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Sodium hydroxide for industrial use — Determination of carbonates content — Titrimetric method

Hydroxyde de sodium à usage industriel — Dosage des carbonates — Méthode titrimétrique

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FOREWORD

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No Member Body expressed disapproval of the document.

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Sodium hydroxide for industrial use — Determination of carbonates content — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

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

The method is applicable to products having carbonates contents, expressed as CO_2, greater than 0,01 % (*m/m*) :

Three cases, selected by a preliminary test, are envisaged :

1.1 Sodium hydroxide containing neither sulphides nor chlorates.

1.2 Sodium hydroxide containing sulphides.

1.3 Sodium hydroxide containing chlorates.

2 REFERENCE

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

3 PRELIMINARY TEST

3.1 Principle

Boiling of an acidified test sample, containing methyl orange and with a lead acetate paper strip in the vapour. The presence of sulphides produces blackening of the paper and the presence of chlorates decolorizes the methyl orange.

3.2 Reagents

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

3.2.1 Hydrochloric acid, approximately 6 N solution.

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

3.2.3 Lead acetate paper, cut into strips about 15 mm X 80 mm.

3.3 Procedure

Place about 20 g of the test sample in a 300 ml conical flask. Add 150 ml of water and 3 drops of the methyl orange solution (3.2.2). Neutralize with the hydrochloric acid solution (3.2.1) and add an excess of 5 ml of this acid.

Fix a lead acetate paper strip (3.2.3) inside the neck of the conical flask, curling it over the outside of the flask.

Boil the solution for 5 min and select the procedure to be followed according to the following table.

Appearance		Case number	Procedure
solution	paper		
red	white	1.1	Clause 4
red	blackened	1.2	Clause 5
colourless	white	1.3	Clause 6

4 SODIUM HYDROXIDE CONTAINING NEITHER SULPHIDES NOR CHLORATES

4.1 Principle

Liberation of carbon dioxide by acidification and heating. Entrainment in a flow of gas and absorption in an excess of barium hydroxide solution. Titration of the excess barium hydroxide with a standard volumetric hydrochloric acid solution, in the presence of thymolphthalein as indicator.

4.2 Reagents

Reagents listed in 3.2, and

4.2.1 Nitrogen, or **air**, free from carbon dioxide.

4.2.2 Barium hydroxide approximately 0,1 N solution.

Weigh about 15,0 g of barium hydroxide octahydrate [$Ba(OH)_2.8H_2O$], dissolve in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Allow precipitated barium carbonate to settle out before use.

4.2.3 Hydrochloric acid, 0,1 N standard volumetric solution.

4.2.4 Thymolphthalein, 5 g/l solution in 95 % (*V/V*) ethanol.

4.3 Apparatus

Ordinary laboratory apparatus and :

4.3.1 Apparatus for the release, absorption and titration of carbon dioxide. See the figure for an example of a typical apparatus, which comprises :

A *Flask*, capacity 500 ml;

B *Separating funnel*, capacity 100 ml;

C_1 and C_2 *Gas-washing bottles*, Drechsel type, containing 20 % (*m/m*) sodium hydroxide solution;

C_3 *Gas-washing bottle*, Drechsel type, containing saturated barium hydroxide solution;

D *Spiral condenser*;

E *Absorption vessel*;

F *Absorption coil*, minimum length 70 cm;

G *Burette*, 50 ml, graduated in 0,1 ml;

H *V-bore stopcock*;

I and J *Inlet and outlet*, for nitrogen or air (4.2.1).

4.4 Procedure

4.4.1 *Test portion*

Weigh, to the nearest 0,01 g, in a weighing bottle with lid, a mass of test sample, solid or liquid (see ISO 3195), corresponding to not more than 25 g of NaOH and which will release not more than 50 mg of carbon dioxide.

4.4.2 *Blank test*

After the determination, carry out a blank test, following the procedure described in 4.4.3, using the same apparatus but omitting the test portion.

4.4.3 *Determination*

4.4.3.1 Pass the nitrogen or air (4.2.1) for 10 min into the apparatus (4.3.1) at the rate of approximately 5 bubbles per second.

