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International Standard ISO 5310

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Fertilizers — Determination of potassium content — Titrimetric method

Engrais — Dosage du potassium — Méthode titrimétrique

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 5310 was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Fertilizers — Determination of potassium content — Titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the potassium content of fertilizers.

2 References

ISO 384, *Laboratory glassware — Principles of design and construction of volumetric glassware.*

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

ISO 7407, *Fertilizers — Determination of acid-soluble potassium content — Preparation of the test solution.*

3 Principle

Preparation of a test solution as specified in ISO 5317 or ISO 7407.

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 an excess of sodium tetraphenylborate (NaTPB) in a weakly alkaline medium. Filtration and determination of the unused NaTPB by back titration with cetyltrimethylammonium bromide solution, using thiazol yellow as indicator.

NOTE — It is essential that the conditions for the precipitation and back titration correspond exactly with those for standardization of the volumetric solutions.

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 (NaTPB), [$NaB(C_6H_5)_4$], standard volumetric solution, $c(\mathrm{NaTPB}) \approx 0{,}02$ mol/l.

Dissolve 6,90 g of NaTPB in about 800 ml of water in a 1 000 ml beaker, stirring mechanically for 10 min. Add 5 ml of the sodium hydroxide solution (4.3) and 10 ml of the magnesium chloride solution (4.2). Stir for 10 min, allow to stand for a few minutes, then filter the solution and collect the filtrate in a 1 000 ml one-mark volumetric flask. Rinse the beaker and wash the precipitate, filtering the combined washings and collecting them in the same flask. Dilute to the mark and mix thoroughly.

Standardize the solution by the method described in 7.2.3.3. The concentration of the solution shall be in the range 0,019 to 0,021 mol/l.

Filter the solution immediately before use; discard it after 2 weeks.

4.2 Magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$), 100 g/l solution.

4.3 Sodium hydroxide solution, $c(\mathrm{NaOH}) \approx 2$ mol/l.

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

If necessary, filter and/or neutralize before use.

4.5 Reagents for back titration

4.5.1 Cetyltrimethylammonium bromide (CTAB), [$CH_3(CH_2)_{15}N(CH_3)_3Br$], standard volumetric solution, $c(\mathrm{CTAB}) \approx 0{,}01$ mol/l.

Dissolve 3,65 g of CTAB in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix, stirring gently to avoid the formation of foam.

4.5.2 Thiazol yellow[1], 0,020 g/l solution.

Dissolve 0,020 g of thiazol yellow in 10 ml of water in a 1 000 ml one-mark volumetric flask, dilute to the mark with acetone and stir.

Discard the solution after 2 weeks.

4.6 Potassium chloride solution, $c(\mathrm{KCl}) = 0{,}02$ mol/l.

Dissolve in water, in a 1 000 ml one-mark volumetric flask, 1,491 g of potassium chloride, which has been previously dried for 2 h in an oven controlled at 105 to 110 °C and then allowed to cool to ambient temperature in a desiccator. Dilute to the mark and mix thoroughly.

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[1] Also known as Titan yellow or Clayton yellow.

4.7 Phenolphthalein, 5 g/l ethanolic solution.

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

4.8 Bromine water, saturated solution.

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

5 Apparatus

Usual laboratory equipment, including graduated glassware complying with the requirements of ISO 384, class A, and:

5.1 Filter paper, fine-textured.

5.2 Microburette, of capacity 10 ml, graduated in 0,02 ml divisions or smaller.

5.3 pH meter.

6 Test solution

Prepare the test solution (solution A) as specified in ISO 5317 or ISO 7407.

For potassium contents, expressed as K_2O, in the range 10 to 60 % (*m*/*m*), prepare test solution B by diluting solution A with water as indicated in column 4 of table 1.

7 Procedure

7.1 Aliquot portion of test solution

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

Depending on the expected potassium content of the sample, by means of a pipette, transfer an aliquot portion of twice the volume of solution A or solution B specified in column 5 of table 1, into a 250 ml beaker. Add 5 ml of bromine water (4.8) and boil the solution until all the bromine has been removed and continue boiling, 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. Dilute to the mark and mix well. Add about 0,5 g of activated charcoal (4.9) and shake vigorously.

