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Organsko-mineralna gnojila - Določevanje frakcije kompleksno vezanih mikrohranil

Organo-mineral fertilizers - Determination of the fraction of complexed micronutrients

Organisch-mineralische Düngemittel - Bestimmung des Anteils an komplexierten Spurennährstoffen

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Organo-mineral fertilizers - Determination of the fraction of complexed micronutrients

Organisch-mineralische Düngemittel - Bestimmung des Anteils an komplexierten Spurennährstoffen

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

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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.

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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 (FprCEN/TS 17788:2021) 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 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 can be either a chelating agent or a complexing agent.

The incorporation of complexing agents in organo-mineral fertilizers is intended to enhance the long term availability to plants of micronutrients in such EU fertilizing products.

WARNING – Users of this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and to ensure compliance with any national regulatory conditions.

IMPORTANT – It is absolutely essential that tests conducted according to this document are carried out by suitably trained staff.

1 Scope

This document specifies a general method for the determination of the micronutrients complexed by complexing agents in organo-mineral fertilizers. The method allows the determination of the total concentration of each complexed micronutrient in complexes after subtraction of the chelated micronutrients content, but it does not identify the individual complexing agents.

This procedure concerns EU organo-mineral fertilizing products which contain complexed micronutrients covered by Regulation (EU) 2019/1009 [6]. The method is applicable to a mass fraction of the metal complexed of at least 0,07 %, 0,006 % and 0,035 % of Fe, Mn and Zn respectively (see [7]). A lower limit of quantification has not been established for Cu and Co.

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 16963, *Fertilizers - Determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc using ICP-AES*

EN 16962, *Fertilizers - Extraction of water soluble micro-nutrients in fertilizers and removal of organic compounds from fertilizer extracts*

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
complexing agent
organic substance forming a flat or steric structure with one di- or tri-valent transition metal cation (zinc (Zn), copper (Cu), iron (Fe), manganese (Mn) or cobalt (Co))

4 Principle

The method is based on the precipitation of the inorganic forms at pH 9. Then the complexed forms of an element remain in solution and are separated from the non complexed forms. The complexed forms are collected and their content determined by spectrometry, as well as the soluble element content.

NOTE For additional information see [7] and [8].

5 Interferences

Any substance combining with a micro-nutrient to form a stable soluble compound (chelate or complex) at pH 9, will prevent the precipitation of the metal, and account for a certain degree of complexation. This is the case for chelating agents. If the presence of chelates is suspected the appropriate analytical method should be used (FprCEN/TS 17786-1 and FprCEN/TS 17786-2) to identify and quantify the amount of element chelated, that should be subtracted from the amount of the element given by this method in order to obtain the actual amount of element complexed.

6 Reagents

All reagents shall be of recognized analytical grade.

6.1 Water

All water used should conform to EN ISO 3696, be degassed and be free of organic contaminants.

6.2 Hydrogen peroxide, H_2O_2, 30 % to 33 %.

6.3 Sodium hydroxide solution, substance concentration c(NaOH) = 0,5 mol/l.

Carefully dissolve 20,0 g of NaOH in water and dilute to 1 l.

This solution is also available commercially.

6.4 Sodium hydroxide solution, c(NaOH) = 0,05 mol/l.

Carefully dissolve 2,0 g of NaOH in water and dilute to 1 l.

This solution is also available commercially.

6.5 Buffer solution, pH = 7,0.

6.6 Buffer solution, pH = 10,0.

7 Apparatus

Usual laboratory equipment, glassware, and the following:

7.1 Magnetic stirrer.

7.2 Balance, capable of weighing to the nearest 1 mg**.**

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

7.4 Membrane filters, micro-membrane filters resistant to aqueous solutions, with porosity of 0,45 µm.

7.5 Cellulose filters, fast filtration quantitative cellulose filters.

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 complexing agents, 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 by the use of a mortar and pestle.

