volumetric solution, commonly used for the mercurimetric determination of chlorides, will be available.

4.9 Mercury(II) nitrate, 0,001 M standard volumetric solution (= 0,002 N).

Dilute 20,00 ml of the standard volumetric mercury(II) nitrate solution (4.8) to the mark in a 1 000 ml one-mark volumetric flask.

Prepare this solution at the time of use.

4.10 Dithizone, 0,5 g/l solution in the acetone (4.1).

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Microburette, capacity 5 ml, graduated in 0,01 ml (G).

5.2 Apparatus for reduction and distillation of which all the components are fitted together with ground glass joints. A typical apparatus is shown in figure 1 and comprises :

5.2.1 Wash bottles (A and B), Drechsel type, capacity 50 ml.

5.2.2 Conical flasks (C), capacity 100 ml.

5.2.3 Reflux condenser (D).

5.2.4 Absorption vessel (E), internal diameter 25 mm and height 100 mm.

5.2.5 Dropping funnel (F), capacity 50 ml.

5.2.6 Stopcocks (1, 2, 3 and 4).

5.3 Apparatus for preparation of the reduction solution (4.6).

A typical apparatus is shown in figure 2, and comprises :

5.3.1 Flask, three-necked (J), capacity 500 ml.

5.3.2 Reflux condenser (K).

5.3.3 Nitrogen supply tube (L).

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,01 g, a mass of the test sample (see ISO 3195), solid or liquid, not exceeding 5 or 10 g respectively, into a dried conical flask (C). Fit with a stopper.

6.2 Check test

The purpose of this test is to check the gas-tightness and functioning of the apparatus (reduction of sulphur compounds and quantitative recovery of the hydrogen sulphide liberated).

Transfer in turn :

– to the wash bottle (A), 25 ml of the potassium permanganate and mercury(II) chloride solution (4.4);

– to the wash bottle (B), 25 ml of the pyrogallol solution (4.5);

– to the absorption vessel (E), 5 ml of the sodium hydroxide solution (4.3), 5 ml of the acetone (4.1) and 0,1 ml of the dithizone solution (4.10);

– to a dried conical flask (C), 2,00 ml of the standard reference sodium sulphate solution (4.7) and a few glass beads;

– to the dropping funnel (F), 50 ml of the reducing solution (4.6);

– to the microburette (G) the standard volumetric mercury(II) nitrate solution (4.9).

Assemble the apparatus taking care to smear the ground joints with a silicone grease and to tighten them with suitable spring-clips in order to ensure perfect gas-tightness. Pass the nitrogen (4.2) through stopcocks (2) and (4), at a speed of about 2 bubbles per second. After 5 min, run the mercury(II) nitrate solution (4.9), drop by drop, into the absorption vessel (E) until the indicator changes from yellow to red.

Open stopcocks (1) and (3) to allow the reducing solution (4.6) to run into the conical flask (C), leaving a few millimetres of liquid in the dropping funnel (F), then close the two stopcocks.

Start the condenser (D) and, while continuing the flow of nitrogen, boil the solution gently in the conical flask (C) for at least 30 min. The presence of hydrogen sulphide is shown by a colour change of the indicator from red to yellow.

Stop the boiling and, while maintaining the flow of nitrogen, titrate the sulphide in the absorption vessel (E) with the standard volumetric mercury(II) nitrate solution (4.9) contained in the microburette (G) until the indicator changes from yellow to red.

The result of the check test is considered satisfactory if the volume of the standard volumetric mercury(II) nitrate solution (4.9) used for the titration is between 1,90 and 2,10 ml, after subtraction of the blank test result (6.3). Otherwise check the apparatus for leaks.

6.3 Blank test

Carry out a blank test under the same conditions as the check test (6.2), but using 2,00 ml of water instead of 2,00 ml of the standard reference sodium sulphate solution (4.7).

6.4 Determination

Carry out the determination under the same conditions as the check test (6.2), using the test portion (6.1) instead of the 2,00 ml of the standard reference sodium sulphate solution (4.7).

7 EXPRESSION OF RESULTS

The content of sulphur compounds, expressed as milligrams of sulphate (SO₄) per kilogram, is given, by the formula

$$(V_1 - V_0) \times \frac{1}{1\,000} \times \frac{1\,000}{m} \times 96 = \frac{(V_1 - V_0) \times 96}{m}$$

where

V_0 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.9) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.9) used for the determination;

m is the mass, in grams, of the test portion (6.1);

96 is the mass, in micrograms, of SO₄ corresponding to 1 ml of the standard volumetric mercury(II) nitrate solution (4.9).

