SLOVENSKI STANDARD
SIST EN 16964:2018

01-junij-2018

Gnojila - Ekstrakcija celotnih mikrohranil iz gnojil z zlatotopko

Fertilizers - Extraction of total micro-nutrients in fertilizers using aqua regia

Düngemittel - Extraktion von Gesamtspurennährstoffen aus Düngemitteln mit Königswasser

Engrais - Extraction des oligo-éléments totaux des engrais à l'eau régale

Ta slovenski standard je istoveten z: **EN 16964:2018**

ICS:

65.080	Gnojila	Fertilizers

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EUROPEAN STANDARD

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English Version

Fertilizers - Extraction of total micro-nutrients in fertilizers using aqua regia

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This European Standard was approved by CEN on 15 October 2017.

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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 16964:2018) 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of an identical text or by endorsement, at the latest by July 2018, and conflicting national standards shall be withdrawn at the latest by July 2018.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.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The preparation of this document was 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].

Aqua regia is applied for determination of many elements in different matrices. A horizontal standard for aqua regia extraction of soils, sludges and biowaste was prepared by CEN/TC 400. Similar procedure was applied for determination of the aqua regia extractable content of arsenic, mercury, cadmium, chromium, nickel and lead in fertilizers and liming materials (European Standard prepared by CEN/TC 260). Wide use of the aqua regia extraction and possibility to prepare a suitable horizontal standard was the main reason to develop the given extraction procedure.

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 total extraction of boron, cobalt, copper, iron, manganese, molybdenum and zinc with aqua regia from mineral fertilizers containing one or more micro-nutrients.

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

This method is also suitable for the extraction of cadmium, chromium, nickel and lead to be determined according to EN 16319; the extraction of mercury to be determined according to EN 16320 and the extraction of arsenic to be determined according to EN 16317.

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)*

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 from the sample with boiling aqua regia.

5 Sampling and sample preparation

Sampling and sample preparation are not part of this European 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 Hydrochloric acid 37 % HCl, $c(HCl)$ = 12 mol/l, ρ = 1,18 g/ml.

6.3 Nitric acid 65 %, $c(HNO_3)$ = 14,3 mol/l; ρ approximately 1,4 g/ml.

7 Apparatus

7.1 Common laboratory glassware.

If the boron content is to be determined, it is necessary to minimize the contact of all solutions with borosilicate glassware.

Suitable plastic or silica ware is needed especially for the extraction step. Glass volumetric flasks may be used for making up to volume but not for storage of extracts and solutions.

7.2 Apparatus for thermal heating digestion, temperature controlled with reaction vessel and reflux condenser.

The capacity of the reaction vessel shall be at least 5 times of the volume of the aqua regia used.

7.3 Filter paper, ash free and of recognized and tested quality.

8 Procedure

8.1 General

CAUTION — Suitable precautions shall be taken to avoid any contact of laboratory staff with acid fumes. It is recommended that the extraction procedure is carried out in a fume cupboard.

8.2 Preparation of the test solutions

8.2.1 Micro-nutrient content ≤ 10 %

For samples with a micro-nutrient content of ≤ 10 % weigh (3 ± 0,003) g of the prepared sample, transfer the sample quantitatively to the reaction vessel (7.2).

8.2.2 Micro-nutrient content > 10 %

For samples with a micro-nutrient content of > 10 % weigh (1 ± 0,001) g of the prepared sample, transfer the sample quantitatively to the reaction vessel (7.2).

8.3 Extraction

Moisten the test portion (8.2.1 or 8.2.2) with about 0,5 ml to 1,0 ml of water (6.1) and add, while mixing, 21 ml of hydrochloric acid (6.2) followed by 7 ml of nitric acid (6.3) drop wise if necessary to reduce foaming. Connect a condenser to the reaction vessel and let the mixture stand at laboratory temperature until any effervescence almost ceases. Transfer to the heating device and slowly raise the temperature of the reaction mixture to reflux conditions and maintain for 2 h ensuring that the condensation zone is lower than 1/3 of the height of the condenser. Then allow to cool and rinse the condenser with 10 ml of water (6.1).

For samples with a micro-nutrient content of ≤ 10 % transfer quantitatively into a 150 ml volumetric flask and dilute to the mark with water. The test solution corresponds to a 50 times dilution of the sample.

For samples with a micro-nutrient content of > 10 % transfer quantitatively into a 500 ml volumetric flask and dilute to the mark with water. The test solution corresponds to a 500 times dilution of the sample.

Test solutions may be filtered or centrifuged if necessary. Use ash-free filter paper of recognized and tested quality (7.3) and discard the first portion of the filtrate (approximately 20 ml).

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

Carry out the measurement immediately or store the extracts in tightly closed plastic vessels for up to 15 d.

If boron is not to be determined standard glassware may be used. Glass condensers may be used for all elements including boron. If only glassware is available it may be used for boron determination only if the concentration of boron in blank samples is negligible.

Procedure using a test portion of (2 ± 0,002) g: 14 ml of hydrochloric acid (6.2), 4,7 ml of nitric acid (6.3) and 100 ml volumetric flask may be used alternatively if 150 ml volumetric flasks are not available.

The amount of added aqua regia is sufficient for oxidation of about 0,5 g organic carbon. If there is more than 0,5 g of organic carbon add 1 ml of concentrated nitric acid (6.3) to every 0,1 g of organic carbon above 0,5 g, but not more than 10 ml of nitric acid.

Addition of one drop of octanol to the reaction vessel may be used as an antifoaming agent.

Erlenmeyer flasks or high beakers covered with watch glasses may be used as an alternative for the extraction step if the laboratory proves that there were no loses of the elements to be determined.

9 Precision

The precision of the extraction method specified in this document has been determined in an inter-laboratory test based on the measurement methods specified in EN 16963 and EN 16965. The statistical data obtained by ICP-AES measurement are given in EN 16963. The statistical data obtained by FAAS measurement are given in EN 16965.

10 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, EN 16964;

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

d) date when the extraction was finished;

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