SLOVENSKI STANDARD
SIST EN 17042:2018

01-september-2018

Gnojila - Določevanje bora v koncentracijah, večjih od 10 %, s kislinsko titracijo

Fertilizers - Determination of boron in concentrations > 10 % using acidimetric titration

Düngemittel - Bestimmung von Bor in Konzentrationen > 10 % durch azidimetrische Titration

Engrais - Dosage du bore dans des concentrations > 10 % par titration acidimétrique

Ta slovenski standard je istoveten z: **EN 17042:2018**

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65.080	Gnojila	Fertilizers

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English Version

Fertilizers - Determination of boron in concentrations > 10 % using acidimetric titration

Engrais - Dosage du bore dans des concentrations > 10 % par titrage acidimétrique

Düngemittel - Bestimmung von Bor in Konzentrationen > 10 % durch azidimetrische Titration

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

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European foreword

This document (EN 17042:2018) 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of an identical text or by endorsement, at the latest by December 2018, and conflicting national standards shall be withdrawn at the latest by December 2018.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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Introduction

The preparation of this document by CEN is based on a mandate by the European Commission and the European Free Trade Association (Mandate M/335), concerning the modernization of methods of analysis of fertilizers in the framework of Regulation (EC) No 2003/2003 [1].

This European Standard is part of a modular approach and concerns the analytical measurement step. "Modular" means that a test standard concerns a specific step in assessing a property and not the whole chain of measurement.

The determination of boron in fertilizers can be executed by inductively coupled plasma-atomic emission spectrometry (ICP-AES). Acidimetric determination after addition of mannitol is more labour intensive but it is an option when ICP-AES is not available.

WARNING — Persons using this European Standard should be familiar with normal laboratory practice. This European Standard does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this European Standard are carried out by suitably trained staff.

1 Scope

This European Standard specifies a method for the determination of total and water extractable boron in mineral fertilizers containing more than 10 % boron.

This method is applicable to water and aqua regia fertilizer extracts obtained according to EN 16962 and/or EN 16964.

NOTE A method used for the determination of boron in mineral fertilizers containing less than or equal to 10 % of boron (spectrophotometric determination by azomethine-H) can be also used for the scope of this method after appropriate dilution of the extracts.

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, *Fertilizers and liming materials and soil improvers - Vocabulary - Part 1: General terms*

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

EN 16962, *Fertilizers - Extraction of water soluble micro-nutrients in fertilizers and removal of organic compounds from fertilizer extracts*

EN 16964, *Fertilizers - Extraction of total micro-nutrients in fertilizers using aqua regia*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

3 Terms and definitions

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

4 Principle

Boric acid forms a complex with mannitol: $H_3BO_3 + C_6H_{14}O_6 \rightarrow C_6H_{15}O_8B + H_2O$.

This complex acid is titrated with sodium hydroxide solution to a pH of 6,3.

5 Sampling and sample preparation

Sampling is not part of this European Standard. A recommended sampling method is specified in EN 1482-1 [2].

Sample preparation shall be carried out in accordance with EN 1482-2. The sample extracts shall be prepared in accordance with EN 16962 and/or EN 16964.

6 Reagents

All reagents shall be of recognized analytical grade. All reagents including water shall have negligible concentration of boron if compared to the lowest concentration of that element in the sample solution.

6.1 Water, grade 2 according to EN ISO 3696 and free from boron.

6.2 Methyl red indicator solution.

Dissolve 0,1 g of methyl red ($C_{15}H_{15}N_3O_2$) in 50 ml of ethanol (95 %) in a 100 ml volumetric flask (7.5). Make up the volume to 100 ml with water (6.1). Mix thoroughly.

6.3 Diluted hydrochloric acid solution, with a concentration of about c = 0,5 mol/l.

Mix 1 volume of hydrochloric acid HCl (ρ = 1,18 g/ml) with 20 volumes of water (6.1).

6.4 Sodium hydroxide solution, with a concentration of about c = 0,5 mol/l.

The solution shall be free from carbon dioxide. Dissolve 20 g of sodium hydroxide (NaOH) in pellet form in a 1 000 ml volumetric flask (7.6) containing about 800 ml of boiled water (6.1). After dissolution and cooling make up to 1 000 ml with boiled water (6.1) and mix thoroughly.

