SLOVENSKI STANDARD
SIST EN 17041:2018

01-september-2018

Gnojila - Določevanje bora v koncentracijah, manjših ali enakih 10 %, s spektrometrijo z azometinom-H

Fertilizers - Determination of boron in concentrations ≤ 10 % using spectrometry with azomethine-H

Düngemittel - Bestimmung von Bor in Konzentrationen ≤ 10 % durch Spektrometrie mit Azomethin-H

Engrais - Dosage du bore dans des concentrations ≤ 10 % par spectrométrie avec l'azomethine-H

Ta slovenski standard je istoveten z: **EN 17041:2018**

ICS:

65.080	Gnojila	Fertilizers

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English Version

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Engrais - Dosage du bore dans des concentrations ≤ 10 % par spectrométrie avec l'azomethine-H

Düngemittel - Bestimmung von Bor in Konzentrationen ≤ 10 % durch Spektrometrie mit Azomethin-H

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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 17041:2018) 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of an identical text or by endorsement, at the latest by December 2018, and conflicting national standards shall be withdrawn at the latest by December 2018.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 on 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 measurements.

The determination of boron in fertilizers can be executed by inductively coupled plasma-atomic emission spectrometry (ICP-AES). Spectrophotometric determination with azomethine-H is more labour intensive than ICP-AES method (EN 16963) but the method is reliable and relatively inexpensive and it is an option when ICP-AES is not available.

The spectrophotometric determination can be influenced by iron and more attention is necessary also to organic matter removal and interferences from extract colour. The procedure for removal of organic matter from the extracts is given in EN 16962.

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 less than or equal to 10 % boron. The method is not suitable for fertilizers with Fe concentrations more than twenty times higher than the concentration of boron.

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

The method can also be used for the determination of boron in mineral fertilizers containing more than 10 % boron 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

Boron reacts with azomethine-H and the yellow complex produced is determined spectrophotometrically at 410 nm. Interfering ions are masked with ethylenediaminetetraacetic acid (EDTA). Interference from intensely coloured Fe(III)-EDTA complex is removed by reduction of Fe(III) to Fe(II).

5 Sampling and sample preparation

Sampling is not part of the method specified in this document. 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 and shall have negligible concentration of boron if compared to the lowest concentration of that element in the sample solution.

All reagents including water shall be tested for presence of boron.

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

6.2 EDTA buffer solution.

6.2.1 Ammonium acetate (CH_3COONH_4).

6.2.2 Disodium salt of ethylenediaminetetraacetic acid (Na_2EDTA).

6.2.3 Acetic acid, ρ = 1,11 g/ml.

Place in a 500 ml volumetric flask (7.4) containing 300 ml of water (6.1): 75 g ammonium acetate (6.2.1), 10 g disodium salt of ethylenediaminetetraacetic acid (6.2.2) and 40 ml acetic acid (6.2.3). Make up to volume with water (6.1) and mix thoroughly. The pH value of the solution, checked by means of a glass electrode (7.9), shall be 4,8 ± 0,1. Store the solution in a plastic bottle (7.6).

6.3 Azomethine-H solution.

6.3.1 Azomethine-H ($C_{17}H_{12}NNaO_8S_2$).

6.3.2 Ascorbic acid ($C_6H_8O_6$).

Place in a 200 ml volumetric flask (7.3): 10 ml of the buffer solution (6.2), 400 mg of azomethine-H (6.3.1) and 2 g of ascorbic acid (6.3.2). Make up to volume with water (6.1) and mix thoroughly. The solution is stable for three days.

6.4 Boron calibration solutions.

6.4.1 Boron stock solution, 100 mg/l.

Dissolve 0,571 9 g boric acid (H_3BO_3) in water (6.1) in a 1 000 ml volumetric flask (7.5). Make up to volume with water (6.1) and mix thoroughly. Transfer to a plastic bottle (7.6) and store at (4 to 8) °C.

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

6.5 Nitric acid, $c(HNO_3)$ = 14,3 mol/l; ρ = 1,4 g/ml.

6.5.1 Diluted nitric acid solution, $c(HNO_3)$ = 5 mol/l.

Add 350 ml of nitric acid (6.5) to 650 ml of water (6.1).

7 Apparatus

7.1 Laboratory glassware.

IMPORTANT — It is necessary to minimize contact of all solutions with borosilicate glassware. Suitable plastic or silica ware should be used. Glass volumetric flasks may be used for making up to volume but not for storage of extracts and solutions.

7.2 Volumetric flasks, capacity 100 ml.

7.3 Volumetric flasks, capacity 200 ml.

7.4 Volumetric flasks, capacity 500 ml.

7.5 Volumetric flasks, capacity 1 000 ml.

7.6 Plastic bottles.

7.7 Cuvettes, 10 mm and 20 mm to 50 mm optical path.

7.8 Spectrophotometer, set to a wavelength of 410 nm with a cuvette having a 10 mm optical path.

If low boron content is to be determined, a cuvette having 20 mm to 50 mm optical path and adequately adopted calibration range may be used.

7.9 pH meter, with glass electrode.

8 Procedure

8.1 General

For determination of boron it is necessary to avoid contamination of the extracts, blanks and calibration solutions with boron from borosilicate glass and/or detergents. Avoid or minimize contact of these solutions with borosilicate glass.

8.2 Preparation of calibration solutions

Pipette 0 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of the boron stock solution (6.4.1) to a series of 100 ml volumetric flasks (7.2), add 10 ml of diluted nitric acid (6.5.1), make up to volume with water (6.1) and mix thoroughly. These solutions contain 0 mg/l, 1 mg/l, 2 mg/l, 3 mg/l, 4 mg/l and 5 mg/l of boron.

8.3 Preparation of the test solutions

Dilute an aliquot portion of the extract, obtained according to EN 16962 or EN 16964, in one or more steps so that the final concentration of the element to be determined is in the given calibration range (8.2). In the final diluting step add a suitable volume of the extract or diluted extract to a 100 ml volumetric flask (7.2), add 10 ml of diluted nitric acid (6.5.1), fill to the mark with water (6.1) and mix well. The final substance concentration of acid in the solution is approximately 0,5 mol/l. Aqua regia extracts shall be diluted at least 5 times with water (6.1) to achieve this acid concentration.

Prepare a blank solution by pipetting only extracting solution diluted in the same way as the test solution.