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

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FOREWORD

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Potassium sulphate 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 sulphate for industrial use.[1]

2 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 radiation emitted at 766 nm, compared with the intensity obtained for standard potassium chloride solutions acidified in the same way.

3 REAGENTS

3.1 Water. Distilled water, or water of equivalent purity, shall be used in the test.

This water shall, moreover, satisfy the following test :

The spectrophotometer having been adjusted so that a zero reading (minimum of the scale) is obtained for water (3.1) and about 100 (maximum of the scale) for a standard solution containing 10 mg/l of potassium, the reading obtained for the water, evaporated in a sodium-free vessel (platinum, silica, etc.) until it is reduced to one fiftieth of of its volume shall not exceed 10.

3.2 Sulphuric acid, approximately N solution, practically free from potassium. This solution, diluted ten times, should not give a spectrophotometer reading of more than 2, under the conditions stated in 3.1.

3.3 Standard potassium chloride solution, corresponding to 4,000 g of potassium per litre.

Weigh, to the nearest 0,001 g, 7,627 g of potassium chloride (KCl) previously dried at 400 °C and cooled in a desiccator. Dissolve it in the water (3.1), dilute to 1 000 ml in a one-mark volumetric flask and mix.

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

4 APPARATUS

Ordinary laboratory apparatus and

4.1 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.

4.2 Flame spectrophotometer, fitted with an atomizer-burner constructed so as to excite emission of the potassium line at 766 nm.

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

The sensitivity of the apparatus should 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 should be reproducible and stable 20 to 40 s after the start of atomization.

5 PROCEDURE

5.1 Preparation of test sample

Grind the laboratory sample until it passes completely through a 500 μm nominal mesh sieve.[2]

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 should also be determined.

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1) See also the same determination in
- ISO 2485, *Gravimetric method as potassium tetraphenylborate.*
- ISO 2486, *Sodium tetraphenylborate volumetric method.*

2) See Table 1, ISO/R 565, *Woven wire cloth and perforated plates in test sieves — Nominal sizes of apertures.*

5.2 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 2850.[1]

5.3 Preparation of the calibration curve

5.3.1 *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 (3.2), place the volumes of the standard potassium solution (3.3) shown in the following table :

Potassium standard solution (3.3)	Corresponding mass of potassium
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 (3.1) and mix.

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

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

In addition, the intensity of the potassium radiations 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 under the conditions of this determination. The influence of the pH is zero 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.

5.3.2 *Spectrophotometric measurements*

Switch on the spectrophotometer (4.2) 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 and to ensure a band pass of not more than 12 nm, centred on the emission maximum (theoretical value 766 nm).

Atomize the standard matching solutions (5.3.1) in succession in the centre of the flame and measure the intensity of the radiation 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 measurements.

5.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, the potassium contents as abscissae 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 curve 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, bracketing the measurement of the sample solution between two measurements carried out on standard matching solutions of sufficiently similar potassium content.

5.4 Determination

5.4.1 *Test portion*

Weigh, to the nearest 0,01 g, 10,00 g of the test sample prepared as described in 5.1.

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.

5.4.2 *Preparation of the test solution*

Place the test portion (5.4.1) in a 400 ml beaker and add 200 ml of cold water (3.1). 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 (3.1) at 20 °C and mix. This is solution A.

Filter, through a dry filter paper (4.1), a sufficient quantity of the solution A, discarding the first portions of the filtrate and collecting the remainder in a dry vessel.

Place 10,0 ml of the filtered solution A in a 1 000 ml one-mark volumetric flask. Add 100 ml of the sulphuric acid solution (3.2), dilute to the mark with water (3.1) and mix. This is the test solution.

5.4.3 *Spectrophotometric measurements*

5.4.3.1 PRELIMINARY MEASUREMENT

Carry out a preliminary measurement on the test solution (5.4.2), according to the procedure specified in 5.3.2, at the same time as the spectrophotometric measurements on the standard matching solutions (5.3.1). From these measurements deduce, using the calibration curve, the approximate potassium content of the test solution.

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[1] See ISO 2850, *Potassium sulphate for industrial use — Determination of loss of mass at 105 °C.* (At present at the stage of draft.)

5.4.3.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution (5.4.2), placing it between two standard matching solutions which differ by only 2 mg/l of potassium.

Prepare these solutions by dilution of the potassium standard solution (3.3) without omitting the addition of the sulphuric acid solution (3.2), so that the acidity is of the order of 0,1 N (pH 0,9 to 1,1).

6 EXPRESSION OF RESULTS

6.1 Method of calculation and formulae

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

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

where

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

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

E is the value of the measurement corresponding to 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.

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

$$C \times \frac{1}{1000} \times \frac{1000}{10} \times \frac{100}{10} = C$$

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

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

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

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).

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

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

where H and h have the same meaning as in 6.1.3.

6.2 Repeatability and reproducibility

The statistical information given below was obtained from analyses carried out in eleven laboratories, each time by two operators, each carrying out two determinations.

		Sample			Global value
		A	B	C	
Mean (% K_2O)		47,9	50,6	53,2	–
Standard deviation for	repeatability (σ_r)	0,13	0,15	0,16	0,15
	reproducibility (σ_R)	0,56	0,32	0,48	0,47

7 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 in the International Standards to which reference is made, or regarded as optional.