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Sodium and potassium silicates for industrial use — Determination of carbonates content — Gas-volumetric method

Silicates de sodium et de potassium à usage industriel — Dosage des carbonates — Méthode gazométrique

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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 1691 and found it technically suitable for transformation. International Standard ISO 1691 therefore replaces ISO Recommendation R 1691-1970.

ISO Recommendation R 1691 was approved by the Member Bodies of the following countries :

Australia
Austria
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India
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Spain
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Thailand
Turkey
United Kingdom
U.S.S.R.
Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1691 into an International Standard.

Printed in Switzerland

Sodium and potassium silicates for industrial use — Determination of carbonates content — Gas-volumetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gas-volumetric method for the determination of the carbonates content, expressed, respectively, as sodium carbonate or potassium carbonate, of sodium and potassium silicates for industrial use.

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

1.1 Sodium or potassium silicates containing neither sulphides nor chlorates.

1.2 Sodium or potassium silicates containing sulphides.

1.3 Sodium or potassium silicates containing chlorates.

2 REFERENCE

ISO 1686, *Sodium and potassium silicates for industrial use — Samples and methods of test — General.*

3 PRELIMINARY TEST

3.1 Principle

Boiling of an acidified test portion 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 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 × 80 mm.

3.3 Procedure

Place about 5 g of the test sample (see ISO 1686) 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 OR POTASSIUM SILICATES CONTAINING NEITHER SULPHIDES NOR CHLORATES

4.1 Principle

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

4.2 Reagents

Reagents specified in 3.2 and

4.2.1 Distilled water, or water of equal purity, free from carbon dioxide at ambient temperature.

Eliminate any carbon dioxide present either by boiling the water for 10 min and cooling in the absence of atmospheric carbon dioxide or, more simply, by bubbling air free from carbon dioxide through it for 15 min : (Free the air from carbon dioxide by passing it through a column containing pellets of sodium hydroxide.)

Store the water in the absence of atmospheric carbon dioxide.

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

4.2.3 Sodium chloride, coloured acid solution.

Dissolve 263 g of sodium chloride in water. Add 5 ml of a sulphuric acid solution, ρ approximately 1,84 g/ml, about 96 % *(m/m)* or approximately 36 N solution. Dilute to 1 000 ml, add a small amount of the methyl orange solution (3.2.2) and mix.

4.2.4 Sodium hydroxide, ρ approximately 1,22 g/ml, about 20 % *(m/m)* or approximately 6 N solution.

Dissolve 120 g of sodium hydroxide in water. Cool to ambient temperature, dilute to 500 ml and mix thoroughly.

4.3 Apparatus

Ordinary laboratory apparatus and

4.3.1 Apparatus as shown in the figure.

4.4 Procedure

4.4.1 *Test portion*

Weigh, to the nearest 0,001 g, 4,9 to 5,1 g of the test sample (see ISO 1686).

4.4.2 *Determination*

4.4.2.1 PREPARATION OF THE APPARATUS

Fill the burette (B) of the apparatus (4.3.1) with the sodium chloride solution (4.2.3), through the levelling bottle (F).

Pour into the absorber (C) the sodium hydroxide solution (4.2.4). (Renew this solution after approximately 100 determinations.)

The burette (B) and the absorber (C) being filled up to the stopcock (R_1) and the mark (*a*) respectively, and the stopcocks (R_1) and (R_2) being closed, quantitatively transfer the test portion (4.4.1), previously mixed with 10 ml of water, to the 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 the flask (A) plus the volume of the condenser tube (D) up to the cock (R_1)).

Place in the flask (A) three porcelain or glass beads of diameter approximately 2 mm, together with a few pieces of pumice stone, the volume of which should be approximately equal to that of the beads. Close the flask and the stopcock (R).

Open the stopcock (R_1) to connect the flask (A) and the burette (B) and lower the levelling bottle (F).

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

4.4.2.2 EVOLUTION AND MEASUREMENT OF CARBON DIOXIDE

By means of the dropping funnel, pour 20 ml of the hydrochloric acid solution (4.2.2) into the flask (A), taking care to avoid loss of gas.

Heat the flask and maintain the solution at boiling point for 5 min while running cold water through the condenser. Then stop heating and add sodium chloride solution (4.2.3) by means of the funnel, lowering the levelling bottle (F) still further so as to make the solution in the flask (A) rise in the condenser tube up to the cock (R_1). Then close the latter and wait 5 min to allow the gas to reach the temperature of the water-jacket.

