



SLOVENSKI STANDARD
SIST-TS CEN/TS 17783:2023

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Sredstva za gnojenje - Določanje stabilnosti sredstev za gnojenje, ki vsebujejo komplekse mikrohranil

Fertilizing products - Determination of the stability of fertilizing products containing micronutrient complexes

Düngeprodukte - Bestimmung der Stabilität von Spurennährstoffkomplexen in Düngeprodukten

Fertilisants - Détermination de la stabilité des fertilisants contenant des oligo-éléments complexés

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ICS:

65.080 Gnojila Fertilizers

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English Version

**Fertilizing products - Determination of the stability of
fertilizing products containing micronutrient complexes**

Fertilisants - Détermination de la stabilité des
fertilisants contenant des oligo-éléments complexes

Düngeprodukte - Bestimmung der Stabilität von
Spurennährstoffkomplexen in Düngeprodukten

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TS 17783:2022) 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 17783:2022 (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 micronutrient metals are Iron (Fe), Manganese (Mn), Cobalt (Co), Copper (Cu) and Zinc (Zn).

An EU fertilizing product shall consist solely of component materials complying with the requirements for one or more of the component material categories (CMCs). CMC 1 corresponds with the virgin material substances and mixtures.

If a micronutrient fertilizing product 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. According to Regulation (EU) 2019/1009 [2], EU fertilizing products containing complexed micronutrients shall remain stable in water solution at pH 6 and pH 7 for at least one day.

This document defines a test method to check the micronutrient complex stability in solution at pH 6 and pH 7.

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1 Scope

This document specifies a method for the determination of the soluble metal that remains in solution after the application of a solution of the fertilizer substance containing micronutrient complexes in water and adjusting the pH to 6 and pH 7 for at least one day.

The method applies to fertilizing products containing micronutrient complexes.

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

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*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

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/>

CEN/TS 17783:2022 (E)

3.1

CSt6**micronutrient complex stability in water at pH 6**

micronutrient complex present in a fertilizer product that is soluble in water after one day of interaction at pH 6, divided by its complexed micronutrient content

Note 1 to entry: An acceptable stability may be considered if at least 80 % of the complexed micronutrient remains in solution at pH 6 after one day.

Note 2 to entry: *CSt6* is expressed as a percentage.

3.2

CSt7**micronutrient complex stability in water at pH 7**

micronutrient complex present in a fertilizer product that is soluble in water after one day of interaction at pH 7, divided by its complexed micronutrient content

Note 1 to entry: An acceptable stability may be considered if at least 80 % of the complexed micronutrient remains in solution at pH 7 after one day.

Note 2 to entry: *CSt7* is expressed as a percentage.

4 Principle

The sample is dissolved in water and then let to interact for one day in water at a specific pH, in particular at pH 6 and pH 7. After filtration, the amount of each metal remaining in solution, determined as in EN 16963 or EN 16965, deducting the chelated amount if any, is obtained. This value is compared with the value of the complexed metal extracted by EN 16962.

5 Interferences

Any substance (chelating or complexing agent) combining with a micronutrient to form a stable compound at the specific working pH may account for a certain degree of *CSt6* or *CSt7*. This is the case for many chelates that may be present in the fertilizer substance.

6 Reagents

All reagents shall be of recognized analytical grade.

6.1 Water.

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

6.2 Potassium hydroxide solution, substance concentration $c(\text{KOH}) = 1,0 \text{ mol/l}$.

Carefully dissolve 28,05 g of KOH in water (6.1) in a 500 ml volumetric flask and make up the volume.

The incorporation of CO_2 from the atmosphere should be carefully avoided.

6.3 Potassium hydroxide solution, $c(\text{KOH}) = 0,1 \text{ mol/l}$.

Dilute 50 ml of the potassium hydroxide solution (6.2) in water (6.1) in a 500 ml volumetric flask and make up to volume.

