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Potassium sulphate for industrial use — Determination of potassium content — Sodium tetraphenylborate volumetric method

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FOREWORD

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Potassium sulphate for industrial use — Determination of potassium content — Sodium tetraphenylborate volumetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric 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 ammonium oxalate to precipitate the calcium ions and of formaldehyde to convert the ammonium ions present into hexamethylenetetramine.

Precipitation of the potassium in an alkaline medium with an excess of a standard volumetric sodium tetraphenylborate (STPB) solution. Filtration followed by titration of this excess with a standard volumetric solution of a quaternary ammonium salt (cetyltrimethylammonium bromide, CTAB) in the presence of thiazol yellow as indicator.

3 REAGENTS

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

3.1 Aluminium hydroxide, high purity.

3.2 Sodium hydroxide, approximately 200 g/l solution.

3.3 Formaldehyde, 35 % (*m/m*) solution.

It is essential that this reagent should be filtered before use.

3.4 Ammonium oxalate, solution saturated at 20 °C (approximately 60 g/l).

3.5 Quaternary ammonium salt (CTAB), approximately 0,02 N solution.

Dissolve 7,3 g of cetyltrimethylammonium bromide (CTAB), $[CH_3(CH_2)_{15}N(CH_3)_3]Br$, in 300 to 500 ml of water. Stir gently and dilute to the mark with water in a 1 000 ml one-mark volumetric flask. Determine the correlation between the STPB and CTAB solutions by titration in the presence of thiazol yellow (3.8) as indicator.

This solution remains stable for over a month.

3.6 Sodium tetraphenylborate (STPB), approximately 0,02 N solution.

Dissolve 6,90 g of STPB $[NaB(C_6H_5)_4]$ in 500 ml of water in a 600 ml beaker. Add 6 g of aluminium hydroxide (3.1) and stir for 5 to 10 min with a magnetic stirrer.

After decanting, filter the solution through a close-textured filter paper into a 1 000 ml one-mark volumetric flask. If the filtrate is turbid, filter it again. Wash the filter paper twice with about 30 ml of water, add 1 ml of the sodium hydroxide solution (3.2), dilute to the mark and mix. The STPB solution should be allowed to age for 48 h. Filter and determine the normality of this solution, before use, by means of the potassium chloride standard reference solution (3.7).

3.6.1 *Standardization of the STPB solution*

Transfer 25,00 ml of the standard reference potassium chloride solution (3.7) to a 100 ml one-mark volumetric flask. Add, in the following order :

- 5 ml of the formaldehyde solution (3.3);
- 2 ml of the sodium hydroxide solution (3.2);
- 1,5 ml of the ammonium oxalate solution (3.4);
- 40,00 ml of the STPB solution (3.6).

Dilute to the mark with water. Swirl and allow to stand for 5 min. Filter the solution through a close-textured, dry, folded filter paper, discarding the first few millilitres of the filtrate and collecting the remainder in a dry vessel. Transfer 50,0 ml of this solution to a 300 ml conical flask. Add 5 ml of the thiazol yellow solution (3.8) and titrate immediately with the CTAB solution (3.5) until the colour changes to a definite pink colour. Let V_2 be the volume used.

1) See also the same determination in

- ISO 2484, *Flame emission spectrophotometric method.*
- ISO 2485, *Gravimetric method as potassium tetraphenylborate.*

In the alternative case of bromophenol blue solution (3.9) being used as indicator, add 10 drops of this solution and the volume of hydrochloric acid solution (3.10) necessary to change the colour of the indicator to a definite yellow colour. Proceed immediately to titrate the solution drop by drop with the CTAB solution (3.5) until the colour of the indicator changes from yellow to green. Again V_2 is the volume used.

3.6.2 *Correlation between the STPB and CTAB solutions*

Pour about 20 ml of water into a 100 ml one-mark volumetric flask and add in the following order :

- 5 ml of the formaldehyde solution (3.3);
- 2 ml of the sodium hydroxide solution (3.2);
- 1,5 ml of the ammonium oxalate solution (3.4).

Stir and add 40,00 ml of the STPB solution (3.6). Dilute to the mark with water, stir and allow to stand for 5 min. Filter the solution through a close-textured, dry, folded filter paper, discarding the first few millilitres of the filtrate and collecting the remainder in a dry vessel.

Transfer 10,0 ml of this solution to a 300 ml conical flask and add, in the following order :

- 40 ml of water;
- 5 ml of the thiazol yellow solution (3.8).

and titrate immediately with the CTAB solution (3.5) until the colour changes to a definite pink colour. Let V_1 be the volume used.

In the alternative case of bromophenol blue solution (3.9) being used as indicator, add 10 drops of this solution and the volume of hydrochloric acid solution (3.10) necessary to change the colour of the indicator to a definite yellow colour. Proceed immediately to titrate the solution drop by drop with the CTAB solution (3.5) until the colour of the indicator changes from yellow to green. Again V_1 is the volume used.

