

Fertilizers - Determination of chelating agents in fertilizers by chromatography - Part 3: Determination of [S,S]-EDDS by ion pair chromatography

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 13368-3:2017 sisaldab Euroopa standardi EN 13368-3:2017 ingliskeelset teksti.	This Estonian standard EVS-EN 13368-3:2017 consists of the English text of the European standard EN 13368-3:2017.
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English Version

Fertilizers - Determination of chelating agents in fertilizers
by chromatography - Part 3: Determination of [S,S]-EDDS
by ion pair chromatography

Engrais - Détermination des agents chélatants dans les engrais par chromatographie - Partie 3: Détermination du [S,S]-EDDS par chromatographie d'appariement d'ions

Düngemittel - Bestimmung von Chelatbildnern in Düngemitteln mit Chromatographie - Teil 3: Bestimmung von [S,S]-EDDS mit Ionen-Paarchromatographie

This European Standard was approved by CEN on 25 September 2017.

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European foreword

This document (EN 13368-3:2017) 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of an identical text or by endorsement, at the latest by June 2018, and conflicting national standards shall be withdrawn at the latest by June 2018.

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

The European Standard 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 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, 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 chelating agent [S,S]-EDDS in fertilizers. The method allows the identification and the determination of the total concentration of the water-soluble fraction of this chelating agent. It does not allow distinguishing between the free form and the metal bound form of the chelating agent.

This method is applicable to EC fertilizers containing chelates of one or more of the following micro-nutrients: cobalt, copper, iron, manganese and zinc, covered by Regulation (EC) No 2003/2003 [4]. It is applicable to a mass fraction of the metal chelated of at least 0,35 %.

NOTE The substance EDDS (ethylenediamine-*N,N'*-disuccinic acid) exists as several different stereo isomeric forms. [S,S] (with CAS Number 20846-91-7), [R,R] and [R,S] optical isomers are possible. [S,S] and [R,R] are mirror images with equal chemical characteristics. However only the [S,S] isomer is biodegradable. When both are present, they form the racemic mixture. The [R,S] isomer (the meso isomer) is only slowly biodegradable.

Only the [S,S]-EDDS isomer is allowed by the Regulation (EC) No 2003/2003. Since the Cu chelate of the [R,R]-EDDS isomer presents the same stability as the [S,S]-EDDS, both should coelute. The absence of [R,S]-EDDS indicates that only a pure isomer ([S,S] or [R,R]) exists. An additional test based on polarimetry can be used to ascertain the isomeric characteristic of the sample, or the standard.

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.

4 Principle

The micronutrients associated with the [S,S]-EDDS present in an aqueous extract of the sample are replaced by Cu(II). The Cu(II) chelates are separated and determined by isocratic ion-pair high-performance liquid chromatography. When a copper 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 copper 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⁺ (tetrabutylammonium) and copper acetate and methanol as eluent. The detection is based on photometry at 254 nm.

A derivatization method should be used to form the Cu chelates, so the chelating agent is determined by