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Gnojila – Določevanje kalija, topnega v vodi

Fertilizers - Determination of the water-soluble potassium content

Düngemittel - Bestimmung von wasserlöslichem Kalium

Engrais - Détermination de la teneur en potassium soluble dans l'eau

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Fertilizers - Determination of the water-soluble potassium content

Engrais - Détermination de la teneur en potassium soluble dans l'eau

Düngemittel - Bestimmung von wasserlöslichem Kalium

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 260.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (prEN 15477:2015) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 15477:2009.

The following changes have been made to the former edition:

a) alternative amount of 1 g test portion taken into account in the case that the fertilizer contains more than 3 % of sulfur (S) and more than 4 % of calcium (Ca);

b) selection of apparatus extended by drying oven, boiling water bath and pipettes and graduated measuring cylinders of different capacities;

c) editorial revised.

1 Scope

This document specifies a method for the determination of water-soluble potassium, which is applicable to all potassium fertilizers listed in Annex I of the Regulation (EC) No 2003/2003 [3].

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

The potassium in the sample to be analysed is dissolved in water. After eliminating or fixing the substances that might interfere with the quantitative determination, the potassium is precipitated in a slightly alkaline medium in the form of potassium tetraphenylborate.

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

5.1 Formaldehyde, clear formaldehyde solution with a mass fraction of 25 % to 35 % formaldehyde.

5.2 Potassium chloride, p. a.

5.3 Sodium hydroxide solution, c = 10 mol/l.

Care should be taken to ensure that only potassium free sodium hydroxide is used.

5.4 Indicator solution.

Dissolve 0,5 g of phenolphthalein in ethanol at 90 % and make the volume up to 100 ml.

5.5 EDTA solution.

Dissolve 4 g of the dihydrated disodium salt of ethylenediaminetetraacetic acid in water in a 100 ml graduated flask. Make up the volume and mix.

Store the reagent in a plastics container.

5.6 STPB solution.

Dissolve 32,5 g of sodium tetraphenylborate in 480 ml of water, add 2 ml of the sodium hydroxide solution (5.4) and 20 ml of a magnesium chloride solution (100 g of $MgCl_2.6H_2O$ per litre).

Stir for 15 min and filter through a fine, ashless filter.

Store this reagent in a plastics container.

5.7 Liquid for washing.

Dilute 20 ml of the STPB solution (5.6) to 1 000 ml with water.

5.8 Bromine water, saturated bromine solution in water

6 Apparatus

6.1 Graduated flasks, capacity 1 000 ml.

6.2 Beakers, capacity 250 ml, 600 ml and 800 ml.

6.3 Filter crucibles, porosity 5 µm to 20 µm.

6.4 Drying oven, regulated at (120 ± 10) °C.

6.5 Bath, boiling water.

6.6 Desiccator.

6.7 Pipettes, capacity 10 ml, 25 ml and 50 ml.

6.8 Graduated measuring cylinders, class A, capacity 250 ml and 500 ml.

7 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2. Grinding is recommended for homogeneity reasons.

8 Procedure

8.1 Test portion

Weigh to the nearest 0,001 g 10 g of the prepared sample (5 g for potassium salts with a mass fraction of potassium oxide of more than 50 %). Place this test portion in a 600 ml beaker (6.2) with approximately 400 ml of water.

Where the fertilizer contains more than 3 % of sulfur (S) and more than 4 % of calcium (Ca) weigh 1 g of the test sample to the nearest 1 mg. Place it in the 600 ml beaker (6.2) with approximately 400 ml of water.

Bring to a boil and allow it to boil for 30 min. Cool, transfer quantitatively into a 1 000 ml graduated flask (6.1), make up the volume, mix and filter into a dry receiver. Discard the first 50 ml of the filtrate (see 8.6).

8.2 Preparation of the aliquot part for precipitation

Transfer by pipette (6.7) or graduated measuring cylinder (6.8) an aliquot part of the filtrate containing 25 mg to 50 mg of potassium oxide (see Table 1) and place it in a 250 ml, 600 ml or 800 ml beaker (6.2) whichever is appropriate. If required make up to 50 ml with water.

To remove any interference, add 10 ml of the EDTA solution (5.5), several drops of the phenolphthalein solution (5.4) and stir in, drop by drop, sodium hydroxide solution (5.3) until it turns red, then finally add a few more drops of sodium hydroxide to ensure an excess (usually 1 ml of sodium hydroxide is sufficient to neutralize the sample and ensure an excess).

To eliminate most of the ammonia, (see 8.6) boil gently for 15 min.

If necessary, add water to make the volume up to 60 ml.

Bring the solution to the boil, remove the beaker from the heat and add 10 ml of formaldehyde (5.1). Add several drops of phenolphthalein (5.4) and, if necessary, some more sodium hydroxide (5.3) until a distinct red colour appears. Cover the beaker with a watch glass and place it on a boiling water (steam) bath (6.5) for 15 min.