8 TEST REPORT

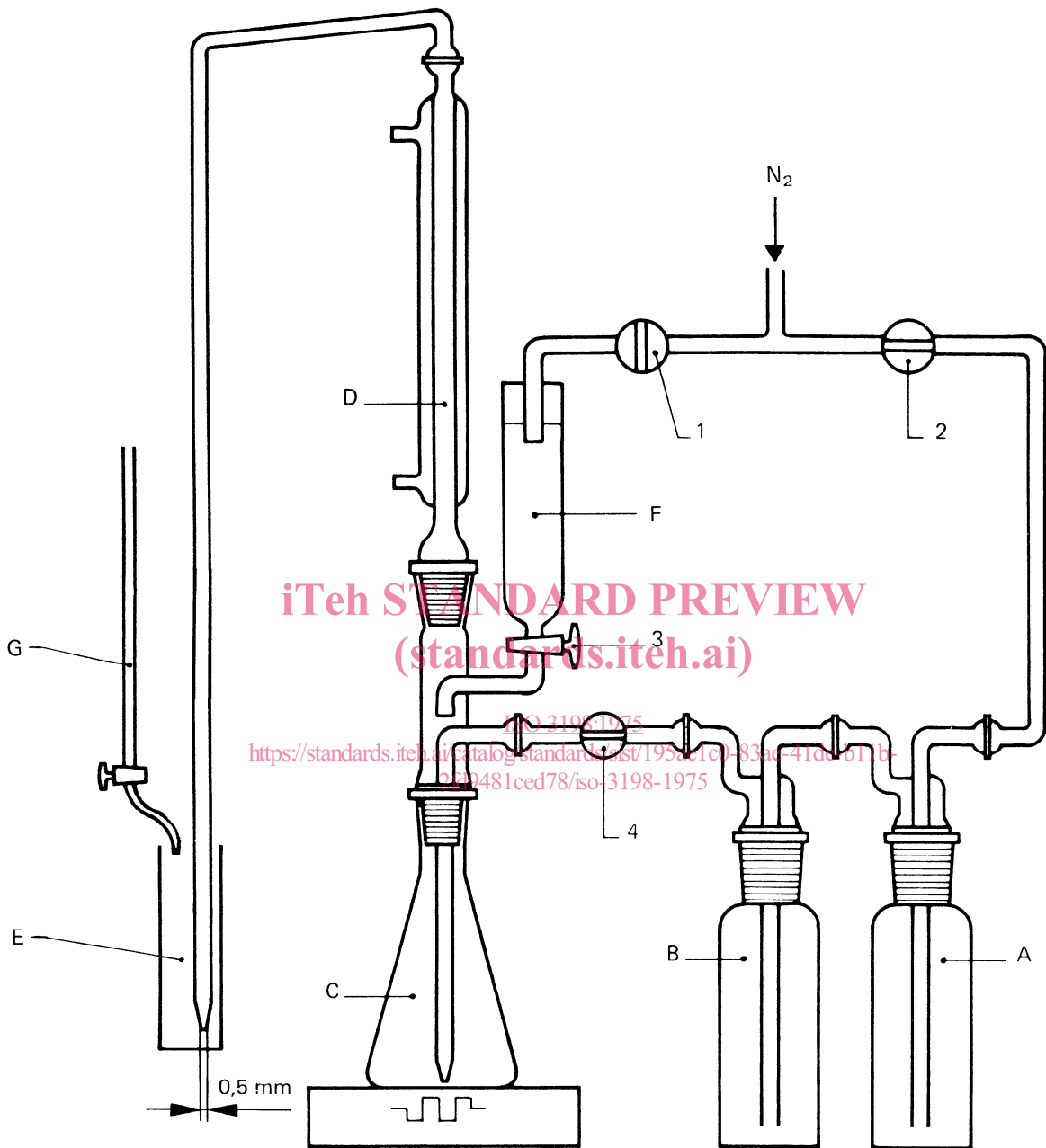
The test report shall include the following particulars :

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.

