VÄETISED. VÄETISTES OLEVATE
KELAADIMOODUSTAJATE KROMATOGRAAFILINE
MÄÄRAMINE. OSA 2: O,O-EDDHA, O,O-EDDHMA JA
HBED ABIL KELAADITUD RAUA VÕI
KELAADIMOODUSTAJATE HULGA MÄÄRAMINE
IOONVAHETUSKROMATOGRAAFIAGA

Fertilizers - Determination of chelating agents in fertilizers by chromatography - Part 2: Determination of Fe chelated by [o,o] EDDHA, [o,o] EDDHMA and HBED, or the amount of chelating agents, by ion pair chromatography



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 13368-2:2017 sisaldab Euroopa standardi EN 13368-2:2017 ingliskeelset teksti.	This Estonian standard EVS-EN 13368-2:2017 consists of the English text of the European standard EN 13368-2:2017.
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 15.11.2017.	Date of Availability of the European standard is 15.11.2017.
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 <u>standardiosakond@evs.ee</u>.

ICS 65.080

Standardite reprodutseerimise 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

November 2017

EN 13368-2

ICS 65.080

Supersedes EN 13368-2:2012

English Version

Fertilizers - Determination of chelating agents in fertilizers by chromatography - Part 2: Determination of Fe chelated by [o,o] EDDHA, [o,o] EDDHMA and HBED, or the amount of chelating agents, by ion pair chromatography

Engrais - Détermination des agents chélatants dans les engrais par chromatographie - Partie 2 : Détermination du fer chélaté [o,o] EDDHA, [o,o] EDDHMA et HBED, ou de la quantité d'agents chélatants, par chromatographie d'appariement d'ions

Düngemittel - Bestimmung von Chelatbildnern in Düngemitteln mit Chromatographie - Teil 2: Bestimmung von Fe chelatisiertem [o,o] EDDHA, [o,o] EDDHMA und HBED, oder der Summe der Chelatbildner, mit Ionen-Paarchromatographie

This European Standard was approved by CEN on 11 September 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: Avenue Marnix 17, B-1000 Brussels

Cont	ents	Page
Furan	ean foreword	1
եսւ օր։ 1	Scope	
2	Normative references	
3	Terms and definitions	
4	Principle	
5	Interferences	
6	Reagents	
7	Apparatus	
-	Sampling and sample preparation	
8	Procedure	9
9 9.1	ProcedurePreparation of the sample solution for iron chelates	
9.1 9.2	Preparation of the sample solution for other micronutrient chelates	
9.3	Preparation of the calibration solutions	10
9.4	Chromatographic analysis	
10	Expression of the results	14
10.1	Fe in Fe-chelates	
10.1.1	Fe chelated by [o,o] EDDHA or [o,o] EDDHMA in Fe-[o,o] EDDHA or Fe-[o,o] EDDHMA	
4040	chelates	
10.1.2 10.2	Fe chelated by HBED in Fe-HBED chelates	
-	Chelating agent in fertilizers Mass fraction of the chelating agent in fertilizers containing [0,0] EDDHA or [0,0]	15
10.2.1	EDDHMA	15
10.2.2	Mass fraction of the chelating agent in fertilizers containing HBED	
11	Precision	16
11.1	Inter-laboratory test	
11.2	Repeatability	
11.3	Reproducibility	16
12	Test report	
Annex	A (informative) Statistical results of the inter-laboratory tests	19
A.1	Statistical results of the inter-laboratory test performed in 2003	19
A.1.1	Test samples	19
A.1.2	Inter-laboratory test procedure	19
A.1.3	Results and statistical interpretation	19
A.2	Statistical results of the inter-laboratory test performed in 2010	21
A.2.1	Test samples	21
A.2.2	Inter-laboratory test procedure	
A.2.3	Results and statistical interpretation	21

A.3	Statistical results of the inter-laboratory test performed in 2014 — Part A	22
A.3.1	Objective	22
A.3.2	Test samples	22
A.3.3	Inter-laboratory test procedure	22
A.3.4	Results and statistical interpretation	22
A.4	Statistical results of the inter-laboratory test performed in 2014 — Part B	24
A.4.1	Objective	24
A.4.2	Test samples	24
A.4.3	Inter-laboratory test procedure	
A.4.4	Results and statistical interpretation	25
Annex	B (informative) General procedure for the determination of the titrimetric purity of the chelating agents using a photometric automatic titrator	
	x C (informative) Complete names of chelating agents	
Biblio	graphy	28
	graphy	3

European foreword

This document (EN 13368-2:2017) 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 May 2018 and conflicting national standards shall be withdrawn at the latest by May 2018.

