INTERNATIONAL STANDARD 2052

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

Chlorure de potassium à usage industriel — Dosage du potassium — Méthode titrimétrique au tétraphénylborate de sodium

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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 2052 and found it technically suitable for transformation. International Standard ISO 2052 therefore replaces ISO Recommendation R 2052-1971 to which it is technically identical.

ISO Recommendation R 2052 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 2052 into an International Standard.

Printed in Switzerland

Potassium chloride for industrial use – Determination of potassium content – Sodium tetraphenylborate titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a sodium tetraphenylborate titrimetric 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 previously ground and sifted laboratory sample and elimination of any ammonium ions present.

Precipitation of the potassium, in a slightly acid medium, by an excess of a standard volumetric sodium tetraphenylborate solution.

Filtration and back-titration of this excess in the filtrate, with a standard volumetric solution of a quaternary ammonium salt, using bromophenol blue as indicator.

4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade, and only distilled water or water of equivalent purity. In particular, use only reagents free from potassium and ammonium, or having a negligible content of these ions.

4.1 Hydrochloric acid, approximately 0,1 N solution.

4.2 Quaternary ammonium salt, approximately 0,01 N solution.

Dissolve 3,65 g of cetyltrimethylammonium bromide $[CH_3(CH_2)_{15}\ N(CH_3)_3]$ Br, or an equivalent quantity of another quaternary ammonium salt, in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix, stirring gently to avoid the formation of foam.

NOTE – As the quaternary ammonium salt, it is possible to use a commercial solution, intended for pharmaceutical purposes, of alkylbenzyldimethyl-ammonium chloride (or benzalconium chloride), formula $[C_6H_5CH_2N(CH_3)_2C_nH_{2n+1}]$ Cl, in which n varies from 8 to 18.

4.3 Sodium tetraphenylborate, approximately 0,02 N solution.

Weigh, to the nearest 0,000 1 g, 6,845 g of sodium tetraphenylborate $[NaB(C_6H_5)_4]$ and dissolve, in a 1 000 ml beaker, with about 800 ml of water, stirring mechanically for 10 min.

Add 10 to 15 drops of a saturated solution of aluminium chloride, 5 to 10 drops of a 1 g/l solution of bromothymol blue of 1 g/l in 95 % (*V/V*) ethanol and finally, drop by drop, while stirring, an approximately 2 N solution of sodium hydroxide until the indicator turns to a pure blue colour. Leave to stand for a few minutes, then filter the solution and collect the filtrate in a 1 000 ml one-mark volumetric flask. Rinse the beaker, wash the precipitate, collecting the washings in the same flask, dilute to the mark and mix thoroughly.

This solution will generally keep well for several weeks. It is advisable to filter it if a precipitate occurs.

Before using this solution, leave it to age for 48 h.

4.3.1 *Determination of the equivalence of the quaternary ammonium salt solution and the sodium tetraphenylborate solution*

Before proceeding with the standardization of the sodium tetraphenylborate solution, it is advisable to determine the equivalence of this solution with that of the quaternary ammonium salt which is to serve to determine the excess by back-titration. It is, in fact, this equivalence that makes it possible to calculate, by difference, the volume of sodium tetraphenylborate solution that has reacted with the potassium. It is essential to check each time in advance that the latter solution is restandardized.

To do this, proceed as follows :

Place 10,00 ml of the sodium tetraphenylborate solution (4.3) in a 50 ml one-mark volumetric flask, dilute to the mark and mix.

Take 10,00 ml of the resultant solution, containing 2 ml of the sodium tetraphenylborate solution (4.3), and place in a 100 ml conical flask. Add 10 drops of the bromophenol blue solution (4.5), the volume of hydrochloric acid

solution (4.1) necessary to cause the colour of the indicator to change to pure yellow, and then an excess of 2 ml of this acid.

Titrate, drop by drop, with the quaternary ammonium salt solution (4.2) until the colour of the indicator changes from yellow to green.

According to the volume used, read to the nearest 0,01 ml, adjust the concentration of this solution so that 2,00 ml is exactly equivalent to 1,00 ml of the sodium tetraphenylborate solution. The volume of quaternary ammonium salt solution to be used for the above titration should, therefore, be 4,00 ml.

4.3.2 *Standardization of the sodium tetraphenylborate solution*

Place 20,00 ml of the standard reference potassium chloride solution (4.4) in a 50 ml one-mark volumetric flask.

Add, in succession, 2 drops of the bromophenol blue solution (4.5), the volume of the hydrochloric acid solution (4.1) necessary to make the colour of the indicator turn to pure yellow and 25,00 ml of the sodium tetraphenylborate solution (4.3). Dilute to the mark, mix and leave to stand for about 5 min.

Pass through a dry filter intended for normal filtration, discarding the first portions of the filtrate and collecting the filtered solution in a dry vessel.

