SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17783:2021

01-december-2021

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

Ta slovenski standard je istoveten z: **FprCEN/TS 17783**

ICS:

65.080 Gnojila Fertilizers

kSIST-TS FprCEN/TS 17783:2021 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

FINAL DRAFT
FprCEN/TS 17783

October 2021

ICS 65.080

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 complexés

Düngeprodukte - Bestimmung der Stabilität von Spurennährstoffkomplexen in Düngeprodukten

This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 260.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Warning : This document is not a Technical Specification. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a Technical Specification.

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

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

Ref. No. FprCEN/TS 17783:2021 E

Contents

Page

European foreword

This document (FprCEN/TS 17783:2021) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This document is currently submitted to the Vote on TS.

This document has been prepared under a Standardization Request given to CEN by the European Commission and the European Free Trade Association.

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.

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

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 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)*

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

FprCEN/TS 17786-2, *Inorganic micronutrient fertilizers - Determination of the chelated micronutrient content and the chelated fraction of micronutrients - 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:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
CSt6
stability of 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 whether 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
stability of 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 whether 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 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(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, mass concentration 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 Buffer solution, c(HEPES) = 0,010 mol/l + c(MES) = 0,010 mol/l + c(KOH) = 0,010 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 ± 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

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 of 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 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 ± 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 ± 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 emission spectrometry (ICP)).

9.4 Complexed micronutrient content quantification

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