INTERNATIONAL STANDARD 986

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Sodium hydroxide for industrial use — Determination of calcium content — EDTA (*di*sodium salt) complexometric method

Hydroxyde de sodium à usage industriel — Dosage du calcium — Méthode complexométrique à l'EDTA (sel disodique)

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 986 and found it technically suitable for transformation. International Standard ISO 986 therefore replaces ISO Recommendation R 986-1969 to which it is technically identical.

ISO Recommendation R 986 was approved by the Member Bodies of the following countries :

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

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

U.S.A.

The Member Body of the following country disapproved the transformation of ISO/R 986 into an International Standard :

United Kingdom

Printed in Switzerland

Sodium hydroxide for industrial use — Determination of calcium content — EDTA (*di*sodium salt) complexometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a complexometric method for the determination of the calcium content of sodium hydroxide for industrial use.

The method is applicable to products having a calcium content, expressed as CaO and calculated on NaOH, equal to or greater than 0,002 % (*m/m*).

2 REFERENCE

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

3 PRINCIPLE

Titration of the calcium with a standard volumetric solution of the *di*sodium salt of (ethylenedinitrilo)tetra-acetic acid (EDTA *di*sodium salt) in the presence of 2,2'-(ethylenedinitrilo)diphenol [glyoxal-bis-(2-hydroxy-anil)] as indicator, after elimination of impurities which may hinder the determination.

4 REAGENTS

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

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 N solution.

4.2 Hydrochloric acid, approximately 2 N solution.

Add some water to 165 ml of the hydrochloric acid solution (4.1) and dilute to 1 000 ml.

4.3 Sulphuric acid, approximately 2 N solution.

Add carefully 55 ml of sulphuric acid solution, ρ approximately 1,84 g/ml, to some water, allow to cool and dilute to 1 000 ml.

4.4 Sodium hydroxide, approximately 2 N solution.

Dissolve 80 g of sodium hydroxide in water and dilute to 1 000 ml.

4.5 Ammonium hydroxide, approximately 2 N solution.

Add some water to 250 ml of ammonium hydroxide solution, ρ approximately 0,94 g/ml, about 14,5 % (*m/m*) or approximately 8 N solution, and dilute to 1 000 ml.

4.6 Iron(III) chloride, hydrochloric solution corresponding to approximately 2 g of Fe per litre.

Weigh, to the nearest 0,1 g, 10 g of crystals of iron(III) chloride hexahydrate ($FeCl_3.6H_2O$). Place in a 1 000 ml one-mark volumetric flask and dissolve in approximately 600 ml of water. Add 10 ml of the hydrochloric acid solution (4.1), dilute to the mark and mix.

4.7 Sodium sulphide, saturated solution at ambient temperature.

Prepare a solution using crystals of sodium sulphide nonahydrate ($Na_2S.9H_2O$) previously washed with water.

4.8 Ethanol, 95 % (*V/V*).

Alternatively, alcohol denatured with acetone, but not coloured, may be used.

4.9 Calcium chloride, 0,02 M standard reference solution.

Weigh, to the nearest 0,001 g, 2,002 g of precipitated calcium carbonate ($CaCO_3$) previously dried at 105 ± 2 °C and allowed to cool in a desiccator, and place in a 600 ml beaker. Mix 75 ml of water with 25 ml of the hydrochloric acid solution (4.2) and transfer this solution to the beaker. Boil for about 5 min to eliminate carbon dioxide. Cool to about 20 °C, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.10 (Ethylenedinitrilo)tetraacetic acid (EDTA), *di*sodium salt, dihydrate, 0,002 M standard volumetric solution.

Dissolve approximately 7,7 g of (ethylenedinitrilo)tetraacetic acid, *di*sodium salt, dihydrate (EDTA *di*sodium salt) in water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Adjust this concentrated solution to 0,02 M exactly, calculated on the standard reference calcium chloride solution (4.9), operating as follows :

Transfer 25,0 ml of the standard reference calcium chloride solution (4.9) to a 250 ml conical flask. Successively add approximately 25 ml of water, then, by means of graduated pipettes, 4 ml of the sodium hydroxide solution (4.4), 15 ml of the ethanol (4.8) and 1,0 ml of the 2,2'-(ethylenedinitrilo)diphenol solution (4.12). Wait for about 1 min, then titrate with the solution whose concentration is to be determined, until the colour turns from red to pure yellow. In order to facilitate the correct evaluation of the end-point, a standard colorimetric solution may be used; this solution can be prepared by successively transferring to a 250 ml conical flask : approximately 75 ml of water, 4 ml of the sodium hydroxide solution (4.4), 15 ml of the ethanol (4.8), and 1,0 ml of the 2,2'-(ethylenedinitrilo) diphenol solution (4.12). The correct theoretical quantity is 25 ml.