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

This document supersedes EN 13368-2:2012.

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

In comparison with EN 13368-2:2012, the following changes have been made:

- a) determination of the chelating agent added to the scope and to the title;
- b) a derivatization method for the determination of the chelating agent explained in Clause 4, Principle;
- c) a new option added for the preparation of the Fe-[*o*,*o*] EDDHA solution, starting from a Fe-[*o*,*o*] EDDHA standard, in 6.6;
- d) calculation of the mass fraction of the chelating agents included in Clause 10 including Formulae (3) and (4);
- e) Table 2 enlarged by the precision data concerning the 2014 inter-laboratory test;
- f) information on the type of standard used for Fe-[o,o] EDDHA samples and the possibility to report on the chelating agent contents included in Clause 12;
- g) results of the inter-laboratory test performed in 2014, part A and B respectively, added (A.3 and A.4);
- h) complete names of chelating agents in Annex C technically revised;
- i) editorially revised.

EN 13368, Fertilizers — Determination of chelating agents in fertilizers by chromatography consists of the following parts:

- Part 1: Determination of EDTA, HEEDTA and DTPA by ion chromatography
- Part 2: Determination of Fe chelated by [0,0] EDDHA, [0,0] EDDHMA and HBED, or the amount of chelating agents, by ion pair chromatography
- Part 3: Determination of [S,S]-EDDS by ion pair chromatography

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, John School Scho Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a method for the chromatographic determination of the iron chelated by each individual ortho(hydroxy)-ortho(hydroxy) isomer of the chelating agents [o,o] EDDHA, [o,o] EDDHMA and by HBED in fertilizers containing one or more of these substances, except for [o,o] EDDHMA and HBED mixes. The method allows the identification and the determination of the total concentration of water soluble iron chelates of these chelating agents. Also, after derivatization with Fe, the soluble amount of the chelating agents can be determined when other micro-nutrients, beside Fe are present in fertilizers containing [o,o] EDDHA, [o,o] EDDHMA or HBED.

This method is applicable to EC fertilizers covered by Regulation (EC) No 2003/2003 [4]. It is applicable to a mass fraction of the metal chelated of at least 0,625 %.

NOTE 1 The substances EDDHA (ethylenediamine-N,N'-di[(hydroxyphenyl)acetic acid] and EDDHMA (ethylenediamine-N,N'-di[(hydroxymethylphenyl)acetic acid] exist as several different isomeric forms. Positional isomers for the hydroxyl or methyl groups (in *ortho, meta,* and *para* positions) as well as stereo isomers (*meso* and dl-racemic forms) are known. Both *meso* and dl-racemic forms of the [*ortho,ortho*] EDDHA and [*ortho,ortho*] EDDHMA are positional isomers for the hydroxyl groups allowed by the Regulation (EC) No 2003/2003. Since *para, meta and ortho* methyl positional isomers of the EDDHMA present quite similar stability, they could be grouped: in the method here described the *para, meta* and *ortho* methyl positional isomers of the [*o,o*] EDDHMA are considered together. HBED (N,N'-bis(2-hydroxybenzyl)-ethylenediamine-N,N'-diacetic acid) does not present isomeric forms.

NOTE 2 At present, analytically pure standards only exist for [ortho,ortho] EDDHA, [ortho,ortho] EDDHMA and HBED. All other substances being unavailable as a standard, the influence of their eventual presence in the samples (with respect to the sensitivity and the selectivity of this method) has not been studied.

NOTE 3 The *meso* and the dl-racemic forms of [o,o] EDDHA and [o,o] EDDHMA can be determined separately by this method.

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 12944-1:1999, Fertilizers and liming materials and soil improvers - Vocabulary - Part 1: General terms

EN 12944-2:1999, Fertilizers and liming materials and soil improvers - Vocabulary - Part 2: Terms relating to fertilizers

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 12944-1:1999 and EN 12944-2:1999 apply.