INTERNATIONAL STANDARD ISO 2050

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Potassium chloride for industrial use — Determination of potassium content — Flame emission spectrophotometric method

Chlorure de potassium à usage industriel — Dosage du potassium — Méthode par spectrophotométrie de flamme en émission

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FOREWORD

ISO (the International Organization for Standardization) is a worlwide 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 2050 and found it technically suitable for transformation. International Standard ISO 2050 therefore replaces ISO Recommendation R 2050-1971 to which it is technically identical.

ISO Recommendation R 2050 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 2050 into an International Standard.

Printed in Switzerland

Potassium chloride for industrial use — Determination of potassium content — Flame emission spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

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

Acidification to pH 1 after suitable dilution, and atomization of the resultant solution in the burner of a flame spectrophotometer.

Determination of the potassium content by measurement of the intensity of the 766 nm ray, compared with the intensity obtained for standard potassium chloride solutions acidified in the same way.

4 REAGENTS

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

4.1 Water must satisfy the following additional requirement :

With the spectrophotometer adjusted to give a reading of 0 (minimum of the scale) for the water (4.1) and approximately 100 (maximum of the scale) for the standard matching solution of potassium containing 10 mg/l (see 6.3.1), the reading obtained for the water evaporated to one-fiftieth of its volume in a potassium-free container (platinum, silicon, etc.), shall not exceed 10.

4.2 Sulphuric acid, approximately 1 N solution, practically free from potassium. This solution, diluted ten times, shall give a spectrophotometer reading of not more than 2, under the conditions specified in 4.1.

4.3 Potassium, standard solution, corresponding to 4,000 g of potassium (K) per litre.

Weigh, to the nearest 0,001 g, 7,627 g of potassium chloride (KCl) previously dried at approximately 400 °C and then allowed to cool in a desiccator. Dissolve this in the water (4.1), dilute to 1 000 ml in a one-mark volumetric flask and mix thoroughly.

1 ml of this standard solution contains 4 mg of K.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Filter paper, of a sufficiently fine texture to retain all matter likely to choke the opening of the atomizer or the spectrophotometer burner, and which does not itself release troublesome fragments.

5.2 Flame spectrophotometer, fitted with an atomizer-burner, fed in such a way as to excite the emission of the 766 nm spectral ray of potassium.

NOTE — In the presence of cations other than potassium, and especially of sodium, the intensity of the radiations of the potassium is increased, the more so the hotter the flame. In order to render this interference from cations negligible, it is advisable to use as "cold" a flame as possible such as the butane-air complete combustion flame.

The sensitivity of the apparatus shall be adjustable within wide limits, for example, for concentrations of potassium of 10 to 100 mg/l, at the bottom of the scale. In addition, after bringing the spectrophotometer into a state of normal operation for 30 to 60 min, the readings shall be reproducible and stable 20 to 40 s after the start of atomization.

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 Preparation of calibration graph

6.3.1 *Preparation of the standard matching solutions*

In a series of ten 1 000 ml one-mark volumetric flasks, each already containing 100 ml of the sulphuric acid solution (4.2), place the volumes of the standard potassium solution (4.3) shown in the following table :

Standard potassium solution (4.3)	Corresponding mass of potassium (K)
ml	mg
2,5	10
5,0	20
7,5	30
10,0	40
12,5	50
15,0	60
17,5	70
20,0	80
22,5	90
25,0	100

Dilute to the mark with the water (4.1) and mix.

The acidity of these standard matching solutions is approximately 0,1 N.

NOTE — These concentrations may be adjusted according to the characteristics of the apparatus used, so that the measurements may be taken in the best sensitivity range of the equipment used.

In addition, the intensity of the radiations of the potassium may differ, at equal concentrations, according to the anion present and according to the pH of the solution. The interference due to the anion is negligible in the conditions of this determination. The influence of the pH is nil when the precaution is taken of fixing its value between 0,9 and 1,1, both in the solutions to be analysed and in the standard matching solutions.

