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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 991

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE
DETERMINATION OF THE CARBON DIOXIDE CONTENT,
EXPRESSED AS POTASSIUM CARBONATE
GAS-VOLUMETRIC METHOD

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BRIEF HISTORY

The ISO Recommendation R 991, *Potassium hydroxide for industrial use – Determination of the carbon dioxide content, expressed as potassium carbonate – Gas-volumetric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led, in 1966, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1100) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Israel	South Africa, Rep. of
Belgium	Italy	Spain
Brazil	Japan	Switzerland
Chile	Korea, Dem. P. Rep. of	Thailand
Czechoslovakia	Netherlands	Turkey
Germany	New Zealand	U.A.R.
Hungary	Poland	United Kingdom
India	Portugal	U.S.S.R.
Ireland	Romania	Yugoslavia

Three Member Bodies opposed the approval of the Draft :

Cuba
France
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE
DETERMINATION OF THE CARBON DIOXIDE CONTENT,
EXPRESSED AS POTASSIUM CARBONATE
GAS-VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a gas-volumetric method for the determination of the carbon dioxide content, expressed as potassium carbonate, in potassium hydroxide for industrial use.

2. FIELD OF APPLICATION

The general method is applicable to the determination of the carbon dioxide content in potassium hydroxide for industrial use.

Special case

Potassium hydroxide containing chlorate.

3. PRINCIPLE

Measurement of the volume of carbon dioxide evolved from a test portion by reaction with a hydrochloric acid solution.

4. REAGENTS

- 4.1 *Distilled water*, or water of equivalent purity, free from carbon dioxide at room temperature.

Eliminate any carbon dioxide present either by boiling the water for 10 minutes and cooling in the absence of atmospheric carbon dioxide or, more simply, by bubbling air free from carbon dioxide through it for 15 minutes.

The air is freed from carbon dioxide by passing it through a column containing pellets of sodium hydroxide. Store in the absence of atmospheric carbon dioxide.

- 4.2 *Hydrochloric acid*, approximately $d = 1.19$, 38 % (m/m) or 12 N solution.

- 4.3 *Sodium chloride*, coloured acid solution.

Dissolve 263 g of sodium chloride in water. Add 5 ml of a 93.6 % (m/m) sulphuric acid solution (approximately $d = 1.83$ or 35 N). Dilute to 1000 ml, add a small amount of 0.5 g/l methyl orange solution and mix thoroughly.

- 4.4 *Sodium hydroxide*, 20 % (m/m) solution, approximately $d = 1.22$ or 6 N solution.

Dissolve 120 g of sodium hydroxide in water. Dilute to 500 ml and mix thoroughly.

5. APPARATUS

- 5.1 *Ordinary laboratory apparatus.*
- 5.2 *Apparatus as shown in the Figure.*

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.01 g, a mass of the test sample (solid or liquid)* corresponding to approximately 10 g of KOH.

6.2 Determination

- 6.2.1 *Preparation of the apparatus.* Fill burette (B) of the apparatus (5.2) with the coloured acid solution (4.3) through levelling bottle (F).

Pour into absorber (C) some of the sodium hydroxide solution (4.4). (Renew this solution after about 100 determinations.)

With burette (B) and absorber (C) filled up to cock (R_1) and graduation mark (a) respectively, and cocks (R_1) and (R_2) closed, place the test portion (6.1) in flask (A) and, in case of solid material, dissolve in approximately 30 ml of water (4.1). Dilute to approximately 40 ml so as to reduce the dead space to a volume slightly greater than 100 ml (volume above the level of the liquid in flask (A) plus the volume of condenser tube (D) up to cock (R_1)).

Place in the flask three porcelain or glass balls, about 2 mm in diameter, and a few pieces of pumice having a total volume approximately equal to that of the balls. Stopper the flask and close cock (R).

Connect flask (A) with burette (B) through cock (R_1) and lower the levelling bottle (F).

Check the tightness of the apparatus by appropriately handling the cocks and the levelling bottle.

- 6.2.2 *Evolution and measurement of carbon dioxide.* By means of the separating funnel, pour 35 ml of the hydrochloric acid solution (4.2) into flask (A) taking care to avoid loss of gas. The acidity of the solution in the flask is thus approximately 2 N after evolution of carbon dioxide.

