



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN 13368-2:2017. It supersedes BS EN 13368-2:2012, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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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.

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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of an identical text or by endorsement, at the latest by May 2018 and conflicting national standards shall be withdrawn at the latest by May 2018.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 [*o,o*] EDDHA, [*o,o*] 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, 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.