International Standard 5318

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Fertilizers — Determination of potassium content — Potassium tetraphenylborate gravimetric method (Reference method)

Engrais — Dosage du potassium — Méthode gravimétrique à l'état de tétraphénylborate de potassium (Méthode de référence)

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Foreword

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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.

International Standard ISO 5318 was developed by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, and was circulated to the member bodies in May 1982.

It has been approved by the member bodies of the following countries :

Austria	Iraq	Portugal
China	Israel	Romania
Czechoslovakia	Italy	South Africa, Rep. of
Egypt, Arab Rep. of	Kenya	Sri Lanka
France	Korea, Rep. of	United Kingdom
Germany, F. R.	Netherlands	USA
Hungary	Norway	USSR
India	Poland	

No member body expressed disapproval of the document.

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Fertilizers — Determination of potassium content — Potassium tetraphenylborate gravimetric method (Reference method)

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 [$NaB(C_6H_5)_4$] 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 ($MgCl_2.6H_2O$). 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 ± 5 °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 portion[1];

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.

8.2 Precision

The statistical information given below was obtained from the analysis of 108 sets of results, from 18 laboratories in 6 different countries.

8.2.1 Repeatability, *r*

Successive results obtained using this method on identical test material, under the same conditions (same operator, same laboratory, same apparatus and same time), shall be considered suspect if they differ by more than the values given in table 1.

Table 1

Potassium content	*r*
% (*m*/*m*) as K_2O	% (*m*/*m*) as K_2O
< 20	0,12
20 to 60	0,39

8.2.2 Reproducibility, *R*

Individual results obtained using this method on identical test material, but under different conditions (different operators, different laboratories, different apparatus and/or different times), shall be considered suspect if they differ by more than the values given in table 2.

Table 2

Potassium content	*R*
% (*m*/*m*) as K_2O	% (*m*/*m*) as K_2O
< 20	0,24
20 to 60	0,73

9 Test report

The test report shall include the following information :

a) all information necessary for the complete identification of the sample;

b) the reference of the method used, i.e. ISO 5318;

c) the result and the method of expression used;

d) the test conditions;

e) any operations not specified in this International Standard or in the International Standard to which reference is made, or regarded as optional, as well as any incidents likely to have affected the results.

Bibliography

[1] Association of Official Analytical Chemists Methods, 11th edition (1970), Nos. 2.706 to 2.080.

[2] Benelux analytical method K-01.

[3] ISO 2051, *Potassium chloride for industrial use — Determination of potassium content — Potassium tetraphenylborate gravimetric method.*

[4] ISO 2485, *Potassium sulphate for industrial use — Determination of potassium content — Gravimetric method as potassium tetraphenylborate.*

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