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Organo-mineral fertilizers - Identification of chelating agents - Part 1: Determination of EDTA, HEEDTA and DTPA by ion chromatography

Engrais organo-minéraux - Identification des agents chélatants - Partie 1 : Détermination de l'EDTA, de l'HEEDTA et du DTPA par chromatographie ionique Organisch-mineralische Düngemittel - Identifizierung von Chelatbildnern - Teil 1: Bestimmung von EDTA, HEEDTA und DTPA mittels Ionenchromatographie

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European foreword

This document (CEN/TS 17789-1:2022) 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 organo-mineral 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 groups¹:

- Group 1: EDTA, DTPA, HEEDTA, IDHA and [S,S]-EDDS;
- Group 2: Chelating agents present in UVCB (unknown or variable composition, complex reaction products or biological materials) chelates including [0,0] EDDHA, [0,p] EDDHA, [0,0] EDDHMA, HBED and EDDHSA.

This document specifies the test method to be used in order to determine the content of certain chelating agents (EDTA, DTPA and HEEDTA) in organo-mineral fertilizers (product function category (PFC) 1(B) Preview Senerales of Files according to Regulation (EU) 2019/1009 [4]).

¹ Abbreviated terms are described in Annex A.

1 Scope

This document specifies a method for the determination by ion chromatography of the total amount of each of the individual chelating agents EDTA, HEEDTA, and DTPA in organo-mineral fertilizers, having an organic matrix based on vegetal residues (cocoa shells, grape residue, soybean residue, ...), algae extract, and animal meal (feather, bones, blood, ...) and 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.

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

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

3 Terms and definitions

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

4 Principle

The micronutrients 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 diluted perchloric acid.

5 Interferences

Several substances can interfere, to a degree largely dependent on the type of column used. With the column described in 7.2, 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 HEEDTA peak.
- c) High concentrations of nitrate, carbonate, sulfate, and phosphate do not interfere. However, large amounts of chloride create a negative fronting peak poorly resolved from the DTPA peak and altering its peak shape.