Measure the volume of gas, V_0, 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 sodium chloride solution (4.2.3) 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 the burette (B) and the 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 the burette (B) by lowering the levelling bottle (F) and, after adjusting the level of the sodium hydroxide solution (4.2.4) to the gauge mark (*a*) on the absorber (C), close the cock (R_2) and read the volume of the residual gas. Repeat these operations until a constant volume V_1 is obtained.

The difference ($V_0 - V_1$) represents the volume of the carbon dioxide contained in the test portion, measured at atmospheric pressure and at the temperature of the water-jacket.

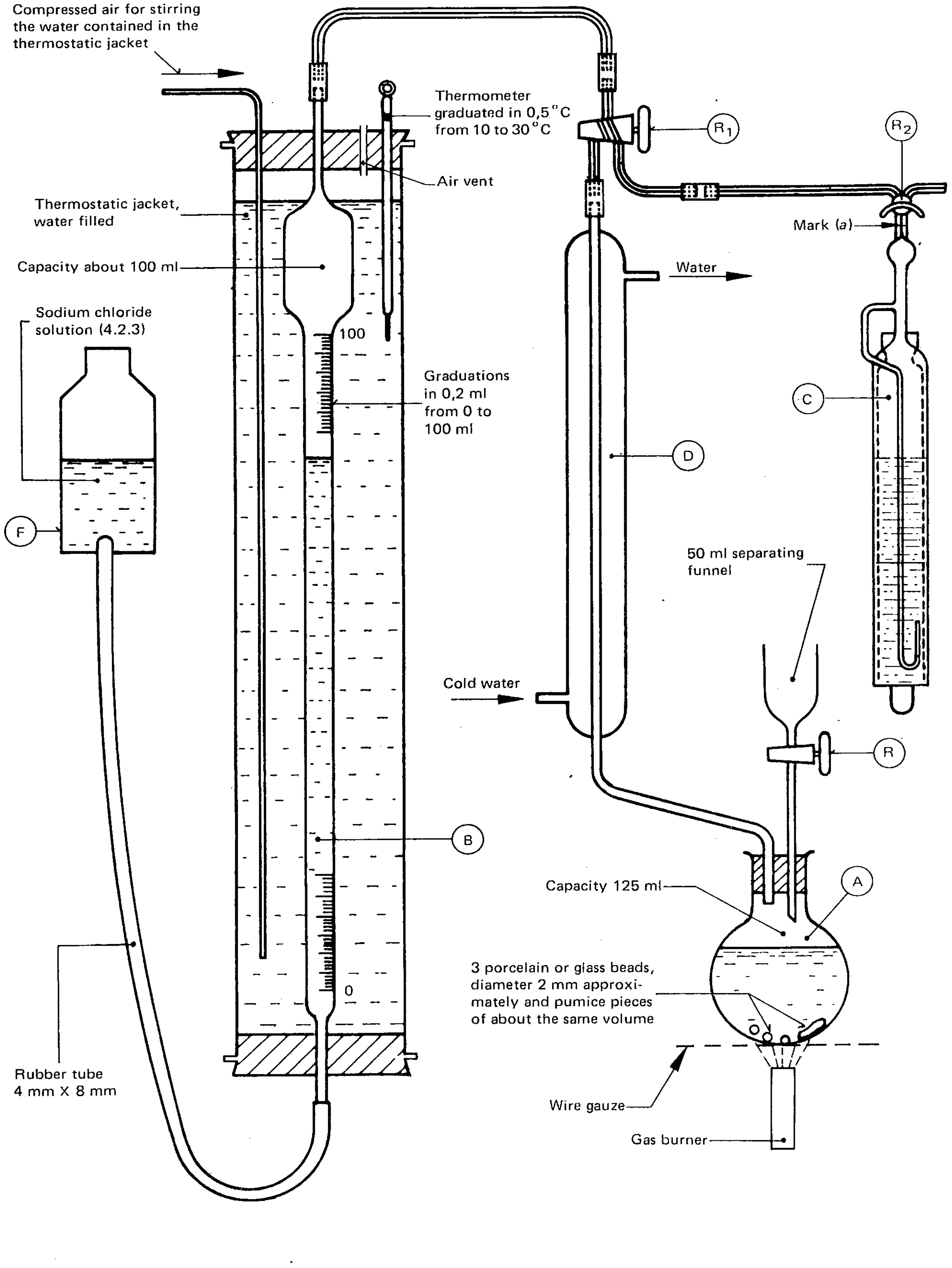

FIGURE – **Apparatus for gas-volumetric determination of carbonates content**

4.5 Expression of results

The content of sodium carbonate (Na_2CO_3) or of potassium carbonate (K_2CO_3), expressed as a percentage by mass, is given, according to the case, by the formulae

