INTERNATIONAL STANDARD ISO 2900

G-96-10

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

Potassium hydroxide for industrial use — Determination of carbon dioxide content — Titrimetric method

First edition — 1973-08-15

UDC 661.312 : 546.264 : 543.24 Ref. No. ISO 2900-1973 (E)

Descriptors : potassium hydroxides, chemical analysis, determination of content, carbon dioxide, volumetric analysis.

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Price based on 3 pages

FOREWORD

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International Standard ISO 2900 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in July 1972.

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

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

No Member Body expressed disapproval of the document.

Printed in Switzerland

Potassium hydroxide for industrial use — Determination of carbon dioxide content — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method for the determination of the carbon dioxide content of potassium hydroxide for industrial use.

The method is applicable to products having carbon dioxide contents greater than 0,01 % (*m/m*).

Three cases are envisaged :

1.1 Potassium hydroxide containing neither sulphides nor chlorates.

1.2 Potassium hydroxide containing sulphides.

1.3 Potassium hydroxide containing chlorates.

2 REFERENCE

ISO 2466, *Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*

3 POTASSIUM HYDROXIDE CONTAINING NEITHER SULPHIDES NOR CHLORATES

3.1 Principle

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

3.2 Reagents

Distilled water, free from carbon dioxide, or water of equivalent purity, shall be used in the test.

3.2.1 Hydrochloric acid, approximately 6 N solution.

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

3.2.3 Barium hydroxide, approximately 0,1 N standard volumetric solution.

Weigh, to the nearest 0,001 g, 15,774 g of barium hydroxide octahydrate, [$Ba(OH)_2 \cdot 8H_2O$], dissolve in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

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

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

3.2.6 Nitrogen or air, free from carbon dioxide.

3.3 Apparatus

Ordinary laboratory apparatus and

3.3.1 Apparatus for the release, absorption and titration of carbon dioxide. See Figure 1 for an example of a typical apparatus which comprises :

A	*Flask,* 500 ml capacity;
B	*Separating funnel,* 100 ml;
C_1 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	*T-bore stopcock;*
I J	*Inlet and outlet,* for nitrogen or air (3.2.6).

3.4 Procedure

3.4.1 Test portion

Weigh, to the nearest 0,01 g, in a weighing bottle with a lid, a mass of test sample, solid or liquid, corresponding to not more than 25 g of potassium hydroxide and containing not more than 50 mg of carbon dioxide (see ISO 2466).

3.4.2 Blank test

After the determination, carry out a blank test following the procedure described in 3.4.3, using the same apparatus but omitting the test portion.

3.4.3 Determination

3.4.3.1 Pass the nitrogen or the air (3.2.6) for 10 min through the apparatus (3.3.1) at a rate of about 5 bubbles per second.

3.4.3.2 Stop the gas flow and add respectively :

- to the separating funnel (B) : about 90 ml of the hydrochloric acid solution (3.2.1);
- to the flask (A) : the test portion (3.4.1), 150 ml of water and 3 drops of the methyl orange solution (3.2.4);
- to the absorption vessel (E) : 50,00 ml of the standard volumetric barium hydroxide solution (3.2.3) and 3 drops of the thymolphthalein solution (3.2.5).

3.4.3.3 Close the apparatus, start the condenser (D) and carefully allow the acid in the separation funnel (B) to run into the flask (A) until the appearance of the red colour of the indicator; then add about 5 ml more of acid and take care to let a few millimetres of liquid remain above the stopcock (H) as a trap.

3.4.3.4 Allow the gas to flow, regulated at 2 bubbles per second, for 10 min and then, without interrupting the flow, heat the contents of the flask (A) just to boiling and maintain there for 20 min. Stop the heating, increase the gas flow to 5 bubbles per second and titrate the excess of the standard volumetric barium hydroxide solution contained in the vessel (E) with the standard volumetric hydrochloric acid solution (3.2.2), using the burette (G), until the blue colour of the indicator changes to colourless.

3.5 Expression of results

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

$$(V_0 - V_1) \times \frac{100}{m} \times 0{,}002\,2 = \frac{0{,}22\,(V_0 - V_1)}{m}$$

where

V_0 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (3.2.2) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (3.2.2) used for the determination;

m is the mass, in grams, of the test portion.

4 POTASSIUM HYDROXIDE CONTAINING SULPHIDES

In the case of potassium hydroxide containing sulphides, the acidification of the product causes a release 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.

4.1 Principle

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

Determination according to the method described in section 3.

4.2 Reagents

Reagents listed in 3.2 and

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

4.3 Apparatus

See 3.3.

4.4 Procedure

Proceed as described in 3.4.3, applying the following modifications :

- 3.4.3.2 Place the test portion (3.4.1) in the flask (A) then add 150 ml of water and 5 drops of the hydrogen peroxide solution (4.2.1).
- 3.4.3.3 Before running the acid into the flask(A), boil the contents for 5 min under a gas flow then cool and add 3 drops of the methyl orange solution (3.2.4).