4.4.3.2 Stop the gas flow and add respectively :

- to the separating funnel (B) : approximately 90 ml of the hydrochloric acid solution (3.2.1);
- to the flask (A) : the test portion (4.4.1), 150 ml of water and 3 drops of the methyl orange solution (3.2.2);
- to the absorption vessel (E) : 50,00 ml of the barium hydroxide solution (4.2.2) and 3 drops of the thymolphthalein solution (4.2.4).

4.4.3.3 Close the apparatus, start the flow of water through the condenser (D) and allow the acid to flow very gently from the funnel (B) into the flask (A) until the indicator changes to red, then add a further 5 ml, approximately, of acid, taking care to leave at least a few millimetres of liquid above the stopcock (H).

4.4.3.4 Allow the gas to flow, regulated at 2 bubbles per second, for 10 min. Then, without interrupting this flow, heat the contents of the flask (A) to boiling and maintain boiling for 20 min. Turn off the heat, increase the flow of gas to 5 bubbles per second, and titrate the excess of barium hydroxide solution, contained in vessel (E), with the standard volumetric hydrochloric acid solution (4.2.3) from the burette (G), until the indicator changes from blue to colourless.

4.5 Expression of results

The carbonates content, expressed as a percentage by mass of carbon dioxide (CO_2), is given by the formula

$$(V_0 - V_1) \times T \times \frac{100}{m} \times 0{,}022 = \frac{2{,}2\,(V_0 - V_1)\,T}{m}$$

where

V_0 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (4.2.3) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (4.2.3) used for the determination;

T is the actual concentration, expressed as normality, of the standard volumetric hydrochloric acid solution (4.2.3);

m is the mass, in grams, of the test portion (4.4.1);

0,022 is the mass, in grams, of carbon dioxide corresponding to 1 ml of exactly 1 N standard volumetric hydrochloric acid solution.

5 SODIUM HYDROXIDE CONTAINING SULPHIDES

In the case of sodium hydroxide containing sulphides, the acidification of the product causes the liberation of hydrogen sulphide which is absorbed in the barium hydroxide solution at the same time as the carbon dioxide, giving a result in excess of the true value. A preliminary treatment of the test portion is therefore necessary.

The method is applicable to products containing up to 1 g of sulphides, expressed as Na_2S, per kilogram.

5.1 Principle

Oxidation of the sulphides by hydrogen peroxide and boiling of the test sample before acidification.

Determination according to the method specified in clause 4.

5.2 Reagents

Reagents listed in 3.2 and 4.2, and :

5.2.1 Hydrogen peroxide, 30 % (*m/m*) solution.

5.3 Apparatus

See 4.3.

5.4 Procedure

Follow the procedure specified in 4.4.3, applying the following modifications :

– in 4.4.3.2 : place the test portion (4.4.1) in the flask (A), then add 150 ml of water and 5 drops of the hydrogen peroxide solution (5.2.1);

– in 4.4.3.3 : before pouring the acid into the flask (A), boil the contents of the flask for 5 min under a gas flow, then cool and add 3 drops of the methyl orange solution (3.2.2).

5.5 Expression of results

See 4.5.

6 SODIUM HYDROXIDE CONTAINING CHLORATES

In the case of sodium hydroxide containing chlorates, acidification of the product causes the liberation of chlorine, which is absorbed in the barium hydroxide solution at the same time as the carbon dioxide, giving a result in excess of the true value. A preliminary treatment of the test portion is therefore necessary.

The method is applicable to products containing up to 2 g of chlorates, expressed as $NaCl_3$, per kilogram.

6.1 Principle

Prior reduction of the chlorates to chlorides by addition of iron(II) sulphate.

Determination according to the method specified in clause 4.

6.2 Reagents

Reagents listed in 3.2 and 4.2, and :

6.2.1 Iron(II) sulphate solution, containing 28 g of iron(II) sulphate heptahydrate ($FeSO_4.7H_2O$) and 4 drops of the hydrochloric acid solution (3.2.1) in 100 ml.

6.3 Apparatus

See 4.3.

