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R 2051

POTASSIUM CHLORIDE FOR INDUSTRIAL USE

DETERMINATION OF POTASSIUM CONTENT

GRAVIMETRIC METHOD AS POTASSIUM TETRAPHENYLBORATE

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BRIEF HISTORY

The ISO Recommendation R 2051, *Potassium chloride for industrial use – Determination of potassium content – Gravimetric method as potassium tetraphenylborate*, 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. 2051, 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	India	South Africa, Rep. of
Belgium	Iran	Spain
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
France	Korea, Rep. of	U.A.R.
Germany	Netherlands	United Kingdom
Greece	New Zealand	U.S.A.
Hungary	Poland	U.S.S.R.

The following Member Body opposed the approval of the Draft :

Portugal

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 2051 | August 1971

POTASSIUM CHLORIDE FOR INDUSTRIAL USE

DETERMINATION OF POTASSIUM CONTENT

GRAVIMETRIC METHOD AS POTASSIUM TETRAPHENYLBORATE

1. SCOPE

This ISO Recommendation describes a gravimetric 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 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 laboratory sample, previously ground and sifted.

Addition of formaldehyde to transform any ammonium ions present into hexamethylenetetramine and of the disodium salt of ethylenediamine tetra-acetic acid to complex any extraneous cations present which could cause excess errors.*

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

Drying and weighing of the precipitate.

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Sodium hydroxide*, approximately 400 g/l solution.

4.2 *Disodium salt of ethylenediamine tetra-acetic acid* (EDTA) 40 g/l solution.

Dissolve 40 g of disodium ethylenediamine tetra-acetate dihydrate in water and dilute to 1000 ml.

4.3 *Sodium tetraphenylborate*, alkaline solution.

Dissolve 32.5 g of sodium tetraphenylborate in 480 ml of water. Add 2 ml of sodium hydroxide solution (4.1) and 20 ml of magnesium chloride ($MgCl_2 \cdot 6H_2O$) 100 g/l solution. Stir with a magnetic stirrer for 15 minutes and filter through a fine grain filter.

Replace this reagent each week and filter immediately before use.

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

4.4 *Wash solution* (saturated solution of potassium tetraphenylborate).

Precipitate 0.5 g of potassium tetraphenylborate in a solution of pure potassium chloride; filter it and wash with distilled water. Bring the precipitate back into suspension in 5 litres of water and shake for about 1 hour. Immediately before use, filter the quantity of reagent required for the determination.

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

Filter 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, base having a pore diameter of 10 to 20 μm.

6. PROCEDURE

6.1 Preparation of the 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 content 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 0.01 g, 10 g of the test sample, prepared as described in clause 6.1.

6.4 Preparation of the sample 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 minutes. Cool to 20 °C, transfer the contents of the beaker quantitatively to a 1000 ml one-mark volumetric flask, dilute to the mark with water at 20 °C and mix thoroughly. 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.

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

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 EDTA solution (4.2), a few drops of phenolphthalein (4.6) and, drop by drop, the sodium hydroxide solution (4.1) until there is a pure red coloration. Then add 10 ml of formaldehyde solution (4.5) and a few drops of sodium hydroxide solution (4.1) until there is a pure red colour. Cover the beaker with a watch glass and heat for 15 minutes on a boiling water bath. The solution should remain red, otherwise add a few drops of the phenolphthalein solution (4.6) 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 minute then cool rapidly below 20 °C under a stream of water. Allow to stand for 10 minutes, then filter through the crucible (5.1) previously heated at 120 °C, cooled in a desiccator and weighed to the nearest 1 mg. Rinse the precipitate with the wash solution (4.4) and wash successively with the same solution.

Dry the crucible and its contents for 90 minutes at 120 °C, allow to cool in a desiccator and weigh.

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 :

$$10\,908 \times \frac{m_2}{5\,m_1} = 2181.6 \times \frac{m_2}{m_1}$$

where

m_1 is the mass, in grammes, of the test portion;

m_2 is the mass, in grammes, of the dried precipitate.

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 :

$$10\,908 \times \frac{m_2}{5\,m_1} \times \frac{100 - H}{100 - h} = 2181.6 \times \frac{m_2}{m_1} \times \frac{100 - H}{100 - h}$$

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 :

$$13\,140 \times \frac{m_2}{5\,m_1} \times \frac{100 - H}{100 - h} = 2628 \times \frac{m_2}{m_1} \times \frac{100 - H}{100 - h}$$