BRITISH STANDARD

BS EN 15477:2009

Fertilizers — Determination of the water-soluble potassium content

ICS 65.080

BSI
British Standards

National foreword

This British Standard is the UK implementation of EN 15477:2009. It supersedes DD CEN/TS 15477:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 28 February 2009

ISBN 978 0 580 62383 7

Amendments/corrigenda issued since publication

Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15477

January 2009

ICS 65.080

Supersedes CEN/TS 15477:2006

English Version

Fertilizers - Determination of the water-soluble potassium content

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

Düngemittel - Bestimmung von wasserlöslichem Kalium

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Contents

Page

Foreword

This document (EN 15477:2009) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

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1 Scope

This European Standard 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 referenced documents are indispensable for the application of this document. 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 analyzed 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

5.1 General

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

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

5.3 Potassium chloride, p. a.

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

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

5.5 Indicator solution

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

5.6 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.

5.7 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.

5.8 Liquid for washing

Dilute 20 ml of the STPB solution (5.7) to 1 000 ml with water.

5.9 Bromine water, saturated bromine solution in water.

6 Apparatus

6.1 Graduated flasks, capacity 1 000 ml.

6.2 Beaker, capacity 250 ml and 600 ml.

6.3 Filter crucibles, porosity 5 µm to 20 µm.

6.4 Oven, regulated at (120 ± 10) °C.

6.5 Desiccator

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 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, 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 an aliquot part of the filtrate containing 25 mg to 50 mg of potassium (see Table 1) and place it in a 250 ml beaker. If required make up to 50 ml with water.

To remove any interference, add 10 ml of the EDTA solution (5.6), several drops of the phenolphthalein solution (5.5) and stir in, drop by drop, sodium hydroxide solution (5.4) 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 (5.2). Add several drops of phenolphthalein and, if necessary, some more sodium hydroxide until a distinct red colour appears. Cover the beaker with a watch glass and place it on a steam bath for 15 min.

8.3 Weighing the crucible

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

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

8.4 Precipitation

Remove the beaker from the steam bath, stir in drop-by-drop 10 ml of the STPB solution (5.7). 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, rinse the beaker with the liquid for washing (5.8), 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 with a filter paper. Place the crucible with its contents in the oven for 1,5 h at 120 °C. Allow the crucible to cool in a desiccator to ambient temperature and weigh immediately.

If the filtrate is dark in colour, transfer by pipette, an aliquot part containing at the most, 100 mg of K_2O and place in a 100 ml graduated flask. Add bromine water (5.9) 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 - 10	4,2 – 8,3	10	-	-	50	26,280	21,812
10 - 20	8,3 – 16,6	10	-	-	25	52,560	43,624
20 - 50	16,6 – 41,5	10{	either –		10	131,400	109,060
			or 50	250	50	131,400	109,060
more than 50	more than 41,5	5{	either –	-	10	262,800	218,120
			or 50	250	50	262,800	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 equation (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 equation (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 equation (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 equation (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 were 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 not 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 not more than 5 % of the cases exceed the values of R given in Table 2.

Table 2 — Mean values, repeatability and reproducibility limits

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

11 Test report

The test report shall contain at least the following information:

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

b) test method used with reference to this document;

c) test results obtained;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method which might have influenced the test result(s).

Annex A
(informative)

Results of the inter-laboratory tests

The precision of the method was established in 2004 by Working Group 7 “Chemical analysis” of CEN/TC 260 “Fertilizers and liming materials” in an inter-laboratory test evaluated in accordance with ISO 5725-1. The statistical results are given in Table A.1.

Table A.1 — Statistical results of the inter-laboratory test

Parameter	Sample	
	NPK1 (14-8-24+8S)	NPK2 (16-16-8+4S)
Number of participating laboratories	16	16
Number of laboratories after elimination of outliers (accepted test results)	13	14
Mean value $\bar{x}$ (%)	24,66	8,18
Repeatability standard deviation s_r (%)	0,09	0,04
RSD_r (%)	0,4	0,5
Repeatability limit r (%)	0,26	0,12
Reproducibility standard deviation s_R (%)	0,25	0,11
RSD_R (%)	1,0	1,4
Reproducibility limit R (%)	0,71	0,32

Bibliography

[1] EN 1482-1, Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling

[2] ISO 5725-1, Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions

[3] Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers, Official Journal L 304, 21/11/2003 P. 0001-0194, Annex IV, method 4.1

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