
Anorganska gnojila z mikrohranili - Določanje vsebnosti kelatiranih mikrohranil in deleža kelatiranih mikrohranil - 1. del: Obdelava s kationsko izmenjevalno smolo

Inorganic micronutrient fertilizers - Determination of the chelated micronutrient content and the chelated fraction of micronutrients - Part 1: Treatment with a cation exchange resin

iTeh STANDARD PREVIEW

Anorganische Spurennährstoff-Düngemittel - Bestimmung des Gehaltes an chelatisierten Spurennährstoffen und des chelatisierten Anteils an Spurennährstoffen - Teil 1: Behandlung mit einem Kationenaustauscherharz

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Engrais inorganiques à base d'oligo-éléments - Détermination de la teneur en oligo-éléments chélatés et de la fraction chélatée des oligo-éléments - Partie 1 : Traitement avec une résine échangeuse de cations

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Inorganic micronutrient fertilizers - Determination of the
chelated micronutrient content and the chelated fraction
of micronutrients - Part 1: Treatment with a cation
exchange resin

Engrais inorganiques à base d'oligo-éléments -
Détermination de la teneur en oligo-éléments chélatés
et de la fraction chélatée des oligo-éléments - Partie 1 :
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Anorganische Spurennährstoffdüngemittel -
Bestimmung des Gehaltes an chelatisierten
Spurennährstoffen und des chelatisierten Anteils an
Spurennährstoffen - Teil 1: Behandlung mit einem
Kationenaustauscherharz

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European foreword

This document (CEN/TS 17786-1:2022) 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 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 groups¹:

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

This document defines the test method to be used in order to measure the compliance with the chelated fraction of iron in product function category (PFC) 1(C) (II)(a) (classified according to Regulation (EU) 2019/1009 [9]) for fertilizers containing one or more chelating agents of Group 2.

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¹ Abbreviated terms are described in Annex A.

1 Scope

This document specifies a method for the determination of the chelated iron content and the chelated fraction of iron, in UVCB chelates, EDDHA, EDDHMA, HBED, EDDHSA, in inorganic micronutrient fertilizers by the treatment with a cation exchange resin.

The limit of determination of the chelated iron content highly depends on the specific electrical conductivity of the sample, on the amount of nutrient present, and varies between 0,005 % in simple matrices with high amounts of micronutrient and 0,5 % in more complex cases (see 9.1).

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 and soil improvers — Vocabulary — Part 1: General terms*

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

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 of a micronutrient

ratio of the chelated micronutrient content and the water-soluble micronutrient content

Note 1 to entry: The chelated fraction of a micronutrient is expressed as a percentage.

4 Principle

The sample is extracted with water and the extract adjusted to a neutral pH. The chelated forms of an element thus having a negative and/or neutral charge are not retained by an ion exchange resin of the strong sulfonated cationic type, and are separated from the non-chelated forms, having a cationic nature. The chelated forms are collected and their content determined by spectrometry, as well as the water soluble content.

5 Interferences

Any substance combining with a micronutrient to form a stable, negative or uncharged compound (chelate or complex) at neutral pH, will prevent the retention by the resin, and account for a certain degree of chelation. This is the case for many complexing agents, e.g. amino acids, citrate, and for chelating agents other than EDTA, HEEDTA, DTPA, [o,o] EDDHA and [o,o] EDDHMA.

In order to determine the presence of the statutory chelating agents in the sample, EN 13368-1 and EN 13368-2 should be applied.

In some cases, especially where complex fertilizer matrices with high amounts of phosphate are handled, or where micronutrients with a low chelated fraction are present, slow precipitation reactions can occur during the contact with the resin, causing equilibrium shifts, adsorption, decreasing the exchange capacity and leading to inaccurate and imprecise results. It is advised to proceed as soon as possible with all steps. Highly unstable solutions cannot be considered.

6 Reagents

All reagents shall be of recognized analytical grade.

6.1 Water

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

6.2 Sulfonated cationic exchange resin

6.2.1 General

Polystyrene divinylbenzene (PS-DVB) copolymer, slightly crosslinked (mass fraction of DVB less than or equal to 8 %), in sodic or protonic form, free from Co, Cu, Fe, Mn, and Zn².

6.2.2 Preparation and determination of cationic exchange capacity

Protonate the resin prior to use, transform to the sodic form, to eliminate any contamination and measure the cationic exchange capacity (CEC) of the sodic resin in wet form as follows:

Transfer 50 g of resin to a 500 ml beaker and add 250 ml of hydrochloric acid solution (6.4).

Place on a magnetic stirrer. After one hour of moderate stirring, the suspension is transferred onto the sieve (7.1). The resin is recovered and transferred back to the beaker. The acidification and separation operations are repeated as described. At the end of the second operation, the protonated resin present on the sieve is washed thoroughly with water until the rinsing water is free from chloride when tested with silver nitrate.