Na_2CO_3, % (m/m)

$$= (V_0 - V_1) \times \frac{273}{273 + t} \times \frac{P - p}{1\,013} \times 0{,}001\,98 \times \frac{106}{44} \times \frac{100}{m}$$

$$= 0{,}128 \times \frac{P - p}{273 + t} \times \frac{V_0 - V_1}{m}$$

K_2CO_3, % (m/m)

$$= (V_0 - V_1) \times \frac{273}{273 + t} \times \frac{P - p}{1\,013} \times 0{,}001\,98 \times \frac{138{,}2}{44} \times \frac{100}{m}$$

$$= 0{,}167 \times \frac{P - p}{273 + t} \times \frac{V_0 - V_1}{m}$$

where

V_0 is the volume, in millilitres, of the gas before absorption of the carbon dioxide;

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

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

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

NOTE — If the atmospheric pressure P is measured in pascals (or kilopascals), multiply this value by 0,01 (or by 10) to convert it to millibars.

p is the vapour pressure, in millibars, of the solution sodium chloride (4.2.3) 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 mbar	11	12	13	15	17	19	21	24	27	30	33

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

0,001 98 is the factor for the conversion of the volume of carbon dioxide, in millilitres, to the mass of carbon dioxide, in grams;

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

$\frac{138{,}2}{44}$ is the factor for the conversion of the mass of carbon dioxide to the corresponding mass of K_2CO_3.

4.6 Precision of the method

The results obtained with this method are reproducible to the nearest ± 0,04 % (m/m) in absolute value.

5 SODIUM OR POTASSIUM SILICATES CONTAINING SULPHIDES

In the case of sodium or potassium silicates containing sulphides, the acidification of the product causes the liberation of hydrogen sulphide which is absorbed in the sodium 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 5 g of sulphides, expressed as Na_2S, per kilogram.

5.1 Principle

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

Determination according to the method specified in clause 4.

5.2 Reagents

Reagents specified in 3.2 and 4.2, and :

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

5.3 Apparatus

See 4.3.

5.4 Procedure

Follow the procedure specified in 4.4, applying the following modifications :

- in 4.4.2.1, 4th paragraph, place in the flask (A), before the porcelain or glass beads, 5 drops of the hydrogen peroxide solution (5.2.1);
- at the end of 4.4.2.1, open the cock (R_1), lower the levelling bottle (F) and boil the contents of the flask (A) for 5 min, while running cold water through the condenser. Cool to a temperature slightly above ambient temperature, to prevent the liquid in the burette from being drawn into the condenser.

5.5 Expression of results

See 4.5.

6 SODIUM OR POTASSIUM SILICATES CONTAINING CHLORATES

In the case of sodium or potassium silicates containing chlorates, acidification of the product causes the liberation of chlorine, which is absorbed in the sodium 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 10 g of chlorates, expressed as $NaClO_3$, 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 specified 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 4.4.2.1, 3rd paragraph, place in the flask (A), after the test portion (4.4.1), 5 ml of the iron(II) sulphate solution (6.2.1).

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.

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM AND POTASSIUM SILICATES FOR INDUSTRIAL USE

ISO 1686 – Samples and methods of test – General.

ISO 1687 – Determination of density at 20 °C of samples in solution – Methods using density hydrometer and pyknometer.

ISO 1688 – Determination of dry matter – Gravimetric method.

ISO 1689 – Calculation of the ratio $\frac{SiO_2}{Na_2O}$ or $\frac{SiO_2}{K_2O}$.

ISO 1690 – Determination of silica content – Gravimetric method by insolubilization.

ISO 1691 – Determination of carbonates content – Gas-volumetric method.

ISO 1692 – Determination of total alkalinity – Titrimetric method.

ISO 2122 – Preparation of solution of products not easily soluble in boiling water and determination of matter insoluble in water.

ISO 2123 – Determination of dynamic viscosity.

ISO 2124 – Determination of silica content – Titrimetric method.

ISO 3200 – Determination of sulphates content – Barium sulphate gravimetric method.

ISO 3201 – Determination of iron content – 1,10-Phenanthroline photometric method.