6.3.2 *Photometric measurements*

Switch on the spectrophotometer (5.2) in advance to allow sufficient time for its stabilization. Adjust the sensitivity of the apparatus and the opening of the slit according to the characteristics of the apparatus used and to ensure a maximum width of the moving band of 12 nm, centred on the maximum emission (theoretical value 766 nm).

Atomize the standard matching solutions in succession in the centre of the flame and measure the intensity of the radiations emitted in each case.

Care should be taken to keep the quantity of the solutions atomized in the flame constant per unit of time throughout the duration of the measurements.

6.3.3 *Plotting of the calibration graph*

Plot a graph having, for example, as abscissae the concentration of potassium, expressed in milligrams per litre, and as ordinates, on a logarithmic scale, the corresponding values of the luminous intensities emitted by the standard matching solutions.

NOTE — The use of this graph leads to only approximate results. It is particularly suitable for carrying out a test intended to serve merely as a guide.

In order to obtain more precise results, it is advisable to proceed by interpolation, enclosing the measurement of the test solution between two measurements carried out on standard matching solutions of only slightly differing potassium content.

6.4 Determination

6.4.1 *Test portion*

Weigh, to the nearest 0,01 g, 10,00 g of the test sample prepared as specified in 6.1.

NOTE — If the homogeneity of the test sample is certain, this mass may be reduced, with a proportional reduction in the volume of the solution and a consequent modification when calculating the result.

6.4.2 *Preparation of the test solution*

Place the test portion (6.4.1) in a 400 ml beaker and add 200 ml of cold water (4.1). Cover the beaker with a watch-glass, heat to boiling and boil gently for 15 min.

Cool to 20 °C, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water (4.1) at 20 °C and mix. This becomes solution A.

Filter, through a dry filter paper (5.1), a sufficient quantity of the solution A, discarding the first portions of the filtrate and collecting the filtered solution in a dry vessel.

Place 10 ml of the filtered solution A in a 1 000 ml one-mark volumetric flask. Add 100 ml of the sulphuric acid solution (4.2), dilute to the mark with water (4.1) and mix. This is the test solution.

6.4.3 *Spectrophotometric measurements*

6.4.3.1 PRELIMINARY MEASUREMENT

Carry out a preliminary measurement on the test solution (6.4.2), following the procedure specified in 6.3.2, at the same time as the spectrophotometric measurements on the standard matching solutions (6.3.1). From these measurements, deduce, using the calibration graph, the approximate potassium content of the test solution.

6.4.3.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution (6.4.2), bracketing it between two standard matching solutions which differ by only 2 mg/l of potassium.

Prepare these solutions by dilution of the standard potassium chloride solution (4.3) without omitting the addition of the sulphuric acid solution (4.2), so that the acidity is of the order of 0,1 N (pH 0,9 to 1,1).

7 EXPRESSION OF RESULTS

7.1 The concentration, C, expressed in milligrams of potassium per litre, of the test solution is given by the formula

$$C = C_1 + (C_2 - C_1)\frac{E_0 - E_1}{E_2 - E_1}$$

where

C_1 is the potassium concentration, in milligrams per litre, of the weaker standard matching solution used for the determination;

C_2 is the potassium concentration, in milligrams per litre, of the stronger standard matching solution used for the determination;

E_0 is the value of the measurement on the test solution;

E_1 is the value of the measurement corresponding to C_1;

E_2 is the value of the measurement corresponding to C_2.

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

$$C \times \frac{1}{1\,000} \times \frac{1\,000}{10} \times \frac{100}{10} = C$$

where C is as defined in 7.1.

NOTE — The test portion and the dilutions are chosen so that the content % (*m/m*) is numerically equal to the concentration, in milligrams per litre, of the test solution.

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

$$C \times \frac{100 - H}{100 - h}$$

where

C is 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).

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

$$C \times 1{,}205 \times \frac{100 - H}{100 - h}$$

where

C is as defined in 7.1;

H and h are as defined in 7.2.

8 REPRODUCIBILITY

The following statistical information is derived from the analysis of the same product, carried out in ten laboratories each time by two operators each effecting two determinations :

- total number of determinations : 40
- arithmetic mean : 61,28 % (*m/m*) as K_2O
- standard deviation : 0,213

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.