3.6.3 *Calculation of the strengths of the CTAB and STPB solutions*

The strengths (T_1 and T_2) of the solutions of CTAB and STPB expressed in milligrams of potassium oxide (K_2O) per millilitre are given by the formulae

$$T_1 = \frac{40}{10\,V_1} \times T_2 = \frac{25}{10\,V_1 - 2\,V_2}$$

$$T_2 = \frac{25 + 2\,V_2\,T_1}{40} = \frac{V_1}{4} \times \frac{25}{10\,V_1 - 2\,V_2}$$

where

V_1 is the volume, in millilitres, of the CTAB solution (3.5) used to establish the correlation between the solutions of CTAB (3.5) and STPB (3.6);

V_2 is the volume, in millilitres, of the CTAB solution (3.5) used to standardize the STPB solution (3.6).

3.7 Potassium chloride, standard reference solution containing 1,00 g/l of K_2O.

Weigh, to the nearest 0,000 1 g, 3,166 g of potassium chloride, previously dried at 400 °C and cooled in a desiccator. Dissolve it in water and transfer the solution quantitatively to a 2 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this solution corresponds to 1,0 mg of K_2O.

3.8 Thiazol yellow, 0,020 g/l solution.

Dissolve 0,020 g of thiazol yellow (also known as titan yellow or Clayton yellow) in 10 ml of water. Dilute to the mark in a 1 000 ml one-mark volumetric flask with pure rectified acetone and mix.

Do not keep this reagent for more than 2 weeks.

Alternatively

3.9 Bromophenol blue, 0,4 g/l solution.

In a 100 ml one-mark volumetric flask, dissolve 0,040 g of bromophenol blue in about 3 ml of approximately 0,01 M sodium hydroxide solution, dilute to the mark with water and mix.

3.10 Hydrochloric acid, approximately 0,1 N solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Automatic burettes, of 50 ml capacity, with a 1 000 ml reagent bottle (for the STPB solution) (3.6), and of 10 ml capacity graduated in 0,02 ml, with a 2 000 ml reagent bottle (for the CTAB solution) (3.5).

4.2 Burette with stopcock, 25 ml capacity, graduated in 0,05 ml, for the potassium chloride standard reference solution (3.7).

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.

1) See Table 1 of 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 Test portion

Weigh, to the nearest 0,001 g, 2,50 g of the test sample, prepared as described in 5.1.

5.4 Preparation of the test solution

Transfer the test portion (5.3) to a 500 ml one-mark volumetric flask containing about 300 ml of water.

Dissolve it, with stirring (intermittent stirring over a period of 30 min is generally sufficient). Dilute to the mark, stir and either filter through a close-textured dry, folded filter paper, or decant the solution.

5.5 Determination

Transfer 10,0 ml of the test solution (5.4) to a 100 ml one-mark volumetric flask and add, in the following order :

- 40 ml of water;
- 5 ml of the formaldehyde solution (3.3);
- 2 ml of the sodium hydroxide solution (3.2);
- 1,5 ml of the ammonium oxalate solution (3.4);
- 40,00 ml of the STPB solution (3.6).

Do not stir during addition of the STPB solution as this causes foam to form, which prevents accuracy in diluting to the mark.

Dilute to the mark with water, stir, allow to stand for 5 to 10 min and filter through a close-textured, dry, folded filter paper, discarding the first few millilitres of the filtrate and collecting the remainder in a dry vessel.

Transfer 50,0 ml of this solution to a 300 ml conical flask. Add 5 ml of the thiazol yellow solution (3.8) and titrate immediately with the CTAB solution (3.5) until the colour changes to a definite pink colour. Let V be the volume used.

In the alternative case of bromophenol blue solution (3.9) being used as indicator, add 10 drops of this solution and the volume of hydrochloric acid solution (3.10) necessary to change the colour of the indicator to a definite yellow colour. Proceed immediately to titrate the solution drop by drop with the CTAB solution (3.5) until the colour of the indicator changes from yellow to green. Again V is the volume used.

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 oxide (K_2O), is given, as a percentage by mass, by the formula

$$\frac{40\,T_2 - 2\,V\,T_1}{1\,000} \times \frac{500}{10} \times \frac{100}{m} = \frac{5\,(40\,T_2 - 2\,V\,T_1)}{m}$$

where

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

V is the volume, in millilitres, of the CTAB solution (3.5) used for the determination;

T_1 is the volume of the CTAB solution (3.5), expressed in milligrams of potassium oxide per millilitre;

T_2 is the volume of the STPB solution (3.6), expressed in milligrams of potassium oxide per millilitre.

6.1.2 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

$$\frac{5\,(40\,T_2 - 2\,V\,T_1)}{m} \times \frac{100 - H}{100 - h}$$

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).

V, T_1 and T_2 have the same meaning as in 6.1.1.

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	50,6	53,0	–
Standard deviation for	repeatability (σ_r)	0,13	0,08	0,13	0,12
	reproducibility (σ_R)	0,21	0,19	0,36	0,27

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

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.