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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1691

SODIUM AND POTASSIUM SILICATES
FOR INDUSTRIAL USE

DETERMINATION OF CARBON DIOXIDE CONTENT EXPRESSED AS SODIUM OR POTASSIUM CARBONATE
GAS-VOLUMETRIC METHOD

1st EDITION

July 1970

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BRIEF HISTORY

The ISO Recommendation R 1691, *Sodium and potassium silicates for industrial use – Determination of carbon dioxide content expressed as sodium or 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 to the adoption of Draft ISO Recommendation No. 1691,which was circulated to all the ISO Member Bodies for enquiry in December 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

- Australia
- Austria
- Belgium
- Brazil
- Colombia
- Czechoslovakia
- France
- Germany
- Greece
- Hungary
- India
- Iran
- Israel
- Italy
- Japan
- Netherlands
- New Zealand
- Peru
- Poland
- Portugal
- Romania
- South Africa, Rep. of
- Spain
- Switzerland
- Thailand
- Turkey
- U.A.R.
- United Kingdom
- U.S.S.R.
- Yugoslavia

No Member Body opposed the approval of the Draft.

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

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ISO Recommendation R 1691 July 1970

SODIUM AND POTASSIUM SILICATES FOR INDUSTRIAL USE

DETERMINATION OF CARBON DIOXIDE CONTENT EXPRESSED AS SODIUM OR POTASSIUM CARBONATE

GAS-VOLUMETRIC METHOD

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1. SCOPE

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

2. FIELD OF APPLICATION

The general method applies to the analysis of sodium or potassium silicates which do not contain reducing sulphur compounds such as sulphides and sulphites.

2.1 Special case

Determination of the carbon dioxide content in sodium or potassium silicates containing reducing sulphur compounds such as sulphides and sulphites (see section 9).

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 equal 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 colomn containing pellets of sodium hydroxide.

Store the water in the absence of atmospheric carbon dioxide.

4.2 *Hydrochloric acid,* approximately ρ 1.19 (g/ml), 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 96 % (m/m) sulphuric acid solution (approximately ρ 1.84 (g/ml) or 36 N). Dilute to 1000 ml, add a small amount of a 0.5 g/l methyl orange solution and mix thoroughly.

4.4 *Sodium hydroxide,* approximately ρ 1.22 (g/ml), 20 % (m/m) or 6 N solution.

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

5. APPARATUS

Ordinary laboratory apparatus and

5.1 Apparatus as shown in the Figure (page 7).

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, a mass of the test sample of 4.9 to 5.1 g.

6.2 Determination

6.2.1 *Preparation of the apparatus.* Fill burette (B) of the apparatus (5.1) with coloured acid solution (4.3) through levelling bottle (F).

Pour into absorber (C) sodium hydroxide solution (4.4). (This solution should be renewed approximately every 100 determinations.)

Burette (B) and absorber (C) being filled up to stopcock (R_1) and mark *a* respectively, stopcocks (R_1) and (R_2) being closed, quantitatively transfer the test portion (6.1), previously mixed with 10 ml of water, to flask (A). 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 of approximately 2 mm diameter, together with a few pieces of pumice stone, the volume of which should be approximately equal to that of the balls. Close the flask and stopcock (R).

Open stopcock (R_1) to connect flask (A) and burette (B) and lower 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.* Pour 20 ml of hydrochloric acid solution (4.2) into flask (A), through the funnel provided with a stopcock, avoiding loss of gas.

Heat the flask and keep the solution boiling for 5 minutes, running cold water through the condenser. Stop heating and add coloured acid solution (4.3) through the funnel by lowering levelling bottle (F) so that the liquid contained in flask (A) rises in the condensing tube up to (R_1). Close (R_1) and wait for 5 minutes so that the gases attain the temperature of the thermostatic jacket.

Measure the volume of gas (V) at atmospheric pressure (P) and at the temperature (t) of the water contained in the jacket. For this purpose, the levelling bottle (F) should be moved in such a way that the coloured acid solution is brought to the same level both in the levelling bottle (F) and burette (B); read the volume in the latter.

Manipulate (R) and (R_2) so that burette (B) and absorber (C) are connected, and lift levelling bottle (F) so that the gases are transferred into the absorber where the carbon dioxide is absorbed. Return the gases that are not absorbed to burette (B) by lowering levelling bottle (F) and read the volume of gas remaining after having brought the level of the sodium hydroxide solution back to mark *a* on absorber (C) and having closed stopcock (R_2). Repeat these operations until a constant volume of gas is obtained (V_1).

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

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7. EXPRESSION OF RESULTS

The content of sodium carbonate (Na_2CO_3) or of potassium carbonate (K_2CO_3) is given as a percentage by mass by the following formulae :

$$Na_2CO_3, \% \text{ (m/m)} = (V - V_1) \times \frac{273}{273 + t} \times \frac{P - p}{1013} \times 0.00198 \times \frac{106}{44} \times \frac{100}{m} = 0.128 \, \frac{P - p}{273 + t} \times \frac{V - V_1}{m}$$

$$K_2CO_3, \% \text{ (m/m)} = (V - V_1) \times \frac{273}{273 + t} \times \frac{P - p}{1013} \times 0.00198 \times \frac{138.2}{44} \times \frac{100}{m} = 0.167 \, \frac{P - p}{273 + t} \times \frac{V - V_1}{m}$$

where

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

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

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

P is the atmospheric pressure, in millibars, during the determination;

p is the vapour pressure, in millibars, of the coloured acid solution of sodium chloride (4.3) at the temperature of the water contained in the thermostatic jacket.

These vapour pressures are given below for temperatures between 10 and 30 °C;

Temperature t **°C**	10	12	14	16	18	20	22	24	26	28	30
Vapour pressure p, **millibars**	11	12	13	15	17	19	21	24	27	30	33

0.00198 is the conversion factor of the volume, in millilitres, of carbon dioxide into the mass, in grammes, of this gas;

$\frac{106}{44}$ is the conversion factor of the mass of carbon dioxide into the corresponding mass of Na_2CO_3 ;

$\frac{138.2}{44}$ is the conversion factor of the mass of carbon dioxide into the corresponding mass of K_2CO_3 ;

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

8. ACCURACY OF THE METHOD

The results obtained with this method are reproducible to the nearest ± 0.04 % (m/m) in absolute value.

9. SPECIAL CASE

If the test sample contains reducing sulphur compounds such as sulphides and sulphites, the addition of hydrochloric acid causes an emission of hydrogen sulphide and sulphur dioxide. These gases are absorbed by sodium hydroxide at the same time as the carbon dioxide. This gives high results.

9.1 Principle

Previous oxidation of the reducing sulphur compounds into sulphates by adding hydrogen peroxide and carrying out the determination according to the general method (see section 3).

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9.2 Reagents

As indicated in section 4 and

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

9.3 Apparatus

See section 5.

9.4 Procedure

9.4.1 *Test portion.* See clause 6.1.

9.4.2 *Determination*

9.4.2.1 PREPARATION OF THE APPARATUS. See clause 6.2.1.

9.4.2.2 EVALUATION AND MEASUREMENT OF CARBON DIOXIDE. Pour 1 or 2 drops of hydrogen peroxide (9.2.1) into flask (A) through the funnel provided with a stopcock and avoid any loss of gas. Mix and add, in the same manner, 20 ml of hydrochloric acid solution (4.2).

Continue the determination following the general method from the 2nd paragraph of clause 6.2.2.

9.5 Expression of results

See section 7.

10. 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 operations not included in this ISO Recommendation, or regarded as optional.

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