Take 25,00 ml of the filtrate and place in a 100 ml conical flask. Add successively 10 drops of the bromophenol blue solution (4.5), then the volume of the hydrochloric acid solution necessary to make the colour of the indicator turn to pure yellow, plus an excess of 2 ml of this acid. Titrate immediately, drop by drop, with the quaternary ammonium salt solution (4.2) until the colour of the indicator changes from yellow to green, then read the volume used to the nearest 0,01 ml.

The potassium content, expressed in milligram-equivalents, of the sodium tetraphenylborate solution is given by the formula :

$$T = \frac{0,4}{25 - \left(\frac{V_0}{2} \times \frac{50}{25}\right)} = \frac{0,4}{25 - V_0}$$

where

T is the number of milligram-equivalents of potassium corresponding to 1 ml of the sodium tetraphenylborate solution (4.3), expressed to four decimal places;

V_0 is the volume, in millilitres, of quaternary ammonium salt solution (4.2) used for the titration, expressed to two decimal places.

4.4 **Potassium chloride,** 0,02 N standard reference solution.

Weigh, to the nearest 0,000 1 g, 1,491 g of potassium chloride previously dried in an oven controlled at 105 to 110 °C for 2 h, then allowed to cool to ambient temperature in a desiccator, and place in a 1 000 ml one-mark volumetric flask. Dissolve in a little water, dilute to the mark and mix.

4.5 **Bromophenol blue,** 0,4 g/l solution.

In a 100 ml one-mark volumetric flask, dissolve 0,040 g of bromophenol blue in about 3 ml of approximately 0,01 N sodium hydroxide solution. Dilute to the mark with water and mix.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 **Microburette,** of capacity 10 ml, graduated in 0,01 ml.

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 contents of the two samples by the method specified in ISO 2053.

6.3 Test portion

Weigh, to the nearest 0,001 g, a mass of the test sample prepared as specified in 6.1, containing a maximum of 500 milligram-equivalents of potassium, i.e. about 37 g of potassium chloride.

6.4 Preparation of test solution

Place the test portion (6.3) in a 1 000 ml beaker and dissolve it in 800 ml of boiling water.

Cool to ambient temperature, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark, and mix. If the solution is cloudy, filter it through a dry filter paper, discard the first portions of the filtrate and collect the remainder in a dry receiver. Take, by means of a burette, 40,0 ml of this solution, place in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

6.5 Determination

Take 20,00 ml of the test solution (6.4) and place in a 50 ml one-mark volumetric flask.

Add, in succession, 2 drops of the bromophenol blue solution (4.5), the volume of the hydrochloric acid solution (4.1) necessary to make the colour of the indicator turn to pure yellow and 25,00 ml of the sodium tetraphenylborate solution (4.3). Dilute to the mark, mix and allow to stand for about 5 min.

Pass through a dry filter intended for normal filtration, discarding the first portions of the filtrate and collecting the filtered solution in a dry vessel.

Take 25,00 ml of the filtrate and place in a 100 ml conical flask. Add successively 10 drops of the bromophenol blue solution, then the volume of the hydrochloric acid solution necessary to make the colour of the indicator turn to pure yellow, plus an excess of 2 ml of this acid. Titrate immediately, drop by drop, with the quaternary ammonium salt solution (4.2) until the colour of the indicator changes from yellow to green, then read the volume used to the nearest 0,01 ml.

NOTE — If the presence of ammonium ions in the sample is suspected, it is advisable to apply the following procedure :

Take 20,00 ml of the test solution (6.4) and place in a 100 ml beaker. Add 1 ml of an approximately 2 N sodium hydroxide solution, and evaporate to dryness. Add a few millilitres of water and heat to dissolve the salts. Cool, add, in succession, 2 drops of bromophenol blue solution (4.5) and the volume of hydrochloric acid solution (4.1) necessary to make the colour of the indicator turn to pure yellow. Transfer the solution quantitatively to a 50 ml one-mark volumetric flask, add 25,00 ml of the sodium tetraphenylborate solution (4.3), dilute to the mark, mix and allow to stand for about 5 min. Continue the determination from the third paragraph.

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 :

$$4\,887{,}6 \times T \times \frac{25 - V_1}{m}$$

where

T is the number of milligram-equivalents of potassium corresponding to 1 ml of the sodium tetraphenylborate solution (4.3), expressed to four decimal places (see 4.3.2);

V_1 is the volume, in millilitres, of the quaternary ammonium salt solution (4.2) used for the titration, expressed to two decimal places;

m is the mass, in grams, of the test portion, expressed to three decimal places.

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 :

$$4\,887{,}6 \times T \times \frac{25 - V_1}{m} \times \frac{100 - H}{100 - h}$$

where

T, V_1 and m 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 :

$$5\,887{,}5 \times T \times \frac{25 - V_1}{m} \times \frac{100 - H}{100 - h}$$

where

T, V_1 and m are as defined in 7.1;

H and h are as defined in 7.2.

8 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.

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