6.4 Procedure

Follow the procedure specified in 4.4.3, applying the following modification :

– in 4.4.3.2 : place in the flask (A) the test portion (4.4.1), 150 ml of water, 3 drops of the methyl orange solution (3.2.2), and 5 ml of the iron(II) sulphate solution (6.2.1) and then mix.

6.5 Expression of results

See 4.5.

7 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.

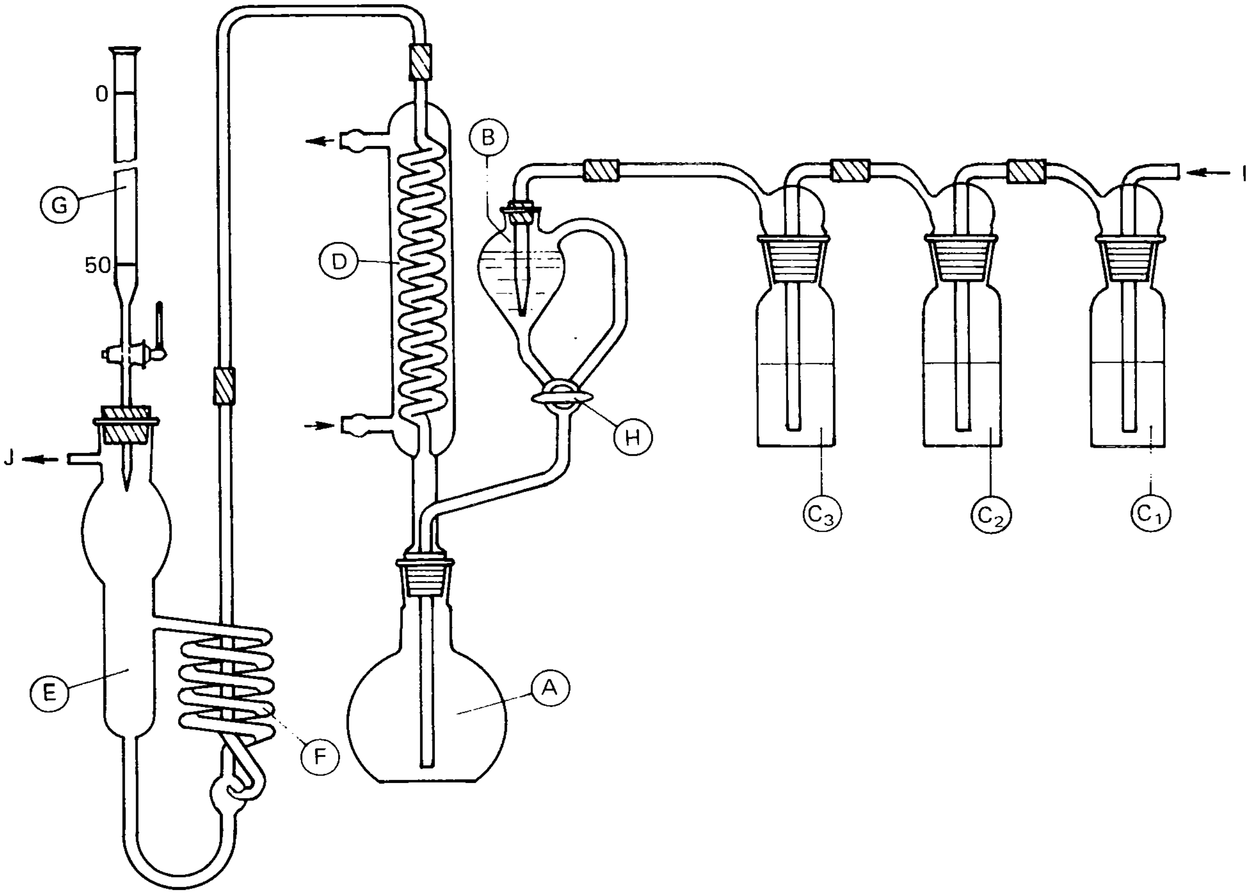

FIGURE – **Typical apparatus for the determination of carbonates by the barium hydroxide titrimetric method**