SLOVENSKI STANDARD
SIST-TS CEN/TS 17060:2017

01-december-2017

Gnojila - Določevanje molibdena v koncentracijah do 10 % z gravimetrično metodo z 8-hidroksikinolinom

Fertilizers - Determination of molybdenum in concentrations > 10 % using a gravimetric method with 8-hydroxyquinoline

Düngemittel - Bestimmung von Molybdän in Konzentrationen > 10 % durch Gravimetrie mit 8-Hydroxychinolin

Engrais - Dosage du molybdène dans des concentrations > 10 % en utilisant une méthode gravimétrique à la 8-hydroxyquinoléine

Ta slovenski standard je istoveten z: **CEN/TS 17060:2017**

ICS:

65.080	Gnojila	Fertilizers

SIST-TS CEN/TS 17060:2017	**en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 17060

May 2017

ICS 65.080

English Version

Fertilizers - Determination of molybdenum in concentrations > 10 % using a gravimetric method with 8-hydroxyquinoline

Engrais - Dosage du molybdène dans des concentrations > 10 % en utilisant une méthode gravimétrique à la 8-hydroxyquinoléine

Düngemittel - Bestimmung von Molybdän in Konzentrationen > 10 % durch Gravimetrie mit 8-Hydroxychinolin

This Technical Specification (CEN/TS) was approved by CEN on 3 March 2017 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. CEN/TS 17060:2017 E

Contents

Page

European foreword

This document (CEN/TS 17060:2017) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The preparation of this document by CEN is based on a mandate by the European Commission and the European Free Trade Association (Mandate M/335), concerning the modernization of methods of analysis on fertilizers in the framework of Regulation (EC) No 2003/2003 [1].

This document is part of a modular approach and concerns the analytical measurement step. “Modular” means that a test standard concerns a specific step in assessing a property and not the whole chain of measurements.

Determination of molybdenum in fertilizers can be executed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to prEN 16963:2016. The gravimetric determination as molybdenyl oxinate is more labour intensive and skill demanding but it is an option when ICP-AES is not available.

WARNING — Persons using this European Technical Specification should be familiar with normal laboratory practice. This European Technical Specification 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 European Technical Specification are carried out by suitably trained staff.

1 Scope

This Technical Specification specifies a method for the determination of total and water extractable molybdenum in mineral fertilizers containing more than 10 % molybdenum.

This method is applicable to water and aqua regia fertilizer extracts obtained according to prEN 16962:2016 and/or prEN 16964:2016.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1482-2, *Fertilizers and liming materials - Sampling and sample preparation - Part 2: Sample preparation*

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*

prEN 16962:2016, *Fertilizers — Extraction of water soluble micro-nutrients in fertilizers and removal of organic compounds from fertilizer extracts*

prEN 16964:2016, *Fertilizers — Extraction of total micro-nutrients in fertilizers and removal of organic compounds from fertilizer extracts*

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

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1 and EN 12944-2 apply.

4 Principle

The molybdenum content is determined by precipitation as molybdenyl oxinate under specific conditions.

5 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2. The sample extracts shall be prepared in accordance with prEN 16962:2016 and/or prEN 16964:2016.

6 Reagents

All reagents shall be of recognized analytical grade and shall have negligible concentrations of molybdenum if compared to the lowest concentration of that element in the sample solution.

6.1 Water, according to EN ISO 3696 grade 2.

6.2 Sulfuric acid, H_2SO_4, ρ = 1,84 g/ml.

6.3 Sulfuric acid solution, approximately 1 mol/l.

Carefully pour 55 ml of sulfuric acid (6.2) into a 1 l volumetric flask (7.5) containing 800 ml water (6.1). Mix. After cooling, make up to 1 l. Mix again.

6.4 Concentrated ammonia solution, NH_4OH, ρ = 0,9 g/ml.

6.5 Diluted ammonia solution, volume fraction ratio = 1:3.

Mix 1 volume of concentrated ammonia solution (6.4) with 3 volumes of water (6.1).

