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POTASSIUM CHLORIDE FOR INDUSTRIAL USE
DETERMINATION OF POTASSIUM CONTENT
SODIUM TETRAPHENYLBORATE VOLUMETRIC METHOD

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 2052, *Potassium chloride for industrial use — Determination of potassium content — Sodium tetraphenylborate 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. 2052, which was circulated to all the ISO Member Bodies for enquiry in July 1970.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

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.

ISO Recommendation R 2052 August 1971

POTASSIUM CHLORIDE FOR INDUSTRIAL USE

DETERMINATION OF POTASSIUM CONTENT

SODIUM TETRAPHENYLBORATE VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of potassium in potassium chloride for industrial use.

2. FIELD OF APPLICATION

The method described is applicable to the determination of potassium content in potassium chloride for industrial use, i.e. in a product containing a minimum of about 95 % KCl. This limit, expressed conventionally as K or K_2O, corresponds to approximately 50 % or 60 %, respectively.

3. PRINCIPLE

Dissolution of a test portion taken from the previously ground and sifted laboratory sample and elimination of ammonium ions if 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

Distilled water or water of equivalent purity should be used in the test. 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 1000 ml one-mark volumetric flask. Dilute to the mark and mix, stirring gently to avoid the formation of foam.

NOTE. – As 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 1 mg, 6.845 g of sodium tetraphenylborate ($NaB(C_6H_5)_4$) and dissolve, in a 1000 ml beaker, with about 800 ml of water, stirring mechanically for 10 minutes.

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 1000 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 hours.

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, indeed, this equivalence that makes it possible to calculate, by difference, the volume of sodium tetraphenylborate 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 thoroughly.

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 solution (4.1).

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 sodium tetraphenylborate solution (4.3). The volume of quaternary ammonium salt solution (4.2) 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 potassium chloride solution (4.4) in a 50 ml one-mark volumetric flask.

Add, in sucession, 2 drops of the bromophenolblue solution (4.5), the volume of 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 minutes.

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 10 drops of the bromophenol blue solution (4.5), then the volume of hydrochloric acid solution (4.1) necessary to make the colour of the indicator turn to pure yellow, plus an excess of 2 ml of this solution (4.1). Without waiting, titrate, 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.

Calculate the potassium content of the sodium tetraphenylborate solution (4.3), expressed in milligramme equivalents, by the following 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 milligramme equivalents of potassium corresponding to 1 ml of the sodium tetraphenylborate solution (4.3), expressed to four decimal places;

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

4.4 *Potassium chloride,* 0.02 N standard solution.

In a 1000 ml one-mark volumetric flask, dissolve 1.491 g of potassium chloride previously dried in an oven at a temperature of 105 to 110 °C for 2 hours, then cooled to room temperature in a desiccator.

Dilute to the mark and mix thoroughly.

4.5 *Bromophenol blue,* 0.4 g/l solution.

In a 100 ml one-mark volumetric flask, dissolve 0.040 g of bromophenol blue using about 3 ml of approximately 0.01 N sodium hydroxide solution. Dilute to the mark with water and mix thoroughly.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *10 ml microburette,* 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 original product (laboratory sample), the moisture content of which should also be determined.

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

Determine the moisture contents of the two samples by the method described in ISO Recommendation R 2053, *Potassium chloride for industrial use – Determination of moisture content – Gravimetric method.*

6.3 Test portion

Weigh, to the nearest 0.001 g, a mass of the test sample prepared as described in clause 6.1, containing a maximum of 500 milligramme equivalents of potassium, i.e. about 37 g of potassium chloride.

6.4 Preparation of the sample solution

Place the test portion (6.3) in a 1000 ml beaker and dissolve it with 800 ml of boiling water.

Cool to ambient temperature, transfer quantitatively to a 1000 ml one-mark volumetric flask, dilute to the mark, and mix thoroughly. If the solution is cloudy, filter it through a dry filter paper, reject 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 1000 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

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* See ISO Recommendation R 565, *Woven wire cloth and perforated plates in test sieves – Nominal sizes of apertures,* Table 1.

6.5 Determination

Take 20.00 ml of the sample solution (6.4) and place in a 50 ml one-mark volumetric flask.

Add, in succession, 2 drops of bromophenol blue solution (4.5), the volume of 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 minutes.

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 10 drops of the bromophenol blue solution (4.5), then the volume of hydrochloric acid solution (4.1) necessary to make the colour of the indicator turn to pure yellow, plus an excess of 2 ml of this solution (4.1). Without waiting, titrate, 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 sample solution (6.4) and place in a 100 ml beaker. Add 1 ml of a sodium hydroxide solution, 2 N approximately, 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 minutes. Continue the determination from the third paragraph of clause 6.5.

7. EXPRESSION OF RESULTS

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

$$4887.6 \times T \times \frac{25 - V_1}{m}$$

where

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

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

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

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

$$4887.6 \times T \times \frac{25 - V_1}{m} \times \frac{100 - H}{100 - h}$$

where

H is the percentage by mass of moisture in the laboratory sample (untreated product);

h is the percentage by mass of moisture in the test sample (ground and sifted product).

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

$$5887.5 \times T \times \frac{25 - V_1}{m} \times \frac{100 - H}{100 - h}$$