

**Fertilizers - Determination of chelating agents in fertilizers by ion chromatography - Part 1: EDTA, HEDTA and DTPA**

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## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

<p>Käesolev Eesti standard EVS-EN 13368-1:2001 sisaldab Euroopa standardi EN 13368-1:2001 ingliskeelset teksti.</p> <p>Käesolev dokument on jõustatud 18.06.2001 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.</p> <p>Standard on kättesaadav Eesti standardiorganisatsioonist.</p>	<p>This Estonian standard EVS-EN 13368-1:2001 consists of the English text of the European standard EN 13368-1:2001.</p> <p>This document is endorsed on 18.06.2001 with the notification being published in the official publication of the Estonian national standardisation organisation.</p> <p>The standard is available from Estonian standardisation organisation.</p>
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<p><b>Käsitlusala:</b> This method describes the procedure for the ion chromatographic determination of the total amount of each individual chelating agent EDTA, HEDTA and DTPA in fertilizers, containing one or more of these substances. The method allows the identification and the determination of the total water soluble fraction of each of these chelating agents. It does not allow to distinguish between the free form and the metal bound form of the chelating agents.</p>	<p><b>Scope:</b> This method describes the procedure for the ion chromatographic determination of the total amount of each individual chelating agent EDTA, HEDTA and DTPA in fertilizers, containing one or more of these substances. The method allows the identification and the determination of the total water soluble fraction of each of these chelating agents. It does not allow to distinguish between the free form and the metal bound form of the chelating agents.</p>
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**ICS** 65.080

**Võtmesõnad:** chelating agents, chromatography, determination, determination procedures, edta, fertilizers, ion-exchange chromatography, soil improving materials, testing

ICS 65.080

English version

Fertilizers

Determination of chelating agents in fertilizers  
by ion chromatography

Part 1: EDTA, HEDTA and DTPA

Engrais – Détermination des agents  
chélatants dans les engrais par  
chromatographie ionique – Partie 1:  
EDTA, HEDTA et DTPA

Düngemittel – Bestimmung von  
Chelatbildnern in Düngemitteln durch  
Ionenchromatographie – Teil 1: EDTA,  
HEDTA und DTPA

This European Standard was approved by CEN on 2001-01-01.

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 Management Centre or to any CEN member.

The European Standards exist 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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

**CEN**

European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

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## Foreword

This European Standard 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 2001, and conflicting national standards shall be withdrawn at the latest by July 2001.

EN 13368 consists of two parts dealing with the quantitative determination of chelating agents in fertilizers by ion chromatography:

- Part 1 : EDTA, HEDTA and DTPA
- Part 2 : EDDHA and EDDHMA

The annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

## 1 Scope

This method describes the procedure for the ion chromatographic determination of the total amount of each of the individual chelating agents EDTA, HEDTA, and DTPA in fertilizers containing one or more of these substances. The method allows the identification and the determination of the total water soluble fraction of each of these chelating agents. It does not allow to distinguish between the free form and the metal bound form of the chelating agents.

NOTE EDTA, HEDTA and DTPA are abbreviations used in the standard for the sake of simplicity. For complete names see annex A.

This method applies to fertilizers containing chelates of one or more of the following micro-nutrients: cobalt, copper, iron, manganese, zinc and with a mass fraction of at least 0,1 %.

## 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1482, *Sampling of solid fertilizers and liming materials*.

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696 : 1987)*.

## 3 Principle

The micro-nutrients associated with the chelating agents present in an aqueous extract of the sample are replaced by iron (III). The iron chelates are separated and determined by ion chromatography. The separation is based on anion exchange, by elution with a nitrate acetate solution. The detection is based on UV photometry at 330 nm, after post-column reaction with dilute perchloric acid.

## 4 Interferences

Several substances can interfere, to a degree largely dependent on the type of column used. With the column described in 6.2, the following phenomena have been observed.

a) Injection of solutions having high concentrations of salts can cause shifts in the retention times, mostly decreasing the retention when compared to the standard solutions. In these cases, the identity of the peaks can be confirmed by standard addition.

b) Solutions having high concentrations of salts can also create a large signal at the void volume, poorly resolved from the HEDTA peak.

c) High concentrations of nitrate, carbonate, sulfate, and phosphate do not interfere. On the other hand, large amounts of chloride create a negative fronting peak poorly resolved from the DTPA peak, and altering its peak shape.

d) Compounds, related to the group of polyamino polycarboxylic acids can interfere. While signals for EDDHA, EDDHMA, and EDDHSA are not detected, relatively weak signals are observed for NTA and CDTA. Under some conditions NTA may coelute with HEDTA or EDTA.

NOTE EDDHA, EDDHMA, EDDHSA, NTA and CDTA are abbreviations used in the standard for the sake of simplicity. For complete names see annex A.

e) No signals have been detected for the following complexing agents : citrate, oxalate, tartrate, phthalate, and 20 naturally occurring amino acids.