Heat the flask and maintain the solution at boiling point for 5 minutes while running cold water through the condenser. Then stop heating and add more of the coloured acid solution (4.3) by means of the separating funnel, lowering the levelling bottle (F) still further so as to make the solution in flask (A) rise in the condenser tube up to cock (R_1). Then close the latter and wait 5 minutes to allow the gas to reach the temperature of the water jacket.

* See ISO Recommendation R 988, *Potassium hydroxide for industrial use – Preparation and storage of test sample*, clause 2.2.

Measure the volume of gas V at atmospheric pressure P and at the temperature t of the water in the jacket. For this purpose move the levelling bottle (F) so as to bring the coloured acid solution (4.3) to the same level both in the flask and in the burette (B); read the volume of the latter.

Adjust the cocks (R_1) and (R_2) so that burette (B) and absorber (C) are connected, then raise the levelling bottle (F) so that the gas is transferred to the absorber where the carbon dioxide is absorbed. Then transfer the non-absorbed gas back to burette (B) by lowering the levelling bottle (F) and, after adjusting the level of the sodium hydroxide solution (4.4) to the gauge mark (a) on absorber (C), close cock (R_2) and read the volume of the residual gas. Repeat these operations until a constant volume V_1 is obtained.

The difference of volume ($V - V_1$) represents the carbon dioxide contained in the test portion, measured at atmospheric pressure and at the temperature of the water jacket.

7. EXPRESSION OF RESULTS

The carbon dioxide content, expressed as potassium carbonate (K_2CO_3), is given as a percentage, by mass, by the following formula :

$$(V - V_1) \times \frac{273}{273 + t} \times \frac{P - p}{760} \times 0.001\,976\,8 \times \frac{138.213}{44.010} \times \frac{100}{E} = 0.223 \frac{P - p}{273 + t} \times \frac{V - V_1}{E}$$

where

V is the volume, in millilitres, of gas before absorption of CO_2 ;

V_1 is the volume, in millilitres, of gas after absorption of CO_2 ;

t is the temperature, in degrees Celsius, of the water contained in the water jacket;

P is the atmospheric pressure, in millimetres of mercury absolute, at the time of the determination;

p is the vapour pressure, in millimetres of mercury absolute, of the coloured acid solution (4.3) at the temperature of the water contained in the jacket; vapour pressures are given in the table below for temperatures from 10 to 30 °C.

Temperature, t °C	10	12	14	16	18	20	22	24	26	28	30
Vapour pressure p mmHg absolute	8	9	10	11	12	14	16	18	20	22	25

0.001 976 8 is the conversion factor for the CO_2 volume in millilitres, to CO_2 mass in grammes;

$\frac{138.213}{44.010}$ is the conversion factor for mass of CO_2 to the corresponding mass of K_2CO_3 ;

E is the mass, in grammes, of the test portion.

8. SPECIAL CASE

In the case of potassium hydroxide containing chlorate, the addition of the hydrochloric acid solution causes evolution of chlorine. This is absorbed by the sodium hydroxide solution at the same time as carbon dioxide, thus giving erroneously high results.

8.1 Principle

Preliminary reduction of sodium chlorate to chloride by addition of iron (II) sulphate.
Determination following the general method.

8.2 Reagents

Same reagents as indicated in section 4 and

8.2.1 *Iron (II) sulphate*, 280 g/l hydrochloric solution.

Dissolve 28 g of iron (II) sulphate heptahydrate in water (4.1).

Add 2 drops of the hydrochloric acid solution (4.2), dilute to 100 ml and mix thoroughly.

8.3 Apparatus

See section 5.

8.4 Procedure

8.4.1 *Test portion*. See clause 6.1.

8.4.2 *Determination*

8.4.2.1 PREPARATION OF THE APPARATUS. See clause 6.2.1.

8.4.2.2 EVOLUTION AND MEASUREMENT OF CARBON DIOXIDE. By means of the separating funnel, introduce 5 ml of the iron sulphate solution (8.2.1), taking care to avoid loss of gas. Mix and then add 35 ml of the hydrochloric acid solution (4.2). The acidity of the solution contained in the flask is then about 2 N after evolution of carbon dioxide.

Continue the determination following the general method from the second paragraph of clause 6.2.2.

8.5 Expression of results

See section 7.

9. TEST REPORT

Give 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 ISO Recommendation or regarded as optional.