INTERNATIONAL STANDARD 2051

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Potassium chloride for industrial use — Determination of potassium content — Potassium tetraphenylborate gravimetric method

Chlorure de potassium à usage industriel — Dosage du potassium — Méthode gravimétrique à l'état de tétraphénylborate de potassium

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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 2051 and found it technically suitable for transformation. International Standard ISO 2051 therefore replaces ISO Recommendation R 2051-1971 to which it is technically identical.

ISO Recommendation R 2051 was approved by the Member Bodies of the following countries :

Austria	Hungary	Poland
Belgium	India	South Africa, Rep. of
Chile	Iran	Spain
Czechoslovakia	Israel	Switzerland
Egypt, Arab Rep. of	Italy	Thailand
France	Korea, Rep. of	United Kingdom
Germany	Netherlands	U.S.A.
Greece	New Zealand	U.S.S.R.

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Portugal*

* Subsequently, this Member Body approved the Recommendation.

No Member Body disapproved the transformation of ISO/R 2051 into an International Standard.

Printed in Switzerland

Potassium chloride for industrial use — Determination of potassium content — Potassium tetraphenylborate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a potassium tetraphenylborate gravimetric method for the determination of the potassium content of potassium chloride for industrial use, i.e. of a product containing a minimum of about 95 % (*m/m*) KCl. This limit, expressed conventionally as K or K_2O, corresponds to approximately 50 % (*m/m*) or 60 % (*m/m*), respectively.

2 REFERENCE

ISO 2053, *Potassium chloride for industrial use — Determination of moisture content — Gravimetric method.*

3 PRINCIPLE

Dissolution of a test portion taken from the laboratory sample, previously ground and sifted.

Addition of formaldehyde solution to transform any ammonium ions present into hexamethylenetetramine, and of ethylenedinitrilotetraacetic acid, disodium salt, to complex any extraneous cations present which could cause excess errors.

NOTE — These additions, which make the method of very general application, in no case have a detrimental effect on its precision or reproducibility.

Precipitation of the potassium by sodium tetraphenylborate in a weakly alkaline medium.

Drying and weighing of the precipitate.

4 REAGENTS

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

4.1 Sodium hydroxide, approximately 400 g/l solution.

4.2 Ethylenedinitrilotetraacetic acid (EDTA), disodium salt, dihydrate, 40 g/l solution.

4.3 Sodium tetraphenylborate, alkaline solution.

Dissolve 32,5 g of sodium tetraphenylborate, $Na[B(C_6H_5)_4]$, in 480 ml of water. Add 2 ml of the sodium hydroxide solution (4.1) and 20 ml of magnesium chloride hexahydrate ($MgCl_2.6H_2O$) 100 g/l solution. Stir with a magnetic stirrer for 15 min and filter through a fine grain filter.

Replace this reagent each week and filter immediately before use.

4.4 Wash solution, saturated solution at ambient temperature of potassium tetraphenylborate.

Precipitate 0,5 g of potassium tetraphenylborate in a solution of potassium chloride. Filter the precipitate and wash with distilled water. Bring the precipitate back into suspension in 5 l of water and shake for about 1 h. Immediately before use, filter the quantity of reagent required for the determination.

4.5 Formaldehyde, 30 % (*m/m*) solution.

Filter immediately before use.

4.6 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Filter crucible, glass or porcelain with suitered disk of porosity grade P16 (pore diameter between 10 and 16 μm).

6 PROCEDURE

6.1 Preparation of test sample

Grind the laboratory sample until it passes completely through a 500 μm nominal mesh sieve.

NOTE — The moisture content of the laboratory sample may vary appreciably as a result of grinding and sifting. It is advisable to determine the moisture content of the ground and sifted product (test sample) before determining its potassium content so as to be able to relate it to the untreated product (laboratory sample), the moisture content of which shall also be determined.

6.2 Determination of moisture content of laboratory sample and of test sample

Determine the moisture content of the two samples by the method specified in ISO 2053.

