SLOVENSKI STANDARD
SIST EN 16962:2018

01-junij-2018

Gnojila - Ekstrakcija v vodi topnih mikrohranil iz gnojil in odstranitev organskih spojin iz ekstraktov gnojil

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

Düngemittel - Extraktion wasserlöslicher Spurennährstoffe aus Düngemitteln und Beseitigung organischer Verbindungen aus Düngemittelextrakten

Engrais - Extraction des oligo-éléments soluble dans l'eau des engrais et élimination des composés organiques dans les extraits d'engrais

Ta slovenski standard je istoveten z: EN 16962:2018

ICS:

65.080	Gnojila	Fertilizers

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EUROPEAN STANDARD

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English Version

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

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

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Contents

European foreword

This document (EN 16962:2018) 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of an identical text or by endorsement, at the latest by July 2018, and conflicting national standards shall be withdrawn at the latest by July 2018.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 is based on a mandate given to CEN 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].

Water extraction is empirical and in many cases cannot be quantitative. The extraction is dependent on temperature and extraction time, on the ratio of water/sample and intensity of the extraction. To reduce the level of uncertainty of the extraction step to a reasonable level it is necessary to strictly follow the given procedure. The method can be applied to mineral fertilizers with micro-nutrient content of ≤ 10 % as well as of > 10 %. Removal of organic compounds is not usually necessary if FAAS or ICP-AES method of determination is used. It is applied only in some cases for determination of boron and molybdenum by spectrophotometric methods, determination of boron by acidimetric titration and gravimetric determination of molybdenum. The method for removal of organic compounds is a part of this standard.

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 extraction of water soluble forms of boron, cobalt, copper, iron, manganese, molybdenum and zinc from mineral fertilizers containing one or more micro-nutrients and the procedure for removal of organic compounds from the water extracts.

The extracts can be analysed according to EN 16963, EN 16965, prEN 17041, prEN 17042, prEN 17043 and CEN/TS 17060.

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 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 16963, *Fertilizers — Determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc using ICP-AES*

EN 16965, *Fertilizers — Determination of cobalt, copper, iron, manganese and zinc using flame atomic absorption spectrometry (FAAS)*

prEN 17043, *Fertilizers — Determination of molybdenum in concentrations ≤ 10 % using spectrometry of a complex with ammonium thiocyanate*

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

The micro-nutrients are extracted by shaking the fertilizer sample in water at (20 ± 2) °C. Extracts are acidified after the extraction to avoid hydrolysis. Organic matter is removed if necessary by boiling after addition of hydrogen peroxide.

5 Sampling and sample preparation

Sampling and sample preparation are not part of this Standard. A recommended sampling method is given in EN 1482-1 [2] and a recommended sample preparation method in EN 1482-2 [3].

6 Reagents

All reagents shall be of recognized analytical grade and they shall have negligible concentration of the element to be determined if compared to the lowest concentration of that element in the sample solution.

6.1 Water for extraction, grade 2 according to EN ISO 3696, free from micro-nutrients.

6.2 Nitric acid 65 %, $c(HNO_3)$ = 14,3 mol/l; ρ = 1,42 g/ml.

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

Add 350 ml of nitric acid (6.2) to 650 ml of water (6.1).

6.3 Hydrogen peroxide solution 30 % H_2O_2, ρ = 1,11 g/ml, free from micro-nutrients.

6.4 Hydrochloric acid 37 % HCl, $c(HCl)$ = 12 mol/l, ρ = 1,18 g/ml.

6.4.1 Diluted hydrochloric acid solution, $c(HCl)$ = 5 mol/l.

Add 420 ml of hydrochloric acid (6.4) to 580 ml of water (6.1).

7 Apparatus

If the boron content is to be determined it is recommended to minimize contact of all solutions with borosilicate glassware. Use suitable plastic or silica ware especially for extraction step and for removal of organic compounds. Glass volumetric flasks may be used for making up to volume but shall not be used for storage of extracts and solutions.

7.1 Shaker, rotary or horizontal eccentric shaker.

Shaking shall prevent any settling of the sample during extraction.