6.6 Concentrated acetic acid, 99,7 % CH_3COOH, ρ = 1,049 g/ml.

6.7 Diluted acetic acid solution, volume fraction ratio = 1:3.

Mix 1 volume of concentrated acetic acid (6.6) with 3 volumes of water (6.1).

6.8 Solution of disodium salt of ethylenediamine tetraacetic acid (Na_2EDTA).

Dissolve 5 g of Na_2EDTA in water (6.1) in a 100 ml volumetric flask (7.4). Make up to the calibration mark and mix.

6.9 Buffer solution.

In a 100 ml volumetric flask (7.4), dissolve 15 ml of concentrated acetic acid (6.6) and 30 g of ammonium acetate in water (6.1). Make up to 100 ml.

6.10 8-Hydroxyquinoline (oxine) solution.

In a 100 ml volumetric flask (7.4), dissolve 3 g of 8-Hydroxyquinoline in 5 ml of concentrated acetic acid (6.6). Add 80 ml of water (6.1). Add the diluted ammonia solution (6.5) drop by drop until the solution becomes cloudy and then add the diluted acetic acid (6.7) until the solution becomes clear again. Make up to 100 ml with water (6.1).

7 Apparatus

7.1 Filter crucible, P16 according to ISO 4793, porosity 4, capacity 30 ml.

7.2 pH meter with glass electrode.

7.3 Drying oven, set at (130 to 135) °C.

7.4 Volumetric flasks, capacity 100 ml.

7.5 Volumetric flasks, capacity 1 l.

7.6 Beaker, capacity 250 ml.

7.7 Desiccator.

8 Procedure

8.1 Preparation of the test solution

Place an aliquot portion containing 10 mg Mo to 100 mg Mo, maximum 50 ml, in a 250 ml beaker (7.6). Make up the volume to 50 ml with water (6.1). Adjust this solution to pH 5, measured by the pH meter (7.2) by adding the sulfuric acid solution (6.3) or diluted ammonia solution (6.5) drop by drop. Add 15 ml of EDTA solution (6.8) followed by 5 ml of buffer solution (6.9). Make up to about 80 ml with water (6.1).

8.2 Precipitation

Heat the solution (8.1) slightly. Stirring constantly, add slowly the oxine solution (6.10) and continue the precipitation until formation of a deposit is no longer observed. Add further reagent until the supernatant solution turns slightly yellow. A quantity of 20 ml of oxine solution (6.10) should normally be sufficient. Continue to heat the precipitate slightly for 2 min or 3 min.

8.3 Filtration and washing

Dry the filter crucible (7.1) at (130 to 135) °C to constant mass (at least 1 h) and weigh the crucible (m_1). Filter the solution with the precipitate (8.2) through a filter crucible (7.1). Rinse the precipitate several times with 20 ml of hot water (6.1). The rinse water should gradually become colourless indicating that oxine is no longer present.

8.4 Weighing the precipitate

Dry the precipitate at (130 to 135) °C to constant mass (at least 1 h). Allow to cool in a desiccator (7.7) and then weigh (m_2).

9 Calculation and expression of the results

1 mg of molybdenyl oxinate, $MoO_2(C_9H_6ON)_2$, corresponds to 0,230 5 mg Mo.

The content of molybdenum, w_{Mo}, in the fertilizer is calculated as a mass fraction in percent according to Formula (1).

$$w_{Mo} = \frac{X \times 0,02305 \times (V \times D)}{a \times M} \quad (1)$$

where

X is the mass in mg of the molybdenyl oxinate precipitate $X = m_2 - m_1$;

V is the volume in ml of the extract solution;

a is the volume in ml of the aliquot taken from the last dilution;

D is the dilution factor of the aliquot;

M is the mass in g of the test sample.