6.3 Test portion

Weigh, to the nearest 0,01 g, 10 g of the test sample, prepared as specified in 6.1.

6.4 Preparation of test solution

Place the test portion (6.3) in a 400 ml beaker and add 200 ml of cold water. Heat and keep boiling gently for 15 min. Cool to 20 °C, transfer the contents of the beaker quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water at 20 °C and mix. This becomes solution A.

Filter a sufficient quantity of solution A through a dry filter paper, discarding the first portions of the filtrate and retaining this in a dry receiver.

6.5 Determination

Place 50,0 ml of the filtrate of solution A in a 500 ml one-mark volumetric flask and dilute to the mark. Take 50,0 ml of this solution and place in a 250 ml beaker.

Add 10 ml of the disodium EDTA solution (4.2), a few drops of the phenolphthalein solution (4.6) and, drop by drop, the sodium hydroxide solution (4.1) until the appearance of a pure red coloration. Then add 10 ml of the formaldehyde solution (4.5) and a few drops of the sodium hydroxide solution (4.1) until the appearance of a pure red colour. Cover the beaker with a watch-glass and heat for 15 min on a boiling water bath. The solution should remain red, otherwise add a few drops of the phenolphthalein solution and, if necessary, restore the colour by adding, drop by drop, the sodium hydroxide solution (4.1).

Add, drop by drop, while stirring, 10 ml of the sodium tetraphenylborate solution (4.3). Continue the stirring for about 1 min, then cool rapidly below 20 °C under a stream of water. Allow to stand for 10 min, then filter through the crucible (5.1) previously heated at 120 °C, allowed to cool in a desiccator to ambient temperature and weighed to the nearest 0,000 1 g. Rinse the precipitate with the wash solution (4.4) and wash successively with the same solution.

Dry the crucible and its contents for 90 min at 120 °C, allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,000 1 g.

7 EXPRESSION OF RESULTS

7.1 The potassium content, expressed as a percentage by mass of potassium (K), in the test sample (ground and sifted product), is given by the formula :

$$10\,908 \times \frac{m_1}{5\,m_0} = 2\,181{,}6 \times \frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.3);

m_1 is the mass, in grams, of the dried precipitate of potassium tetraphenylborate.

7.2 The potassium content, expressed as a percentage by mass of potassium (K), in the laboratory sample (untreated product), is given by the formula :

$$10\,908 \times \frac{m_1}{5\,m_0} \times \frac{100-H}{100-h} = 2\,181{,}6 \times \frac{m_1}{m_0} \times \frac{100-H}{100-h}$$

where

m_0 and m_1 are as defined in 7.1;

H is the moisture content, as a percentage by mass, of the laboratory sample (untreated product);

h is the moisture content, as a percentage by mass, of the test sample (ground and sifted product).

7.3 The potassium content, expressed as a percentage by mass of potassium oxide (K_2O), in the laboratory sample (untreated product), is given by the formula :

$$13\,140 \times \frac{m_1}{5\,m_0} \times \frac{100-H}{100-h} = 2\,628 \times \frac{m_1}{m_0} \times \frac{100-H}{100-h}$$

where

m_0 and m_1 are as defined in 7.1;

H and h are as defined in 7.2.

8 REPRODUCIBILITY

Comparative analyses carried out in fourteen laboratories, with two operators in each, working in duplicate on two samples, gave the statistical information shown in the following table :

Sample No.	Total number of determinations	K_2O, % (*m/m*) Arithmetic mean	Standard deviation
1	54	61,41	0,16
2 (containing about 5 % (*m/m*) of $MgSO_4$)	54	51,40	0,15

9 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 the International Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO POTASSIUM CHLORIDE FOR INDUSTRIAL USE

ISO 2050 — Determination of potassium content — Flame emission spectrophotometric method.

ISO 2051 — Determination of potassium content — Potassium tetraphenylborate gravimetric method.

ISO 2052 — Determination of potassium content — Sodium tetraphenylborate titrimetric method.

ISO 2053 — Determination of moisture content — Gravimetric method.