7.2 Electric hotplate, equipped with variable temperature control.

7.3 Extraction vessels, capacity 500 ml and 1 000 ml.

7.4 Filter paper, ash-free and of recognized and tested quality.

7.5 Beaker, capacity 100 ml.

7.6 Volumetric flasks, capacity 25 ml and 50 ml.

7.7 Watch glass.

8 Procedure

8.1 Preparation of the test solutions

8.1.1 Micro-nutrient content ≤ 10 %

For samples with a micro-nutrient content of ≤ 10 % weigh (5 ± 0,005) g of the prepared sample, transfer the sample quantitatively to a 500 ml extraction vessel (7.3) and add (250 ± 0,2) ml of water (6.1) at (20 ± 2) °C.

8.1.2 Micro-nutrient content > 10 %

For samples with a micro-nutrient content of > 10 % weigh (2 ± 0,002) g of the prepared sample, transfer the sample quantitatively to a 1 000 ml extraction vessel (7.3) and add (500 ± 0,5) ml of water (6.1) at (20 ± 2) °C.

8.2 Extraction

Close the vessel tightly and shake vigorously by hand to disperse the sample. Then place the vessel on the shaker (7.1) and shake for 60 min. The intensity of shaking shall be adjusted to prevent any settling of the sample during the extraction. The temperature of the extraction solution shall be kept at (20 ± 2) °C during the whole extraction time. Filter the extract if necessary immediately after the extraction into a plastic vessel. Use ash-free filter paper of recognized and tested quality (7.4) and discard the first portion of the filtrate (approximately 20 ml). Pipette 20 ml of the extract into a 25 ml volumetric flask, add 2,5 ml of diluted nitric acid solution (6.2.1), fill to the mark with water (6.1) and mix well. The final concentration of nitric acid is 0,5 mol/l.

Account shall be taken of this dilution when taking aliquot portions and calculating the percentage of micro-nutrient in the sample.

Prepare a blank test solution following the same procedure as for samples.

Acidification of the water extract with nitric acid is not suitable for spectrophotometric determination of molybdenum according to prEN 17043. In this case hydrochloric acid (6.4) shall be added; the final concentration of hydrochloric acid is 0,5 mol/l. Acidification with hydrochloric acid shall be used also if precipitation of Fe(III) is observed after the addition of nitric acid. If boron is not to be determined standard glassware may be used. If the measurement is not carried out immediately the acidified extracts may be stored in tightly closed plastic vessels for up to 15 days.

If a higher volume of the extract is needed for the measurement to follow, a different volume of the extract may be pipetted but the final concentration of nitric acid (0,5 mol/l) and the final diluting factor (5/4) shall be the same. For example, 40 ml of the extract is pipetted into a 50 ml volumetric flask (7.6), 5 ml of diluted nitric acid solution (6.2.1) is added and the flask is filled to the mark with water (6.1). Centrifugation of the extracts may be used instead of filtration.

Filter paper with a hydrophobic edge or centrifugation is recommended for finely ground samples.

Some chelated micro-nutrients may undergo photolytic decomposition during the extraction resulting in precipitation. In this case, opaque vessels shall be used for the extraction step and samples shall be stored in the dark.

8.3 Removal of organic compounds

Transfer 25 ml of the extract prepared for measurement (see 8.2) quantitatively into a 100 ml beaker (7.5) (use a maximum of 10 ml of water for quantitative transfer from the 25 ml volumetric flask (7.6)), add 5 ml of hydrogen peroxide (6.3). Cover with a watch glass (7.7) and allow oxidation to occur at laboratory temperature for about 1 h, then bring gradually to boiling on an electric hotplate (7.2) and boil for 0,5 h. If the extract is still visibly coloured by the presence of organic matter, add a further amount of 5 ml of hydrogen peroxide (6.3) to the solution once it has cooled. Then boil for 0,5 h to remove the excess hydrogen peroxide. Allow to cool and transfer quantitatively to a 50 ml volumetric flask, add 2,5 ml of diluted nitric acid (6.2.1) and make up to volume.

The final concentration of nitric acid is 0,5 mol/l, the dilution factor (final volume/extract volume) is 5/2.

For spectrophotometric determination of molybdenum according to prEN 17043, add 2,5 ml of diluted hydrochloric acid (6.4.1) instead of diluted nitric acid (6.2.1).

Account shall be taken of this dilution when taking aliquot portions and calculating the percentage of micro-nutrients in the sample.

NOTE In most cases, the presence of small quantities of organic matter will not affect determinations by FAAS and ICP-AES and it is not necessary to apply organic matter removal.