



BS 5551 : Subsection 4.3.3 : 1984 ISO 5318-1983

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British Standard

Fertilizers

Part 4. Chemical analysis

Section 4.3 Determination of potassium

Subsection 4.3.3 Potassium tetraphenylborate gravimetric method

[ISO title: Fertilizers — Determination of potassium content —

Potassium tetraphenylborate gravimetric method (Reference method)]

Engrais

Partie 4. Analyse chimique

Section 4.3 Dosage du potassium

Sous-section 4.3.3 Méthode gravimétrique à l'état de tétraphénylborate de potassium

Düngemittel

Teil 4. Chemische Analyse

Abschnitt 4.3 Bestimmung des Kaliumgehalts

Unterabschnitt 4.3.3 Gravimetrisches KTPB-Verfahren

National foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee in order to standardize terminology and labelling, sampling and methods of physical and chemical testing for fertilizers.

For some years the United Kingdom has participated in the standardization of methods of analysing fertilizers through Subcommittee 4, Chemical analysis, of Technical Committee 134, Fertilizers and soil conditioners, of the International Organization for Standardization (ISO). As international agreement is reached on the methods, it is proposed to publish them as Sections of BS 5551.

The standard is to be published in four Parts, each Part being subdivided into Sections and, where appropriate, Subsections. The four Parts are:

- Part 1 Terminology and labelling
- Part 2 Sampling
- Part 3 Physical properties
- Part 4 Chemical analysis

Part 4 is to be divided into five Sections as follows.

- Section 4.1 Determination of nitrogen
- Section 4.2 Determination of phosphorus
- Section 4.3 Determination of potassium
- Section 4.4 Determination of water
- Section 4.5 Determination of other constituents

This Subsection of Part 4 is identical with ISO 5318-1983 'Fertilizers — Determination of potassium content — Potassium tetraphenylborate gravimetric method (Reference method)'.

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Cross-reference

International standard	Corresponding British Standard
ISO 5317-1983	BS 5551 Fertilizers Subsection 4.3.1 : 1983 Preparation of the test solution for determination of water-soluble potassium content (Identical)

Additional information. (1) This British Standard describes a method of test only and should not be used as a specification defining limits of purity. Reference to the standard should indicate that the method of test used is in accordance with BS 5551 : Subsection 4.3.3.

(2) With reference to clause 4, water complying with BS 3978 'Water for laboratory use' is suitable.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the potassium content of test solutions of fertilizers. It is suitable for use in arbitration and for reference purposes.

2 Reference

ISO 5317, *Fertilizers — Determination of water-soluble potassium content — Preparation of the test solution.*

3 Principle

Preparation of a test solution as specified in ISO 5317.

Precipitation of potassium ions present in an aliquot portion of the test solution (previously treated with bromine water and activated charcoal if cyanamide and/or organic materials are present) by sodium tetraphenylborate in a weakly alkaline medium in the presence of disodium ethylenediaminetetraacetate dihydrate (EDTA, disodium salt) and formaldehyde to eliminate interference by ammonium ions.

Filtration of the precipitate, drying and weighing.

4 Reagents

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

4.1 Sodium tetraphenylborate, approximately 15 g/l solution.

Dissolve 7,5 g of sodium tetraphenylborate $[\text{NaB}(\text{C}_6\text{H}_5)_4]$ in 480 ml of water. Add 2 ml of the sodium hydroxide solution (4.5) and 20 ml of a 100 g/l solution of magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$). Stir for 15 min and filter through the filter paper (5.2).

This solution may be stored in a plastics bottle for not longer than 1 month. Filter immediately before use.

4.2 Sodium tetraphenylborate, wash solution.

Dilute 1 volume of the sodium tetraphenylborate solution (4.1) with 10 volumes of water.

4.3 EDTA, disodium salt, 40 g/l solution.

Dissolve 4,0 g of disodium ethylenediaminetetraacetate dihydrate (EDTA, disodium salt) in 100 ml of water.

4.4 Formaldehyde, 30 % (m/m) solution.

If necessary, filter before use.

4.5 Sodium hydroxide, 400 g/l solution.

4.6 Phenolphthalein, 5 g/l ethanolic solution.

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

4.7 Bromine water, saturated solution.

4.8 Charcoal, activated, which does not adsorb or liberate potassium ions.

5 Apparatus

Usual laboratory equipment, and

5.1 Filter crucibles, having a sintered glass or porcelain disc of porosity grade P 10 or P 16 (pore size index 4 to 16 μm).

