SLOVENSKI STANDARD
SIST-TS CEN/TS 17782:2023

01-februar-2023

Sredstva za gnojenje - Določanje stabilnosti sredstev za gnojenje, ki vsebujejo kelate mikrohranil pri različnih pH-vrednostih

Fertilizing products - Determination of the stability of fertilizing products containing micronutrient chelates at different pHs

Düngeprodukte - Bestimmung der Stabilität von Spurennährstoffchelaten in Düngeprodukten bei verschiedenen pH Werten

Fertilisants - Détermination de la stabilité des fertilisants contenant des oligo-éléments chélatés à différents pH

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65.080	Gnojila	Fertilizers

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TECHNICAL SPECIFICATION

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ICS 65.080

English Version

Fertilizing products - Determination of the stability of fertilizing products containing micronutrient chelates at different pHs

Fertilisants - Détermination de la stabilité des fertilisants contenant des oligo-éléments chélatés à différents pH

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (CEN/TS 17782:2022) 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 micronutrient metals are Iron (Fe), Manganese (Mn), Cobalt (Co), Copper (Cu) and Zinc (Zn).

An EU fertilizing product consists 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. The EU fertilizing products containing chelates remain stable for at least three days in a solution having any pH within the range declared as guaranteeing acceptable stability. Metal chelates can be used in agriculture directly to soils, in nutrient solution in fertirrigation or soilless culture and in solution for foliar application. Tap water is a good model for interaction with chelates since it contains calcium and magnesium that may react chemically with the metal chelates. Composition described for CIPAC (Collaborative International Pesticides Analytical Council) standard tap water D is a good representative of a tap water of medium hardness and will be used in this standard to determine the stability for at least three days of micronutrient chelates at any specific pHs.

This document defines a test method to check the chelate stability in solution, using as a model the interaction in a reference tap water similar to the CIPAC D.

1 Scope

This document specifies a method for the determination of the soluble metal that remains in solution at different pHs after the application of a solution of the fertilizer substance containing micronutrient chelates in a tap water solution used as a reference.

The method applies to fertilizing products containing chelated micronutrients.

2 Normative references

The following documents are referred 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 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/

3.1
ChSt
stability of the metal chelate
micronutrient present in a fertilizer product that is soluble in the reference tap water after three days of interaction at a specific pH, divided by its metal chelated content

Note 1 to entry: ChSt is expressed as a percentage.

4 Principle

The sample is dissolved in water and then let to interact for three days in a reference tap water at a specific pH. After filtration, the amount of each metal remaining in solution, determined in accordance with EN 16963 or EN 16965, is compared with the chelated metal extracted by CEN/TS 17786-1 or CEN/TS 17786-2.

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 ChSt. This is the case for many complexing agents, e.g. lignosulfonates, heptagluconates and for chelating agents other than those declared for 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(KOH) = 1,0 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(KOH) = 0,1 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(HNO_3) = 0,1 mol/l

Carefully dilute 6,9 ml of nitric acid (65 % HNO_3, ρ = 1,40 g/ml) in water (6.1) in a 1 000 ml volumetric flask and make up to volume.

6.5 Nitric acid solution, c(HNO_3) = 0,01 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 Stock solutions for buffered reference tap water preparation.

6.6.1 Solution A, c($CaCl_2$) = 0,054 8 mol/l.

Dissolve 4,028 g of $CaCl_2 \cdot 2H_2O$ in water (6.1) in a beaker, transfer quantitatively to a 500 ml volumetric flask and make up to volume.

6.6.2 Solution B, c($MgSO_4$) = 0,013 7 mol/l.

Dissolve 1,668 g of $MgSO_4 \cdot 7H_2O$ in water (6.1) in a beaker, transfer quantitatively to a 500 ml volumetric flask and make up to volume.

6.6.3 Solution C, *c*(NaAc) = 0,010 mol/l + c(HEPES) = 0,010 mol/l + c(MES) = 0,010 mol/l + c(AMPSO) = 0,010 mol/l + c(CAPS) = 0,010 mol/l + c(KOH) = 0,010 mol/l.

Dissolve 0,082 g of NaAc (sodium acetate, CAS 127-09-3), 0,238 g of HEPES (4-(2-Hydroxyethyl)piperazine-1-ethanesulfonic acid, CAS 7365-45-9), 0,195 g of MES (2-(N-morpholino)ethanesulfonic acid, CAS 4432-31-9), 0,227 g of AMPSO (N-(1,1-Dimethyl-2-hydroxyethyl)-3-amino-2-hydroxypropanesulfonic acid, CAS 68399-79-1) and 0,221 g of CAPS (3-(Cyclohexylamine)-1-propanesulfonic acid, CAS 1135-40-6) in water (6.1) in a beaker, add 10,0 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 ± 1 °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 µm.

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

Test samples can have one micronutrient chelated. They can be straight inorganic micronutrient fertilizers having a declared content of not more than one micronutrient, inorganic macronutrients containing one chelated micronutrient or organo-mineral fertilizers containing one chelated

micronutrient. Since the range of concentrations is very large, preparation of sample solutions should be done considering the soluble micronutrient content.

Test samples can also have several micronutrients chelated. They can be compound inorganic micronutrient fertilizers, inorganic macronutrients containing several chelated micronutrient or organo-mineral fertilizers containing several chelated micronutrients. The preparation of the sample solutions should be done considering the sum of all the soluble micronutrient content.

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 [g]	**Volume of the extract** V_e [ml]	**Aliquot to add to 100 ml reference tap water** V_a [ml]
< 2	5	250	20
2 to ≤ 10	5	250	10
> 10	1	250	20

9.2 Preparation of fertilizer solution in reference tap water

Add approximately 50 ml of water (6.1) in a 100 ml beaker.

Add, following this sequence and stirring after each addition: 5,00 ml solution A (6.6.1), 5,00 ml solution B (6.6.2) and 1,00 ml solution C (6.6.3). Adjust to the desired pH (7.5), to the nearest 0,1 unit with the aid of potassium hydroxide (6.3) and/or nitric acid solutions (6.5). 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 all the desired pHs.

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 three days. 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 emission spectrometry (ICP)).

9.4 Metal chelated content quantification

Determine the metal chelated content ($Ch(i)$) in the fertilizing product as indicated in CEN/TS 17786-1 or CEN/TS 17786-2.