6.5 Standard sodium hydroxide solution, with a concentration of about c = 0,025 mol/l.

The solution shall be free from carbon dioxide. Dilute 0,5 mol/l sodium hydroxide solution (6.4) 20 times with boiled water and mix thoroughly. The value of the solution expressed as boron (B) is to be determined (8.3).

6.6 Boron calibration solution, ρ = 100 mg/l.

Weigh to the nearest 0,1 mg 0,571 9 g of boric acid (H_3BO_3), dissolve in water (6.1), transfer quantitatively into a 1 000 ml volumetric flask (7.6), make up to volume with water (6.1) and mix thoroughly. Transfer to a plastic bottle for storage in a refrigerator at 4 °C to 8 °C.

Commercially available stock solution with adequate specification may be used. The solution is generally considered to be stable for one year, but in reference to guaranteed stability, the recommendations of the manufacturer shall be considered.

6.7 D-mannitol, ($C_6H_{14}O_6$) powder.

6.8 Sodium chloride (NaCl).

7 Apparatus

7.1 pH meter, with glass electrode.

7.2 Magnetic stirrer.

7.3 Beaker, capacity 400 ml.

7.4 Polytetrafluoroethylene (PTFE) (Teflon®)[1] **rod**.

7.5 Volumetric flasks, capacity 100 ml.

[1] This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7.6 Volumetric flasks, capacity 1 000 ml.

8 Procedure

8.1 Test solution

Place an aliquot (a) of the extract containing 2 mg to 4 mg boron into a 400 ml beaker (7.3). Add 150 ml of water (6.1). Add several drops of the methyl red indicator solution (6.2). If necessary acidify by adding hydrochloric acid solution (6.3) up to the point of change of the indicator. Then add 3 g of sodium chloride (6.8), bring to boiling to drive off the carbon dioxide. Allow to cool.

Place the beaker on the magnetic stirrer (7.2) and mix by using a PTFE rod (7.4). Insert the pre-calibrated pH meter electrodes (7.1) into the beaker and adjust the pH first approximately up to 5 with the 0,5 mol/l sodium hydroxide solution (6.4), then to exactly pH = 6,3 with the 0,025 mol/l sodium hydroxide solution (6.5). Add 20 g of D-mannitol (6.7), dissolve completely and titrate with the 0,025 mol/l sodium hydroxide solution (6.5) to pH 6,3 (at least 1 min stability). Let X_1 be the volume of 0,025 mol/l sodium hydroxide solution (6.5) required.

8.2 Blank solution

Prepare a blank solution by repeating the whole procedure from the preparation of solution stage, omitting only the fertiliser. Let X_0 be the volume of 0,025 mol/l sodium hydroxide solution (6.5) required.

8.3 Boron (B) value of the sodium hydroxide solution (6.5)

Pipette 20 ml of the calibration solution (6.6) into a 400 ml beaker (7.3) and add several drops of methyl red indicator solution (6.2). The beaker contains 2,0 mg of boron. Add 3 g of sodium chloride (6.8) and the hydrochloric acid solution (6.3) up to the point of change of the indicator solution (6.2). Make up the volume with water (6.1) to about 150 ml and bring gradually to the boil so as to eliminate carbon dioxide. Allow to cool.

Place the beaker (7.3) on the magnetic stirrer (7.2) and mix by using a PTFE rod (7.4). Insert the pre-calibrated pH meter electrodes (7.1) into the beaker and adjust the pH first approximately up to 5 with the 0,5 mol/l sodium hydroxide solution (6.4), then to exactly pH = 6,3 with the 0,025 mol/l sodium hydroxide solution (6.5).

Add 20 g of D-mannitol (6.7), dissolve completely and titrate with the 0,025 mol/l sodium hydroxide solution (6.5) to pH 6,3 (at least 1 min stability). Let V_1 be the volume of 0,025 mol/l sodium hydroxide solution (6.5) required.

Prepare a blank solution in the same way, substituting 20 ml of water for the calibration solution. Let V_0 be the volume required.

Calculate the boron value (F) in mg/ml of the standard NaOH solution (6.5) according to Formula (1).

$$F = 2 / (V_1 - V_0) \quad (1)$$

where

F Is the boron (B) value, in mg/ml, of the 0,025 mol/l sodium hydroxide solution;

V_0 is the volume of the 0,025 mol/l sodium hydroxide solution (6.5) required for titration of the blank solution, in ml;

V_1 is the volume of the 0,025 mol/l sodium hydroxide solution (6.5) required for titration of the calibration solution, in ml.