9 Procedure

9.1 Preparation of the sample solution

Weigh, to the nearest 1 mg, a quantity of the fertilizer between 5 g and 10 g, depending on the declared content of the metal complex, into a 250 ml or 500 ml beaker. Add 200 ml or 400 ml of water. Stir using a magnetic stirrer (7.1) for 1 h. Transfer quantitatively into a 250 ml or 500 ml volumetric flask. Dilute to the mark with water and homogenize. The mass of the test sample and volume of extract should be taken according to Table 1.

Table 1 — Preparation of sample solution

Parameter	Declared content of complexed micronutrient in the fertilizer	
	< 0,5 %	**≥ 0,5%**
Mass of test portion (g)	10	5
Mass of element in the sample (mg)	< 50	≥ 25
Volume of the extract (ml)	250	500
Concentration of the element in the extract after precipitation (mg/l)	< 40	≥ 10

If insoluble matter is observed, filter immediately after the final volume has been reached using cellulose filters (7.5).

9.2 Precipitation

Pipette 20 ml of the solution (9.1) into a 50 ml beaker. Add two drops of H_2O_2 (6.2), stir and rise pH, with NaOH 0,5 mol/l (6.3) or 0,05 mol/l (6.4), to 9,0 as fast as possible in order to avoid reaction with atmospheric carbon dioxide. Cover the beaker. Rise pH again to 9,0 after 30 min and cover the beaker again. Let the solution stand for a minimum of 18 h and a maximum of 24 h in the dark. Readjust the pH to 9,0, transfer the sample to a 100 ml volumetric flask and dilute to the mark with water (6.1). Filtrate the solution through the membrane filter (7.4). If precipitation is observed and filtration is difficult then samples may be centrifuged at 7 500 min^{-1} at 20 °C to 25 °C for 10 min before filtration.

The acidification of the solution required for the spectrometric determination (9.3) should be made as soon as possible after the precipitation process, in order to stabilize the solution.

9.3 Spectrometric determination

Determine the micronutrient concentration in the filtrate after precipitation procedure (9.2) specified in EN 16965 (flame atomic absorption spectrometry (FAAS)) or EN 16963 (inductively coupled plasma emission spectrometry (ICP-AES)). If FAAS is used, removal of the organic compounds is required, and should be made in accordance with EN 16962.

Let $d_{(i)}$ be the micronutrient concentration of the filtrate, in milligrams per litre.

9.4 Water-soluble micronutrient content determination

Extract the water-soluble micronutrient content following method EN 16962. Determine the water-soluble micronutrient content in the sample according to EN 16965 (flame atomic absorption spectrometry (FAAS)) or to EN 16963 (inductively coupled plasma emission spectrometry (ICP-AES)). If FAAS is used, removal of the organic compounds is required, and should be made in accordance with EN 16962.

Let $S_{(i)}$ be the water-soluble micro-nutrient content in the sample, expressed as mass fraction in percent.

10 Expression of results

10.1 Complexed micronutrient content in the fertilizer

The content of a complexed micronutrient (i) in the fertilizer, $w_{(i)}$, expressed as mass fraction in percent, is given by the following Formula (1):

$$w_{(i)} = \frac{c_{(i)} \times V}{2\,000 \times m} \tag{1}$$

where

$c_{(i)}$ is the micronutrient (i) concentration of the filtrate solution, in mg/l;

m is the mass of the test portion, in g;

V is the volume of the extract in ml.

10.2 Complexed fraction of a micronutrient in the fertilizer

The complexed fraction $F_{(i)}$ of a micronutrient (i) is the ratio of the complexed micronutrient content $w_{(i)}$ to the water-soluble micronutrient content $S_{(i)}$ in the fertilizer, expressed as a percentage, and is given by the following Formula (2):

$$F_{(i)} = 100 \times \frac{w_{(i)}}{S_{(i)}} \tag{2}$$

where

$w_{(i)}$ is the complexed micro-nutrient (i) content in percent (mass fraction);

$S_{(i)}$ is the water-soluble micro-nutrient (i) content in percent (mass fraction).