The *di*sodium EDTA solution should, therefore, be adjusted to the correct concentration and rechecked. Repeat the operation if necessary.

Then transfer 100,0 ml of the concentrated *di*sodium EDTA solution thus adjusted to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

The solution thus obtained is 0,002 M.

4.11 Methyl orange, 0,5 g/l solution.

4.12 2,2'-(Ethylenedinitrilo)diphenol [glyoxal-bis-(2-hydroxy-anil)], approximately 2,5 g/l ethanolic solution.

4.12.1 Place approximately 0,25 g of 2,2'-(ethylenedinitrilo)diphenol in a 100 ml dark glass container with ground glass stopper. Add 100 ml of the ethanol (4.8) and stir until completely dissolved.

4.12.2 Should this indicator be difficult to obtain, it can be prepared as follows :

Place 8,8 g of *o*-aminophenol in a 3 000 ml conical flask containing 2 000 ml of water at 80 °C. After dissolution, add 8 g of a 300 g/l aqueous solution of glyoxal.

Keep the mixture at 80 °C for 30 min, then cool to ambient temperature under running water. Filter the precipitate obtained through a Büchner funnel fitted with a high-speed filter paper and wash three times, carefully drying after each wash. Then remove the precipitate from the filter and place in a 250 ml beaker. Add 100 ml of acetone. After dissolution, add approximately 3 g of active carbon, stir for 1 min, then filter on a dry pleated filter paper and collect the filtrate in a 750 ml conical flask.

Add 300 ml of water to the filtrate and again filter the precipitate through a high-speed filter on a Büchner funnel. Drain thoroughly, dry for 2 h in an oven controlled at 70 °C without exceeding this temperature, crush and store in a dark glass airtight container.

5 APPARATUS

Ordinary laboratory apparatus.

6 PROCEDURE

6.1 Test portion

In a 250 ml beaker, weigh, to the nearest 0,1 g, a mass of the solid or liquid test sample corresponding to 16 ± 0,1 g of NaOH (see ISO 3195).

6.2 Determination

6.2.1 *Preparation of the test solution*

If the product is solid, dissolve the test portion (6.1) in approximately 50 ml of water; if the product is liquid, dilute to approximately 50 ml.

Cool the solution to ambient temperature.

Add slowly, while stirring, 25 ml of the hydrochloric acid solution (4.1) and 3 drops of the methyl orange solution (4.11) and continue adding the hydrochloric acid solution (4.1), drop by drop, until the indicator turns from yellow to red. Then add the sodium hydroxide solution (4.4), drop by drop, until the indicator turns back to yellow, then 3 ml of the sulphuric acid solution (4.3) and boil for 2 min.

Stop heating. Add 1 drop of the methyl orange solution to the hot solution. Neutralize exactly by means of the ammonium hydroxide solution (4.5), then add successively 1 ml of the hydrochloric acid solution (4.2), 0,5 ml of the iron(III) chloride solution (4.6), 4 drops of the sodium sulphide solution (4.7) and 2 ml of the ammonium hydroxide solution (4.5), mixing after each addition. Again boil for 30 s to agglomerate the precipitate, cool to ambient temperature under running water and transfer the turbid solution quantitatively to a 100 ml one-mark volumetric flask. Dilute to the mark, mix and filter on a high-speed dry filter paper free from calcium.

6.2.2 *Titration*

Transfer 50,0 ml of the filtrate to a 250 ml conical flask.

Add successively, by means of graduated pipettes, 10 ml of the sodium hydroxide solution (4.4), 15 ml of the ethanol (4.8) and 1,0 ml of the 2,2-(ethylenedinitrilo)diphenol solution (4.12). The pH of the solution is then greater than 12. Wait for about 2 min, then titrate by means of the *di*sodium EDTA standard volumetric solution (4.10) until the indicator turns from red to pure yellow.

The titration shall be carried out in less than 3 min.

7 EXPRESSION OF RESULTS

The calcium content, expressed as a percentage by mass of calcium oxide (CaO), is given by the formula :

$$V \times \frac{100}{50} \times \frac{100}{m} \times 0{,}000\ 112\ 16 = 0{,}022\ 43 \frac{V}{m}$$

where

V is the volume, in millilitres, of the *di*sodium EDTA solution (4.10) used for the titration;

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

0,000 112 16 is the mass, in grams, of calcium oxide (CaO) corresponding to 1 ml of exactly 0,002 M *di*sodium EDTA solution.

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 the International Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HYDROXIDE FOR INDUSTRIAL USE

ISO 979 — Method of assay.

ISO 980 — Determination of carbonates content — Gas-volumetric method.

ISO 981 — Determination of chloride content — Mercurimetric method.

ISO 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 985 — Determination of silica content — Gravimetric method by precipitation of quinoline molybdosilicate.

ISO 986 — Determination of calcium — EDTA (*di*sodium salt) complexometric method.

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 chloride 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 method.

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