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 Ground glass joints

FIGURE 1 – Typical apparatus for the determination of sulphur compounds

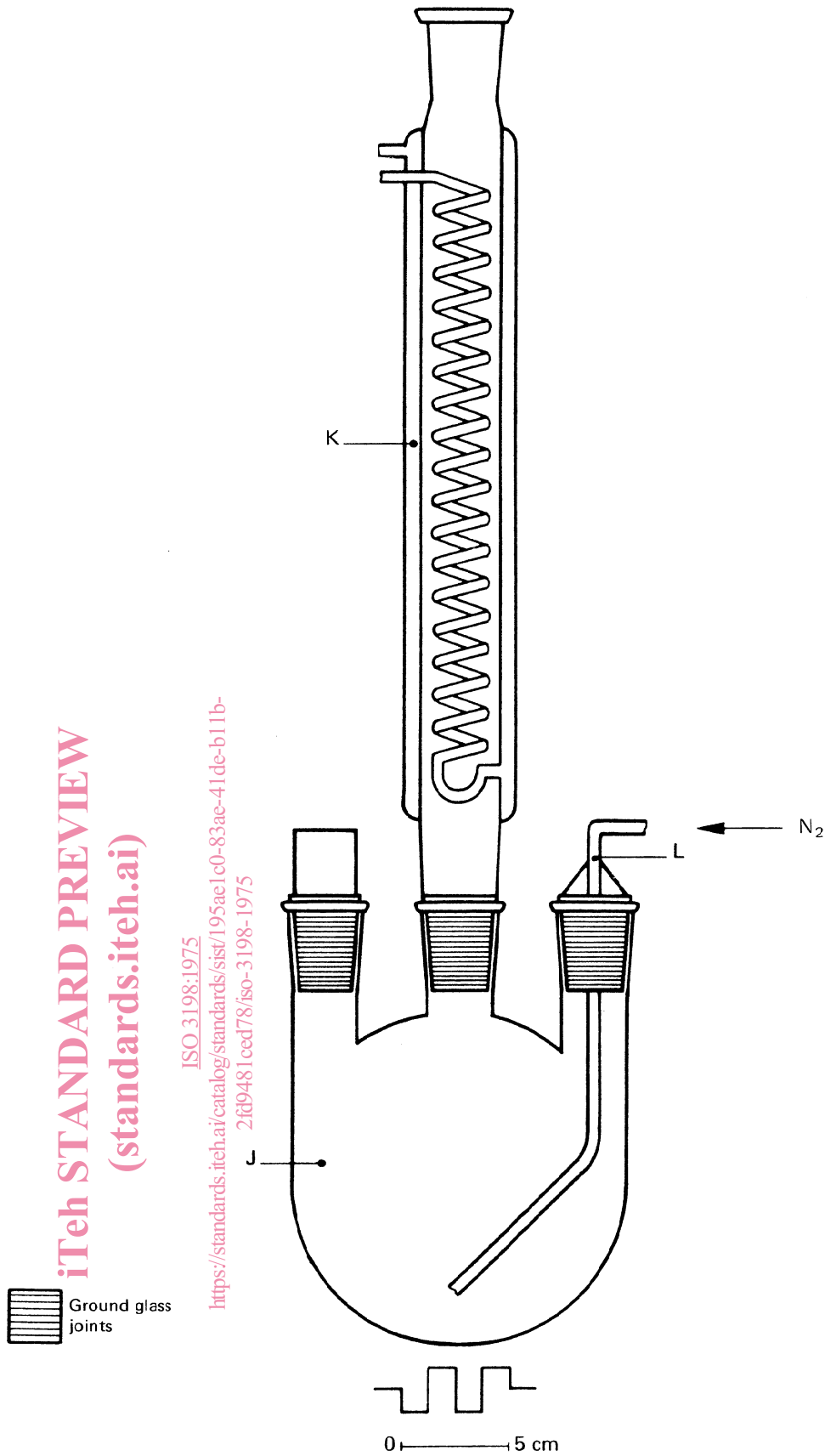


FIGURE 2 – Typical apparatus for the preparation of the reducing solution

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HYDROXIDE FOR INDUSTRIAL USE

- ISO 979 – Method of assay.
- ISO/R 980 – Determination of the carbon dioxide content, expressed as sodium carbonate – Gas-volumetric method.
- ISO 981 – Determination of chloride content – Mercurimetric method.
- ISO/R 982 – Determination of sulphate content – Barium sulphate gravimetric method.
- ISO 983 – Determination of iron content – 1,10-Phenanthroline photometric method.
- ISO 984 – Determination of silica content – Reduced silicomolybdic complex photometric method.
- ISO/R 985 – Determination of silica content – Gravimetric method by precipitation of the quinoline-silicomolybdic complex.
- ISO/R 986 – Determination of calcium – EDTA complexometric method.
- ISO/R 987 – Determination of water-insoluble matter.
- ISO 3195 – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.
- ISO 3196 – Determination of carbon dioxide content – Titrimetric method.
- ISO 3197 – Determination of chlorides content – Photometric method.
- ISO 3198 – Determination of sulphur compounds – Method by reduction and titrimetry.
- ISO 3697 – Determination of calcium and magnesium contents – Flame atomic absorption spectrophotometric method.

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