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

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

Depending on the expected potassium content of the sample, transfer an aliquot portion of the volume of solution A or solution B specified in column 5 of table 1, into a 250 ml beaker by means of a pipette.

7.2 Determination

7.2.1 Preparation of the solution

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

Add, drop by drop, sodium hydroxide solution (4.3) until a pH of 8,2 is obtained, measured by means of a pH meter. Remove the electrodes and rinse them with water into the solution. Add a further 5,0 ml of sodium hydroxide solution and boil for 5 min to remove the ammonia. Add 5 ml of formaldehyde solution (4.4) and allow to cool to ambient temperature.

Check whether the pH is 11,2. If lower, add a few more drops of sodium hydroxide solution. Then add a further 1,0 ml of sodium hydroxide solution.

Use this solution for the determination.

7.2.2 Precipitation and filtration

Calculate the volume of the NaTPB solution (4.1) required for precipitation as follows. Multiply the expected potassium content, expressed as K_2O in % (*m*/*m*), of the fertilizer by the appropriate volume from column 6 of table 1. Round up the calculated volume to the next whole number. This is the volume, in millilitres, required (V_3).

Table 1 — Test solution data

1	2	3	4		5		6	7
Expected potassium content as K_2O	Approximate mass of test portion, *m*	Volume of test solution (Solution A)	Preparation of solution B		Volume used for the determination		Volume of NaTPB solution per 1 % of K_2O	Dilution factor, *f*
			Volume of solution A taken	Final volume of solution B	Solution A	Solution B		
% (*m*/*m*)	g	ml	ml	ml	ml	ml	ml	
5 to 6	5	1 000	—	—	50,00	—	4,00	20
6 to 10	5	1 000	—	—	50,00	—	3,50	20
10 to 20	5	1 000	50,00	100,00	—	50,00	1,75	40
20 to 40	5	1 000	25,00	100,00	—	50,00	0,90	80
40 to 60	5	1 000	25,00	200,00	—	50,00	0,45	160

Examples:

a) If the expected potassium content, expressed as K_2O, is 7 % (m/m), the calculated volume of NaTPB solution is $(3,5 \times 7)$ ml = 24,5 ml; the required volume is therefore 25,00 ml.

b) If the expected potassium content, expressed as K_2O, is 36 % (m/m), the calculated volume of NaTPB solution is $(0,9 \times 36)$ ml = 32,4 ml; the required volume is therefore 33,00 ml.

Using a burette, add the required volume of NaTPB solution (4.1) to the prepared test solution (7.2.1). Bring the solution to the boil, allow it to cool to ambient temperature and transfer it quantitatively to a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix thoroughly. Filter the solution through a dry filter paper (5.1), discarding the first part of the filtrate and collecting the remainder in a dry vessel.

7.2.3 Back titration

7.2.3.1 Introduction

Before carrying out the back titration (see 7.2.3.5), determine the equivalence of the NaTPB and CTAB solutions (see 7.2.3.2) to allow the calculation, by difference, of the volume of NaTPB solution which has reacted with the potassium, and then standardize the NaTPB solution (see 7.2.3.3).

NOTE — It is essential to check the equivalence of the two solutions prior to restandardizing the NaTPB solution.

7.2.3.2 Determination of equivalence of NaTPB and CTAB solutions.

Transfer about 20 ml of water into a 100 ml one-mark volumetric flask and add, in the following order, 5 ml of formaldehyde solution (4.4) and 5 ml of sodium hydroxide solution (4.3). Swirl and by means of a pipette, transfer 25,0 ml of NaTPB solution (4.1) into the flask. Dilute to the mark with water, mix well and allow to stand for 5 min. Filter the solution through a dry filter paper (5.1) discarding the first few millilitres of the filtrate and collecting the remainder in a dry vessel.

By means of a pipette, transfer 5,0 ml of the clear filtrate into a 250 ml conical flask and add, in the following order, 40 ml of water and 5 ml of thiazol yellow solution (4.5.2). The pH shall be 11 to 12. Titrate with CTAB solution (4.5.1) until the colour of the solution changes to a distinct pink. Let V_1 be the volume used.