4.5 Expression of results

See 3.5.

5 POTASSIUM HYDROXIDE CONTAINING CHLORATES

In the case of potassium hydroxide containing chlorates, the acidification results in a release 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.

5.1 Principle

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

Determination according to the method described in section 3.

5.2 Reagents

Reagents listed in 3.2 and

5.2.1 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.

5.3 Apparatus

See 3.3.

5.4 Procedure

Proceed as described in 3.4.3 applying the following modifications :

— 3.4.3.2. Place 5 ml of the iron(II) sulphate solution (5.2.1) in the flask (A) in addition to the test portion, 150 ml of water and 3 drops of the methyl orange solution (3.2.4) and mix.

5.5 Expression of results

See 3.5.

6 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 that document to which reference is made, or regarded as optional.

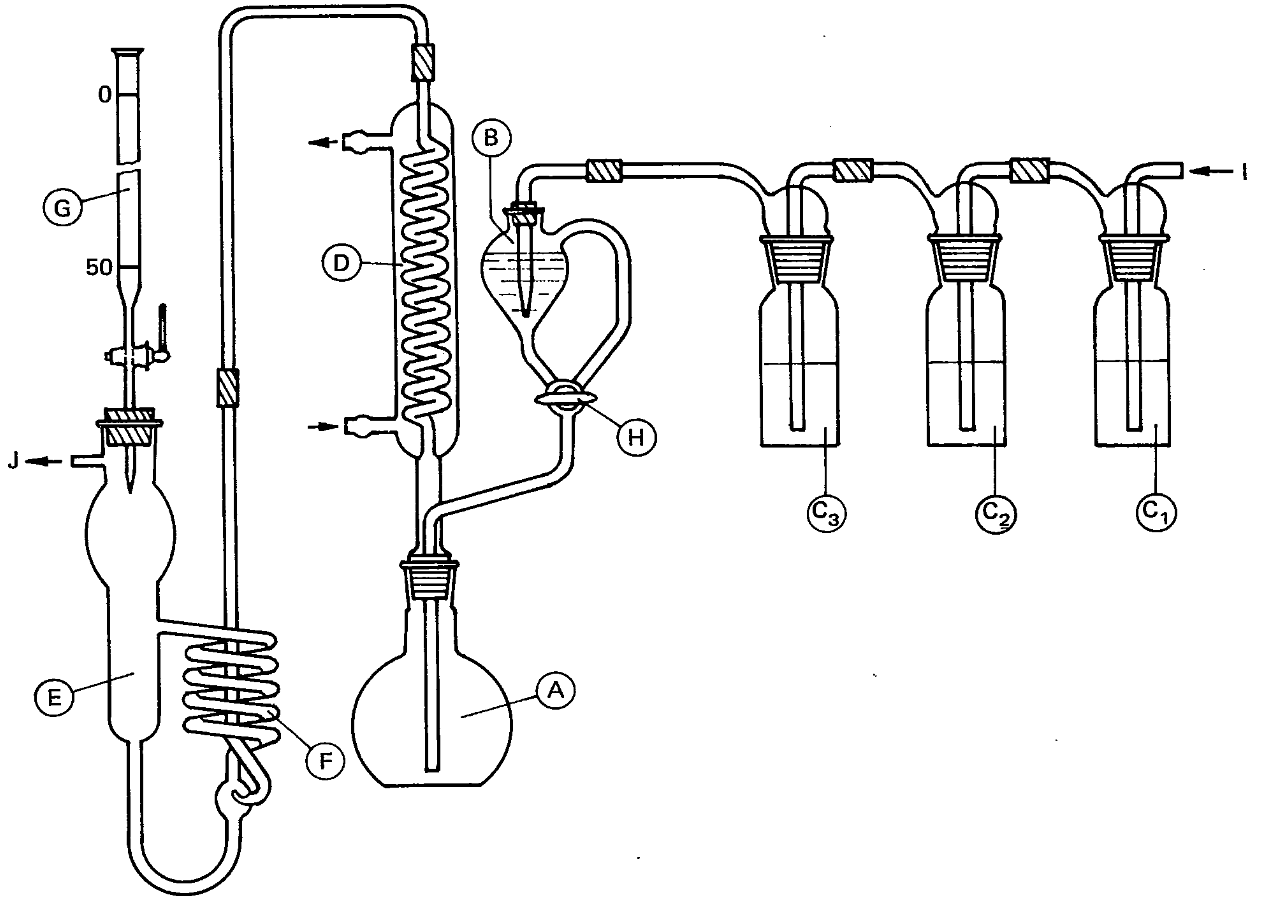

FIGURE —Typical apparatus for the determination of carbon dioxide by the barium hydroxide titrimetric method