8.3 Weighing the crucible

Dry the filter crucible (6.3) to a constant mass (about 15 min) in the drying oven (6.4) at 120 °C.

Allow the crucible to cool in a desiccator (6.6) and then weigh it.

8.4 Precipitation

Remove the beaker from the boiling water (steam) bath (6.5), stir in drop-by-drop 10 ml of the STPB solution (5.6). This addition takes about 2 min. Wait for at least 10 min before filtering.

8.5 Filtering and washing

Filter under vacuum into the weighed crucible (6.3), rinse the beaker with the liquid for washing (5.7), wash the precipitate three times with the liquid for washing (60 ml in all of the liquid for washing), and twice with 5 ml to 10 ml of water.

Dry the precipitate thoroughly.

8.6 Drying and weighing

Wipe the outside of the crucible (6.3) with a filter paper. Place the crucible with its contents in the drying oven (6.4) for 1,5 h at 120 °C. Allow the crucible to cool in a desiccator (6.6) to ambient temperature and weigh immediately.

If the filtrate is dark in colour, transfer by pipette (6.7), an aliquot part containing at the most, 100 mg of K_2O and place in a 100 ml graduated flask (6.1). Add bromine water (5.8) and bring to a boil to eliminate any surplus bromine. After cooling make up the volume, filter and quantitatively determine the potassium in an aliquot part of the filtrate.

Where there is little or no ammoniacal nitrogen present there is no need to boil for 15 min.

8.7 Aliquot parts to be taken as samples and conversion factors

Table 1 — Aliquot parts and conversion factors

K_2O in the fertilizer %	K in the fertilizer %	Sample for analysis g	Sample of the extract solution for the dilution ml	Dilution to ml	Aliquot part to be taken as a sample for precipitation ml	Conversion factor F $\frac{\%\ K_2O}{g\ TPBK}$	Conversion factor F' $\frac{\%\ K}{g\ TPBK}$
5 to 10	4,2 to 8,3	10	-	-	50	26,280	21,812
5 to 10	4,2 to 8,3	1	-	-	500	26,280	21,812
10 to 20	8,3 to 16,6	10	-	-	25	52,560	43,624
10 to 20	8,3 to 16,6	1	-	-	250	52,560	43,624
20 to 50	16,6 to 41,5	10 {	either – or 50	250	10 50	131,400 131,400	109,060 109,060
more than 50	more than 41,5	5{	either – or 50	- 250	10 50	262,800 262,800	218,120 218,120

8.8 Blank test

For each series of determinations, carry out a blank test using only the reagents in the proportions used in the analysis and allow for this when calculating the final result.

8.9 Control test

In order to obtain a control for the method of analysis, carry out a determination on an aliquot part of an aqueous solution of potassium chloride, containing at the most 40 mg of K_2O.

9 Calculation and expression of the result

9.1 Dilution according to Table 1

Calculate the K_2O content, w_{K_2O}, as mass fraction in percent of the fertilizer according to Formula (1):

$$w_{K_2O} = (m_1 - m_2) \times F \quad (1)$$

Calculate the K content, w_K, as mass fraction in percent of the fertilizer according to Formula (2):

$$w_K = (m_1 - m_2) \times F' \quad (2)$$

where

m_1 is the mass of the precipitate from the sample, in grams;

m_2 is the mass of the precipitate from the blank, in grams;

F and F' conversion factors (see Table 1).

9.2 Dilution different from Table 1

Calculate the K_2O content, w_{K_2O}, as mass fraction in percent of the fertilizer according to Formula (3):

$$w_{K_2O} = \frac{(m_1 - m_2) \times F \times D \times 100}{m} \quad (3)$$

Calculate the K content, w_K, as mass fraction in percent of the fertilizer according to Formula (4):

$$w_K = \frac{(m_1 - m_2) \times F' \times D \times 100}{m} \quad (4)$$

where

m_1 is the mass of the precipitate from the sample, in grams;

m_2 is the mass of the precipitate from the blank, in grams;

F conversion factor, KTPB into K_2O = 0,1314;

F' conversion factor, KTPB into K = 0,109;

D dilution factor;

m is the mass of the sample for analysis (test portion), in grams.

10 Precision

10.1 Inter-laboratory test

An inter-laboratory test was carried out in 2004 with 16 participating laboratories and two different samples of fertilizers and phosphate types. This test yielded the data given in Annex A. Repeatability and reproducibility was calculated according to ISO 5725-1.

The values derived from this inter-laboratory test might not be applicable to concentration ranges and matrices other than those given in Annex A.

10.2 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in no more than 5 % of the cases exceed the values of r given in Table 2.

10.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in no more than 5 % of the cases exceed the values of R given in Table 2.

Table 2 — Mean values, repeatability and reproducibility limits

Sample	$\overline{x}$ %	r %	R %
NPK1 (14–8-24+8S)	24,66	0,26	0,71
NPK2 (16–16–8+4S)	8,18	0,12	0,32