

VÄETISED. VÄETISTES OLEVA BOORI, KOOBALTI, VASE,
RAUA, MANGAANI, MOLÜBDEENI JA TSINGI MÄÄRAMINE
ICP-AES MEETODIGA

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16963:2018 sisaldab Euroopa standardi EN 16963:2018 ingliskeelset teksti.	This Estonian standard EVS-EN 16963:2018 consists of the English text of the European standard EN 16963:2018.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 24.01.2018.	Date of Availability of the European standard is 24.01.2018.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 65.080

Standardite reproduutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:
Koduleht www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:

Homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 16963

January 2018

ICS 65.080

English Version

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

Engrais - Dosage du bore, du cobalt, du cuivre, du fer,
du manganèse, du molybdène et du zinc par ICP-AES

Düngemittel - Bestimmung von Bor, Cobalt, Kupfer,
Eisen, Mangan, Molybdän und Zink mit ICP-AES

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of 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 United Kingdom.



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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents

	Page
European foreword.....	3
Introduction	4
1 Scope.....	5
2 Normative references.....	5
3 Terms and definitions	5
4 Principle	5
5 Interferences	6
5.1 General.....	6
5.2 Spectral interferences	6
5.3 Transport interferences	6
5.4 Excitation interferences	6
5.5 Chemical interferences	6
5.6 Memory interferences.....	6
6 Reagents	7
7 Apparatus.....	7
8 Procedure.....	8
8.1 Preparation of test and blank solution	8
8.2 Preparation of the calibration solutions	8
8.3 Measurement.....	8
8.3.1 Instrument conditions.....	8
8.3.2 Optimization of the instrument conditions	9
8.3.3 Interferences	9
8.3.4 Matrix effects	9
8.3.5 Spiking.....	10
9 Calculation and expression of the results	11
10 Precision.....	12
10.1 Inter-laboratory tests	12
10.2 Repeatability.....	12
10.3 Reproducibility	12
11 Test report.....	16
Annex A (informative) Statistical results of the inter-laboratory test.....	17
Bibliography.....	32

European foreword

This document (EN 16963:2018) 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 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].

This document 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. Inductively coupled plasma atomic emission spectrometry (ICP-AES) is nowadays widely used and well established in many laboratories. The European Standard can be used for determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc in all extracts prepared according to EN 16962 and EN 16964. The method can be applied to mineral fertilizers with micro-nutrient contents of $\leq 10\%$ as well as contents of $> 10\%$.

The inter-laboratory study reflects the final properties of the method for determination of individual micro-nutrients in water and aqua regia extracts including extraction steps.

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 boron, cobalt, copper, iron, manganese, molybdenum and zinc in fertilizer extracts using inductively coupled plasma-atomic emission spectrometry (ICP-AES).

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

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

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 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

The method is based on the ICP-AES measurement of the concentration of boron, cobalt, copper, iron, manganese, molybdenum and zinc in fertilizer extracts prepared according to EN 16962 or EN 16964. The elements are determined after appropriate dilution of the extract. The solution is dispersed by a nebulizer of the ICP-AES instrument and the resulting aerosol is transported into the plasma. Element specific emission spectra are produced by a radio-frequency inductively coupled argon plasma where atoms or ions are excited at high temperature. The emission spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Multi-element determinations using sequential or simultaneous optical systems and axial or radial viewing of the plasma may be used.