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Inorganic micronutrient fertilizers - Determination of the concentration of free, chelated or complexed micronutrients and the chelating and/or complexing agents present in compound inorganic micronutrient fertilizers

Engrais inorganiques - Détermination de la concentration en oligo-éléments libres, chélatés ou complexés et des agents chélatants et/ou complexants présents dans les engrais inorganiques composés à base d'oligo-éléments

Anorganische Spurennährstoffdüngemittel - Bestimmung der Konzentration freier, chelatisierter oder komplexgebundener Spurennährstoffe sowie der Chelatbildner und/oder Komplexbildner in einem anorganischen Mehrnährstoff-Spurennährstoffdüngemittel

This Technical Specification (CEN/TS) was approved by CEN on 13 March 2022 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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European foreword

This document (CEN/TS 17764:2022) 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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CEN/TS 17764:2022 (E)**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 products, that substance is either a chelating agent or a complexing agent.

In this document the test method is defined to be used in order to determine free, chelated or complexed micronutrients and chelating and/or complexing agents present in compound inorganic micronutrient fertilizers (classified as product function category (PFC) 1(C)(II)(b) according to Regulation (EU) 2019/1009 [7]).

This method allows the determination of the content of Co, Cu, Fe, Mn, Zn as free and/or chelated and/or complexed micronutrients.

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1 Scope

This document specifies the method for the determination of free, chelated or complexed micronutrients and chelating and/or complexing agents present in compound inorganic micronutrient fertilizers.

This method applies to compound inorganic micronutrient fertilizers when micronutrients are chelated and/or complexed.

The method is based on the determination of the following specific parameters¹:

- the water-soluble micronutrient concentration;
- the fraction of chelated micronutrients in relation;
- identification of chelating agents EDTA, DTPA, HEEDTA, IDHA, [S,S]-EDDS, [o,o] EDDHA, [o,o] EDDHMA, [o,p] EDDHA, HBED and EDDHSA;
- the fraction of complexed micronutrients;
- identification of complexing agents (lignosulfonates, heptagluconic acid (HGA)).

The method is based on

- ICP (inductive coupled plasma) or FAAS (flame atomic absorption spectrometry) measurement of the concentration of water-soluble micronutrients according to EN 16963 or EN 16965 after extraction according to EN 16962;
- LC (liquid chromatography) measurement of the chelating agents according to EN 15950, EN 13368-1, EN 13368-2, EN 13368-3, EN 15451, EN 15452; and/or complexing agents according to EN 16109 and EN 16847;
- determination of the concentration of chelated micronutrients by CEN/TS 17786-1 and/or CEN/TS 17786-2;
- determination of the complexed micronutrients by EN 15962.

To avoid duplication of the analytical methods, CEN/TS 17786-2 describes the determination of micronutrients and the identification and determination of chelating agents.

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*

¹ Abbreviated terms are described in Annex A.

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EN 13368-2, *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*

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

EN 15451, *Fertilizers — Determination of chelating agents — Determination of iron chelated by EDDHSA by ion pair chromatography*

EN 15452, *Fertilizers — Determination of chelating agents — Determination of iron chelated by o,p-EDDHA by reversed phase HPLC*

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

EN 15962, *Fertilizers — Determination of the complexed micro-nutrient content and of the complexed fraction of micro-nutrients*

EN 16109, *Fertilizers — Determination of complexed micro-nutrient ions in fertilizers — Identification of lignosulfonates*

EN 16847, *Fertilizers — Determination of complexing agents in fertilizers — Identification of heptagluconic acid by chromatography*

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)*

CEN/TS 17786-1, *Inorganic micronutrient fertilizers — Determination of the chelated micronutrient content and the chelated fraction of micronutrients — Part 1: Treatment with a cation exchange resin*

CEN/TS 17786-2, *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*

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:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1

free micronutrient concentration

difference between the water-soluble micronutrient concentration and the sum of the chelating and complexing agents concentration

4 Principle

The principle of the method is to determine the concentration of micronutrients and the chelating and/or complexing agents present in compound inorganic micronutrient fertilizer.

The content of the water-soluble micronutrient is determined using ICP or FAAS methods using EN 16963 or EN 16965 standards after previous extraction using EN 16962.

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

- EN 13368-1 for EDTA, DTPA and HEEDTA;
- EN 13368-2 for [o,o] EDDHA, [o,o] EDDHMA, [o,p] EDDHMA and HBED;
- EN 13368-3 for [S,S]-EDDS;
- EN 15451 for EDDHSA;
- EN 15452 for [o,p] EDDHA;
- EN 15950 for IDHA.

The chelated fraction of Fe in compound inorganic micronutrient fertilizers with Fe-UVCB (substances of unknown or variable composition, complex reaction products and biological materials) is determined by CEN/TS 17786-1.

The chelated fraction of compound inorganic micronutrient fertilizers containing EDTA, HEEDTA, DTPA, IDHA, [S,S]-EDDS, is calculated according to CEN/TS 17786-2 by comparing the water-soluble micronutrient content with the content of all chelating agents.

The content of complexing agents is determined using one of the following methods:

- EN 16847 for HGA;
- EN 16109 for lignosulfonates.

The complexed fraction is determined by EN 15962.

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5 Interferences

Other chelating agents such as DTPA, [o,o]-EDDHA or [o,p]-EDDHA do not interfere the determination of IDHA. EDTA can 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. Micronutrient chelates with [o,o] EDDHA, [o,o] EDDHMA, HBED, EDDHSA, EDTA, DTPA, HEEDTA, IDHA, [o,p] EDDHA, lignosulfonates and heptagluconic acid (HGA) 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

All reagents shall be of recognized analytical grade. The reagents included in the following standards shall be used:

- CEN/TS 17786-2 when determining the chelated fraction of compound inorganic micronutrient fertilizers containing EDTA, HEEDTA, DTPA, IDHA, [S,S]-EDDS;
- EN 13368-1 when determining the content of EDTA, HEEDTA and DTPA;
- EN 13368-2 when determining the content of [o,o] EDDHA, [o,o] EDDHMA, [o,p] EDDHMA and HBED;
- EN 13368-3 when determining the content of [S,S]-EDDS;
- EN 15451 when determining the content of EDDHSA;
- EN 15452 when determining the content of [o,p] EDDHA;
- EN 15950 when determining the content of IDHA;
- EN 15962 when determining the complexed fraction;
- EN 16109 when determining the content of lignosulfonates;
- EN 16847 when determining the content of HGA;
- 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.