SLOVENSKI STANDARD
SIST-TS CEN/TS 17786-2:2023

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Anorganska gnojila z mikrohranili - Določanje vsebnosti kelatiranih mikrohranil in deleža kelatiranih mikrohranil - 2. del: Določevanje EDTA, DTPA, HEEDTA, IDHA ali EDDS

Inorganic micronutrient fertilizers - Determination of the chelated micronutrient content and the chelated fraction of micronutrients - Part 2: Determination of EDTA, DTPA, HEEDTA, IDHA or EDDS

Anorganische Spurennährstoff-Düngemittel - Bestimmung des Gehaltes an chelatisierten Spurennährstoffen und des chelatisierten Anteils an Spurennährstoffen - Teil 2: Bestimmung von EDTA, DTPA, HEEDTA, IDHA oder EDDS

Engrais inorganiques à base d'oligo-éléments - Détermination de la teneur en oligo-éléments chélatés et de la fraction chélatée des oligo-éléments - Partie 2 : Dosage de l'EDTA, du DTPA, du HEEDTA, de l'IDHA ou de l'EDDS

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TS 17786-2:2022) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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Introduction

Micronutrients are considered to be, in plant nutrition, a number of elements known to be needed in small amounts for proper plant growth and development. The most common are Iron (Fe), Manganese (Mn), Molybdenum (Mo), Copper (Cu), Zinc (Zn) and Boron (B).

If an inorganic micronutrient fertilizer contains a substance, or one of the substances in the mixture, which is intended to enhance the long term availability to plants of micronutrients in the EU fertilizing product, that substance is either a chelating agent or a complexing agent.

The chelating agents are divided into two groups[1]:

— Group 1: EDTA, DTPA, HEEDTA, IDHA and [S,S]-EDDS;

— Group 2: Chelating agents present in UVCB (unknown or variable composition, complex reaction products and biological materials) chelates including [o,o] EDDHA , [o,p] EDDHA , [o,o] EDDHMA, HBED and EDDHSA.

This document defines the test method to be used in order to measure the compliance with the chelated fraction of micronutrients in product function category (PFC) 1(C) (II) (classified according to Regulation (EU) 2019/1009 [7]) as inorganic micronutrient fertilizer containing one or more chelating agents of Group 1.

[1] Abbreviated terms are described in Annex A.

1 Scope

This document specifies a method for the determination of the chelated fraction of micronutrients for fertilizers containing one or many micronutrients chelated by EDTA, DTPA, HEEDTA, IDHA or [S,S]-EDDS in fertilizers.

This method is used for inorganic micronutrient fertilizers when micronutrients are chelated only by EDTA, DTPA, HEEDTA, IDHA or [S,S]-EDDS or for mixtures in which EDTA, DTPA, HEEDTA, IDHA or [S,S]-EDDS is one of the chelating agents.

The method is applicable to all inorganic micronutrient fertilizers containing EDTA, DTPA, HEEDTA, IDHA or [S,S]-EDDS as chelating agent for contents > 0,1 % (*w*/*w*).

The method is based on ICP or AAS measurement of the concentration of micronutrients according to EN 16963 or EN 16965 after water extraction according to EN 16962 and LC measurement of the chelating agents according to EN 15950, EN 13368-1 and EN 13368-3.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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, *Fertilizers and liming materials — Vocabulary — Part 1: General terms*

EN 12944-2, *Fertilizers and liming materials — Vocabulary — Part 2: Terms relating to fertilizers*

EN 13368-1, *Fertilizers — Determination of chelating agents in fertilizers by chromatography — Part 1: Determination of EDTA, HEEDTA and DTPA by ion chromatography*

EN 13368-3, *Fertilizers — Determination of chelating agents in fertilizers by chromatography — Part 3: Determination of [S,S]-EDDS by ion pair chromatography*

EN 15950, *Fertilizers — Determination of N-(1,2-dicarboxyethyl)-D,L-aspartic acid (Iminodisuccinic acid, IDHA) using high-performance liquid chromatography (HPLC)*

EN 16962, *Fertilizers — Extraction of water soluble micro-nutrients in fertilizers and removal of organic compounds from fertilizer extracts*

EN 16963, *Fertilizers — Determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc using ICP-AES*

EN 16965, *Fertilizers — Determination of cobalt, copper, iron, manganese and zinc using flame atomic absorption spectrometry (FAAS)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1 and EN 12944-2 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
chelated fraction
ChF
chelating agent content divided by the water-soluble micronutrient content

Note 1 to entry: The chelated fraction (ChF) is expressed as a percentage.

4 Principle

The principle of the method is to determine the content of micronutrients and the content of one or more chelating agents declared in the fertilizer followed by calculating the chelated fraction.

The content of micronutrients is determined using ICP-AES (inductive coupled plasma atomic emission spectrometry) or FAAS (flame atomic absorption spectrometry) methods using EN 16963 or EN 16965 standards after previous extraction using the EN 16962 method.

The content of the declared chelating agent is determined using one of the following methods:

— for EDTA, DTPA and HEEDTA, EN 13368-1 shall be used;

— for [S,S]-EDDS, EN 13368-3 shall be used;

— for IDHA, EN 15950 shall be used.

The chelated fraction is calculated by comparing the water soluble micronutrient content with the chelating agent content.

5 Interferences

Other chelating agents such as DTPA, [o,o] EDDHA or [o,p] EDDHA do not interfere the determination of IDHA. EDTA may interfere with the determination of IDHA with some equipment, especially with certain columns in the HPLC-equipment.