5.2 Fine textured filter paper.

5.3 Drying oven, capable of being controlled at $120 \pm 5^\circ\text{C}$.

6 Test solution

Use the clear solution obtained as specified in ISO 5317.

NOTE — In general, a test portion of about 5 g will be required and the volume of the resulting solution will generally be 1 000 ml.

7 Procedure

7.1 Aliquot portion of test solution

7.1.1 Procedure in the presence of cyanamide and/or organic materials

Transfer, by means of a pipette, an aliquot portion, V_1 , of the test solution (clause 6), containing 50 to 100 mg of potassium oxide (K_2O), and preferably about 80 mg, to a 250 ml beaker. Add 5 ml of the bromine water (4.7) and boil the solution until all the bromine has been removed and then, if necessary, to reduce the volume to less than 100 ml. Allow the solution to cool to ambient temperature and transfer it to a 100 ml one-mark volumetric flask. Add about 0,5 g of the activated charcoal (4.8) and shake vigorously. Dilute to the mark and mix well.

Filter the solution and, using a pipette, transfer 50 ml of the filtrate into a 250 ml beaker.

7.1.2 Procedure in the absence of cyanamide and/or organic materials

Transfer, by means of a pipette, an aliquot portion, V_1 , of the test solution (clause 6) containing 25 to 50 mg of potassium oxide (K_2O), and preferably about 40 mg, to a 250 ml beaker and adjust the volume to 50 ml by dilution or evaporation.

7.2 Determination

Further treat the aliquot portion of the test solution (7.1) as follows.

Add 20 ml of the EDTA solution (4.3) and a few drops of the phenolphthalein solution (4.6). Add, drop by drop, the sodium hydroxide solution (4.5) until a red colour appears and then add 1 ml of the sodium hydroxide solution in excess.

NOTE — Too high a concentration of sodium hydroxide in the solution during boiling or heating on the steam bath may cause precipitation of iron(III) hydroxide and co-precipitation of sodium tetraphenylborate.

Boil for 15 min.

Add 10 ml of the formaldehyde solution (4.4) and, if necessary, a few drops of the sodium hydroxide solution (4.5) until the red colour persists. Cover the beaker with a watch-glass and heat for 15 min on a steam bath.

The solution should remain red. If it does not, add a few drops of the phenolphthalein solution (4.6) and, if necessary, restore the red colour by adding, drop by drop, the sodium hydroxide solution (4.5).

Remove the beaker from the steam bath and immediately add, drop by drop, while stirring, 40 ml of the sodium tetraphenylborate solution (4.1).

Continue stirring for 1 min, then cool rapidly to below 20 °C in running water and allow to stand for 10 min.

Weigh the filter crucible (5.1), previously dried in the oven (5.3), controlled at 120 ± 5 °C, and cooled in a desiccator, to the nearest 0,000 1 g.

Decant the supernatant liquid through the crucible. Wash the precipitate in the beaker with 40 ml of the wash solution (4.2) and decant again. Repeat this procedure. Transfer the precipitate to the crucible, rinsing the beaker with about 40 ml of the wash solution (4.2), followed by 5 ml of water.

Dry the crucible and precipitate in the oven (5.3), controlled at 120 ± 5 °C, for 90 min, allow to cool in a desiccator and weigh to the nearest 0,000 1 g.

7.3 Blank test

Carry out a blank test at the same time as the determination.

8 Expression of results

8.1 Calculation

The potassium content, expressed as a percentage by mass as potassium (K) or as potassium oxide (K_2O), is given by the formula

- a) if cyanamide and/or organic materials are present

$$\frac{[(m_2 - m_1) - (m_4 - m_3)] \times f \times V_0 \times 200}{m_0 \times V_1}$$

- b) if cyanamide and/or organic materials are absent

$$\frac{[(m_2 - m_1) - (m_4 - m_3)] \times f \times V_0 \times 100}{m_0 \times V_1}$$

where

m_0 is the mass, in grams, of the test portion¹⁾;

m_1 is the mass, in grams, of the filter crucible;

m_2 is the mass, in grams, of the crucible and precipitate;

m_3 is the mass, in grams, of the crucible used for the blank test;

m_4 is the mass, in grams, of the crucible used for the blank test and the corresponding precipitate;

V_0 is the volume, in millilitres, of the test solution (volume in which the test portion was dissolved)¹⁾;

V_1 is the volume, in millilitres, of the aliquot portion of the test solution taken for the determination;

f is a factor which, if the potassium content is expressed as potassium (K), is equal to 0,109 1 or, if the potassium content is expressed as potassium oxide (K_2O), is equal to 0,131 4.

1) See ISO 5317.