² Dowex® 50 x 4-400, Amberlite® IR 120 or equivalent are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of these products.

Transfer the wet protonated resin into a 500 ml beaker and add 250 ml of sodium chloride solution (6.5). While stirring on a magnetic stirrer and using a pH meter, titrate the resin using the sodium hydroxide solution (6.6) until a stable pH of 7,0 is obtained.

Transfer quantitatively the resin which is now in sodic form onto the sieve (7.1). Rinse thoroughly with water until the rinsing water is free from chloride (silver nitrate test). Once rinsing is complete, allow the resin to drain. Weigh the drained wet resin to within 0,01 g. The wet sodic resin can be stored in a stoppered opaque flask at ambient temperature for two years.

The cationic exchange capacity of the resin is given by Formula (1):

$$Z = 2 \times \frac{V_0}{m} \quad (1)$$

where

Z is the cationic exchange capacity (CEC), in mmol/g of wet resin;

V_0 is the required volume of NaOH (6.6), needed for obtaining a stable pH 7, in ml;

m is the mass of the drained wet resin, in g.

6.3 Hydrochloric acid 1:1, substance concentration $c(\text{HCl}) \approx 6 \text{ mol/l}$

Hydrochloric acid, diluted 1 + 1 with water.

6.4 Hydrochloric acid solution, $c(\text{HCl}) = 1 \text{ mol/l}$

Dilute 165 ml of hydrochloric acid 1:1 (6.3) to 1 000 ml.

6.5 Sodium chloride solution, $c(\text{NaCl}) = 1 \text{ mol/l}$

Dissolve 58,4 g of NaCl in water and dilute to 1 000 ml.

6.6 Sodium hydroxide solution, $c(\text{NaOH}) = 2 \text{ mol/l}$

Carefully dissolve 80,0 g of NaOH in water and dilute to 1 000 ml.

6.7 Sodium hydroxide solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$

Dilute 25 ml of the sodium hydroxide solution (6.6) to 500 ml.

6.8 Nitric acid solution, $c(\text{HNO}_3) = 0,1 \text{ mol/l}$

Carefully dilute 6,9 ml of nitric acid (65 % HNO_3 , $\rho = 1,40 \text{ g/ml}$) to 1 000 ml.

6.9 Sodium hydroxide solution, $c(\text{NaOH}) = 0,01 \text{ mol/l}$

Dilute 50 ml of the sodium hydroxide solution (6.7) to 500 ml.

6.10 Nitric acid solution, $c(\text{HNO}_3) = 0,01 \text{ mol/l}$

Dilute 50 ml of the nitric acid solution (6.8) to 500 ml.

7 Apparatus

All glassware, filters, and equipment parts coming in contact with samples and solutions shall be appropriate for micronutrient analysis and be very clean and free from contamination, especially by the element Fe.

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Usual laboratory equipment and, in particular, the following:

7.1 Sieve

Sieve having a plastic body and a nylon mesh of aperture size less than the minimum diameter of the resin particles. The mass of the dry sieve shall be determined to within 0,01 g.

7.2 Tumbling shaker

Tumbling or rotary shaker operating at a rotational speed between 30 min⁻¹ and 40 min⁻¹, at a temperature of 18 °C to 22 °C.

7.3 Conductivity meter

Equipped with a conductivity cell and a temperature controller. The cell shall be rinsed and dried before immersion, and calibrated with a 0,01 mol/l potassium chloride solution, having a specific conductivity at 20 °C of 1,28 mS/cm.

7.4 Shaking flasks

Polyethylene flasks each having a capacity of 50 ml and a stopper.

7.5 Membrane filter

Micromembrane filters resistant to aqueous solutions, with porosity of 0,45 µm.

7.6 pH-meter

pH-meter, equipped with a glass electrode; the system shall be calibrated with pH 7 and pH 10 calibration buffers.

7.7 Graduated flasks, capacity 500 ml, 250 ml and 100 ml volumetric flasks.**8 Sampling and sample preparation**

Sampling and sample preparation are not part of the method specified in this document. Recommended sampling methods are given in EN 1482-1 and, for sample preparation, in EN 1482-2.

For the size reduction of samples with a high amount of chelating agent, 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.

9 Procedure**9.1 Extraction of the sample**

Weigh an amount of sample, depending on the declared content of water soluble micronutrient, to within 1 mg, into a volumetric flask of 250 ml or 500 ml, according to Table 1:

Table 1 — Sample mass/volume ratios

Micronutrient content [%]	Mass of sample <i>E</i> [g]	Volume of the extract <i>V</i> [ml]
< 0,01 %	10	250
0,01 % to <5 %	5	500
≥ 5 %	2	500