

**Väetised. Väetistes olevate kelaadimoodustajate kromatograafiline määramine. Osa 2: o,o-EDDHA ja o,o-EDDHMA abil kelaaditud raua määramineioonvahetuskromatograafiaga**

**Fertilizers - Determination of chelating agents in fertilizers by chromatography - Part 2: Determination of Fe chelated by o,o-EDDHA, o,o-EDDHMA and HBED by ion pair chromatography**

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

See Eesti standard EVS-EN 13368-2:2012 sisaldab Euroopa standardi EN 13368-2:2012 ingliskeelset teksti.	This Estonian standard EVS-EN 13368-2:2012 consists of the English text of the European standard EN 13368-2:2012.
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.
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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 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 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 mit Ionen-Paarchromatographie

This European Standard was approved by CEN on 6 July 2012.

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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.

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## Foreword

This document (EN 13368-2:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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 13368-2:2007.

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

The following has been added to the former edition (EN 13368-2:2007) of this European Standard:

- a) new substance Fe-HBED added to the scope and to the title;
- b) requirements concerning the use of different columns in order to avoid overlapping of the *meso* isomer of Fe-[*o,o*] EDDHMA and Fe-HBED peaks added in Clause 5, Interferences;
- c) iron-nitrate solution and Fe-HBED solution added in Clause 6, Reagents;
- d) the sampling method is not part of the standard; informative reference to EN 1482-1 added;
- e) Table 1 enlarged by a column for total chelate;
- f) typical chromatogram of a commercial Fe-HBED sample added (Figure 4);
- g) Formula (2) on calculation of Fe-HBED added;
- h) Table 2 enlarged by the precision data concerning determination of Fe-HBED;
- i) A.2: Results of the inter-laboratory test performed in 2010 added;
- j) Annex C: Complete names of chelating agents technical revised;
- k) Bibliography revised.

This European Standard "*Fertilizers — Determination of chelating agents in fertilizers by ion chromatography*" consists of the following parts:

*Part 1: Determination of EDTA, HEEDTA and DTPA by ion chromatography*

*Part 2: Determination of Fe chelated by o,o-EDDHA, o,o-EDDHMA and HBED by ion pair chromatography*

According to the CEN/CENELEC Internal Regulations, the national standards organisations 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, 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. It does not determine the free form of the chelating agents.

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 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 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

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.

## 4 Principle

The iron chelates are separated and determined by isocratic ion-pair high-performance liquid chromatography. When an iron chelate (anion) is added to a polar fluid (eluent), containing a large cation, an ion pair is formed. This ion pair is retained by an apolar solid phase (stationary phase). The strength of the retention depends on the molecular size and its acidity. Then, each iron chelate presents a characteristic retention time depending on the chelating agent, and it is separated from the other substances present in the sample. The separation is carried out on a reverse phase silica column and an aqueous solution of TBA<sup>+</sup> (tetrabutylammonium) and acetonitrile as eluent. The detection is based on photometry at 280 nm.

NOTE For additional information, see [5], [6] and [7].