7.2.3.3 Standardization of the NaTPB solution

Pipette 20,0 ml of potassium chloride solution (4.6) into a 100 ml one-mark volumetric flask. Then add, in the following order, 5 ml of sodium hydroxide solution (4.3) and 5 ml of formaldehyde solution (4.4). Swirl and, by means of a pipette, transfer 25,0 ml of NaTPB solution (4.1) into the flask. Dilute to the mark with water, mix well and allow to stand for 5 min. Filter the solution through a dry filter paper (5.1), discarding the first few millilitres of the filtrate and collecting the remainder in a dry vessel.

By means of a pipette, transfer 50,0 ml of the clear filtrate into a 250 ml conical flask. Add 5 ml of thiazol yellow solution (4.5.2) and titrate with CTAB solution (4.5.1) until the colour of the solution changes to a distinct pink. Let V_2 be the volume used.

7.2.3.4 Calculation of the concentrations of the CTAB and NaTPB solutions

The concentrations, in moles per litre, of the CTAB and the NaTPB solutions are given, respectively, by the formulae

$$c(\text{CTAB}) = \frac{0,2}{10V_1 - V_2}$$

and

$$c(\text{NaTPB}) = \frac{0,16\ V_1}{10V_1 - V_2}$$

where

V_1 is the volume, in millilitres, of the CTAB solution (4.5.1) used in determining the equivalence of the CTAB and NaTPB solutions;

V_2 is the volume, in millilitres, of the CTAB solution used in standardizing the NaTPB solution.

7.2.3.5 Back titration

By means of a pipette, transfer 50,0 ml of the clear filtrate (7.2.2) into a 250 ml conical flask. Add 5 ml of thiazol yellow solution (4.5.2) and titrate with CTAB solution (4.5.1) until the colour changes to a distinct pink. Let V_4 be the volume used.

It is essential that V_4 lies in the range 2,5 to 8,5 ml. If it does not, repeat the determination with a recalculated volume of NaTPB solution required for the precipitation (7.2.2).

8 Expression of results

8.1 Calculation

The potassium content, expressed as K_2O as a percentage by mass, is given by the formula

$$\frac{4,71\,f\,[V_3 \cdot c\,(\text{NaTPB}) - 2V_4 \cdot c\,(\text{CTAB})]}{m}$$

where

f is the dilution factor (see table 1, column 7);

$c(\text{CTAB})$ is the concentration, in moles per litre, of the CTAB solution (see 7.2.3.4);

$c(\text{NaTPB})$ is the concentration, in moles per litre, of the NaTPB solution (see 7.2.3.4);

V_3 is the volume, in millilitres, of the NaTPB solution required for precipitation (7.2.2);

V_4 is the volume, in millilitres, of the CTAB solution used in the back titration (7.2.3.5);

m is the mass, in grams, of the test portion (see table 1, column 2).

8.2 Precision

Statistical analysis, in accordance with ISO 5725[1], of 108 sets of results, from 18 laboratories in six different countries, gave the following information on repeatability and reproducibility.

8.2.1 Repeatability

Successive results obtained using this method on identical test material and under the same conditions (same operator, same laboratory, same apparatus and same time) shall be considered suspect if they differ by more than the value r given in table 2.

Table 2 — Repeatability

Potassium content K_2O, % (m/m)		r K_2O, % (m/m)
from	to	
—	10	0,27
10	60	0,33

8.2.2 Reproducibility

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 value R given in table 3.

Table 3 — Reproducibility

Potassium content K_2O, % (m/m)		R K_2O, % (m/m)
from	to	
—	10	0,31
10	60	0,93

9 Test report

The test report shall include the following information:

a) the information required by ISO 5317 or ISO 7407;

b) a reference to this International Standard (i.e. ISO 5310);

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

10 Bibliography

Association of Official Analytical Chemists methods, 14th edition (1984), No. 2.119 to 2.121.

ISO 2052, *Potassium chloride for industrial use — Determination of potassium content — Sodium tetraphenylborate titrimetric method.*

ISO 2486, *Potassium sulphate for industrial use — Determination of potassium content — Sodium tetraphenylborate volumetric method.*

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[1] ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*