In case of [S,S]-EDDS no interferences have been detected. Metal chelates with [o,o] EDDHA, [o,o] EDDHMA, HBED, EDDHSA, EDTA, DTPA, HEEDTA, IDHA, [o,p] EDDHA, lignosulfonates and heptagluconates as well as the chelating agents do not interfere since after Cu derivatization they are separate from Cu-[S,S]-EDDS. Since retention times depend on the column type, interferences should be checked if a mixture with other chelating or complexing agents is suspected.

6 Reagents

6.1 Water

All water used should conform to EN ISO 3696 and be degassed.

6.2 Reagents of recognized analytical grade.

Those included in the following standards shall be used:

— EN 16962, when extracting boron, cobalt, copper, iron, manganese, molybdenum and zinc;

— EN 16963, when determining the content of boron, cobalt, copper, iron, manganese, molybdenum and zinc by ICP-AES;

— EN 16965, when determining the content of cobalt, copper, iron, manganese and zinc by FAAS;

— EN 13368-1, when determining the content of EDTA, HEEDTA and DTPA;

— EN 13368-3, when determining the content of [S,S]-EDDS;

— EN 15950, when determining the content of IDHA.

7 Apparatus

The usual laboratory apparatus and, in particular, the following:

7.1 Rotary shaker, capable of operation at a rotational speed of about 35 min^{-1} to 45 min^{-1}.

7.2 Inductive coupled plasma-atomic emission spectrometer, with axial or radial viewing of the plasma and with suitable background correction.

7.3 Atomic absorption spectrometer, equipped with hollow cathode lamp or other suitable light emission source, background correction and burner.

7.4 Chromatograph, equipped with pump, injection valve and injection loop. The columns included in the following standards shall we used:

— EN 13368-1 when determining the content of EDTA, HEEDTA and DTPA;

— EN 13368-3 when determining the content of [S,S]-EDDS;

— EN 15950 when determining the content of IDHA.

7.5 Membrane filters, micro membrane filters, resistant to aqueous solutions, with porosity of respectively 0,45 µm and 0,2 µm**.**

8 Sampling and sample preparation

Sampling and sample preparation are not part of the method specified in this document. A recommended sampling method is given in EN 1482-1 and a recommended sample preparation method is given in EN 1482-2.

For the size reduction of samples with a high amount of chelating agents, it is not recommended to use a high speed laboratory mill. It is more convenient to grind the sample to a particle size less than 1 mm with a mortar. Special care shall be taken with NPK samples due to their high hygroscopicity.

9 Procedure

9.1 General

For the determination of the micronutrient content and the content of the chelating agents, proceed in accordance with the procedures included in the standards mentioned below.

9.2 Water-soluble micronutrient content

Perform the extraction of the water-soluble micronutrients according to EN 16962 and determine the content of the water soluble micronutrients according to EN 16963 or EN 16965.

9.3 Chelating agent content

Determine the content of the chelating agent(s) by one or more of the following standards: EN 13368-1; EN 13368-3; EN 15950. Perform the analysis according to the procedures included in these standards.

NOTE To determine the total content of chelating agents, the adequate standard to determine the chelating agents can be based on, yet not limited to, the declared chelating agents.

10 Expression of results

10.1 Water-soluble micronutrient content

The water-soluble micronutrient concentration obtained from EN 16963 or EN 16965 is presented directly as mass percentage, w_x (w/w) in percent, because of the methods.

In order to obtain the chelated fraction (ChF) described in 10.3, the mass percentage w_x (w/w) should be transformed in mmol per 100 g by Formula (1):

$$c_x = \frac{w_x}{M_x} \times 1\,000 \quad (1)$$

where

c_x is the concentration of water-soluble micronutrient, in mmol per 100 g;

w_x is the mass fraction of water-soluble micronutrient, in g per 100 g;

M_x is the molar mass of the water-soluble micronutrient, in g per mol.

NOTE For Fe: 55,8 g/mol; for Mn: 54,9 g/mol; for Zn: 65,4 g/mol; for Cu: 63,5 g/mol.

In the case of a mixture of micronutrients calculate the sum of all the micronutrients expressed in mmol per 100 g.

10.2 Chelating agent content

The mass fraction of the chelating agents EDTA, HEEDTA and DTPA is obtained from EN 13368-1 the mass fraction of [S,S]-EDDS is obtained from EN 13368-3 and the mass fraction of IDHA is obtained from EN 15950. The result is presented as w_L in mass percentage, expressed as free acid.

In order to obtain the chelated fraction (ChF) described in 10.3, the mass percentage w_L (w/w) should be transformed in mmol per 100 g as given by Formula (2):

$$c_{LCh} = \frac{w_L}{M_L} \times 1\,000 \quad (2)$$

where

c_{LCh} is the content of the chelating agent, expressed as free acid, in mmol per 100 g;

w_L is the mass fraction of the chelating agent, expressed as free acid, in g per 100 g;

M_L is the molar mass, in g/mol, of the chelating agent, expressed as free acid.

NOTE For EDTA: 292,0 g/mol, HEEDTA: 278,0 g/mol, DTPA: 393,0 g/mol [S,S]-EDDS: 292,2 g/mol and for IDHA 249,1 g/mol.

The chelating agent content ($C_{L,total}$) in mmol/100 g is the sum of the content of EDTA, HEEDTA, DTPA, [S,S]-EDDS and IDHA, expressed as free acid, in mmol per 100 g.