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

Carefully dilute 6,9 ml of nitric acid (65 % HNO_3 , mass concentration $\rho = 1,40 \text{ g/ml}$) in water (6.1) in a 1 000 ml volumetric flask and make up to volume.

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

Dilute 50 ml of the nitric acid solution (6.4) in water (6.1) in a 500 ml volumetric flask and make up to volume.

6.6 Buffer solution, $c(\text{HEPES}) = 0,010 \text{ mol/l} + c(\text{MES}) = 0,010 \text{ mol/l} + c(\text{KOH}) = 0,010 \text{ mol/l}$.

Dissolve 0,238 g of HEPES (4-(2-Hydroxyethyl)piperazine-1-ethanesulfonic acid, CAS 7365-45-9) and 0,195 g of MES (2-(N-morpholino)ethanesulfonic acid, CAS 4432-31-9) in water (6.1), in a beaker, add 1,00 ml of potassium hydroxide solution (6.2) and transfer quantitatively to a 100 ml volumetric flask and make up to volume.

7 Apparatus

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

Usual laboratory equipment and, in particular, the following:

7.1 Graduated flasks, capacity 1 000 ml, 500 ml, 250 ml and 100 ml volumetric flasks.**7.2 Shaker**, horizontal eccentric shaker.**7.3 Thermostatic bath or chamber**

Incubator or thermostatic bath able to maintain $25 \pm 1 \text{ }^\circ\text{C}$.

7.4 Shaking flasks

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

7.5 pH-meter

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

7.6 Membrane filter

Micromembrane filters resistant to aqueous solutions, with porosity of $0,45 \text{ }\mu\text{m}$.

7.7 Filter paper, ash free and of recognized and tested quality.

8 Sampling and sample preparation

Sampling is not part of the method specified in this document. Recommended sampling methods are given in EN 1482-1.

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 in a mortar to a particle size less than 1 mm.

9 Procedure

9.1 Preparation of the test sample solution

Weigh an amount of sample according to Table 1, depending on the sum of the declared content of soluble metal micronutrients Fe, Mn, Cu, Zn and Co to within 1 mg, into a 500 ml Erlenmeyer flask. Add 200 ml of water (6.1). Place the flask in the shaker (7.2) and shake in the dark for 60 min. Filter the extract with filter paper (7.7), if necessary, into a 250 ml volumetric flask and make up to volume with water (6.1).

Table 1 — Sample mass and volume

Sum of soluble micronutrient content	Mass of sample m	Volume of the extract V_e	Aliquot to add to 100 ml of water V_a
[%]	[g]	[ml]	[ml]
< 2 %	5	250	20
2 % to ≤ 10 %	5	250	10
> 10 %	1	250	20

9.2 Preparation of fertilizer solution in water

Add approximately 60 ml of water (6.1) in a 100 ml beaker.

Add 1,00 ml solution of buffer solution (6.6). Adjust to the desired pH, $6,0 \pm 0,1$ (7.5), with the aid of potassium hydroxide (6.3) and/or nitric acid (6.5) solutions. Then add an aliquot of the test sample solution (9.1) as indicated in Table 1.

Stir vigorously. Adjust the pH again to the desired value with the aid of potassium hydroxide (6.3) and/or nitric acid solutions (6.5). Make up to volume (100 ml) in a volumetric flask with water (6.1).

Repeat the same procedure with the same components and quantities but adjusting the solution pH to $7,0 \pm 0,1$.

Fill polyethylene shaking flasks (7.4) with these solutions and keep them stand in the dark at 25 °C in the incubator (7.3) for 24 h. After this time, filter an aliquot of each solution using 0,45 µm membrane filters (7.6).

9.3 Spectrometric determination

Determine the amount of the micronutrients under study in the filtrate by EN 16965 (atomic absorption spectrometry (AAS)) or by EN 16963 (inductively coupled plasma atomic emission spectrometry (ICP-AES)).

9.4 Complexed micronutrient content quantification

Determine the complexed micronutrient content ($C_{(i)}$) in the fertilizing product as indicated in EN 15962.