INTERNATIONAL STANDARD ISO 2485

G-92-28

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Potassium sulphate for industrial use — Determination of potassium content — Gravimetric method as potassium tetraphenylborate

First edition — 1973-04-01

UDC 661.832.53 : 546.32 : 543.21

Ref. No. ISO 2485-1973 (E)

Descriptors : potassium sulphates, chemical analysis, determination of content, potassium, gravimetric analysis.

Price based on 3 pages

FOREWORD

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International Standard ISO 2485 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in August 1971.

It has been approved by the Member Bodies of the following countries :

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No Member Body expressed disapproval of the document.

Printed in Switzerland

INTERNATIONAL STANDARD

ISO 2485-1973 (E)

Potassium sulphate for industrial use — Determination of potassium content — Gravimetric method as potassium tetraphenylborate

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric 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.

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 excessive errors.

NOTE — These additions, which make the method of general application, have no detrimental effect on its precision or reproducibility.

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

Drying and weighing of the precipitate.

3 REAGENTS

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

3.1 Sodium hydroxide, approximately 400 g/l solution.

3.2 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 1 000 ml.

3.3 Sodium tetraphenylborate, alkaline solution.

Dissolve 32,5 g of sodium tetraphenylborate in 480 ml of water. Add 2 ml of sodium hydroxide solution (3.1) and 20 ml of magnesium chloride ($MgCl_2.6H_2O$) 100 g/l solution. Stir for 15 min and filter through a fine texture filter.

Replace this reagent each week and filter immediately before use.

3.4 Wash solution (saturated solution of potassium tetraphenylborate).

Precipitate 0,5 g of potassium tetraphenylborate in a solution of pure potassium chloride. Filter the precipitate and wash it with water. Bring the precipitate back into suspension in 5 l of water and shake for about 1 h. Immediately before use, filter the quantity of reagent required for the determination.

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

Filter immediately before use.

3.6 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Filter crucible, glass or porcelain, of porosity grade P10 or P16 (i.e. maximum pore size between 4 and 16 μm).

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[1] See also the same determination in

- ISO 2484, *Flame emission spectrophotometric method.*
- ISO 2486, *Sodium tetraphenylborate volumetric method.*

5 PROCEDURE

5.1 Preparation of the test sample

Grind the laboratory sample until it passes completely through a 500 µm nominal mesh sieve.[1]

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.

5.2 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 2850.[2]

5.3 Test portion

Weigh, to the nearest 0,001 g, 10 g of the test sample, prepared as described in 5.1.

5.4 Preparation of the test solution

Place the test portion (5.3) in a 400 ml beaker and add approximately 200 ml of cold water. Cover the beaker with a watch glass, heat and keep boiling gently for 15 min. Cool to 20 °C, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water at 20 °C and mix. This is solution A.

Filter a sufficient quantity of the solution A through a dry filter paper, discarding the first portions of the filtrate and retaining the remainder in a dry receiver.

5.5 Determination

Place 50,0 ml of the filtered solution A in a 500 ml one-mark volumetric flask and dilute to the mark. Take 50,0 ml of this solution and place it in a 250 ml beaker.

Add 10 ml of the EDTA solution (3.2), a few drops of the phenolphthalein solution (3.6) and, drop by drop, the sodium hydroxide solution (3.1) until there is a definite red colour. Then add 10 ml of the formaldehyde solution (3.5) and a few drops of the sodium hydroxide solution (3.1) until the definite red colour reappears. Cover the beaker with a watch glass and heat for 15 min on a boiling water bath. The solution should remain red, otherwise add a few drops of the phenolphthalein solution (3.6) and, if necessary, restore the colour by adding, drop by drop, the sodium hydroxide solution (3.1).

Add, drop by drop, while stirring, 10 ml of the sodium tetraphenylborate solution (3.3). Continue the stirring for about 1 min then cool rapidly to below 20 °C under a stream of water. Allow to stand for 10 min, then filter through the crucible (4.1) previously heated at 120 °C, cooled in a desiccator and weighed to the nearest 1 mg. Rinse the precipitate with the wash solution (3.4) and wash successively with the same solution.

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

6 EXPRESSION OF RESULTS

6.1 Method of calculation and formulae

6.1.1 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

$$10\,908 \times \frac{m_1}{5\,m_0} = 2\,181{,}6 \times \frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the dried precipitate.

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

$$10\,908 \times \frac{m_1}{5\,m_0} \times \frac{100-H}{100-h} = 2\,181{,}6 \times \frac{m_1}{m_0} \times \frac{100-H}{100-h}$$

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);

m_0 and m_1 have the same meaning as in 6.1.1.

6.1.3 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

$$13\,140 \times \frac{m_1}{5\,m_0} \times \frac{100-H}{100-h} = 2\,628 \times \frac{m_1}{m_0} \times \frac{100-H}{100-h}$$

where the symbols have the same meaning as in 6.1.1 and 6.1.2.

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

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

6.2 Repeatability and reproducibility

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

		Sample			Global value
		A	B	C	
Mean (% K_2O)		48,0	50,5	53,2	
Standard deviation for	repeatability (σ_r)	0,14	0,11	0,13	0,13
	reproducibility (σ_R)	0,43	0,15	0,30	0,29

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 regarded as optional.