SLOVENSKI STANDARD
SIST EN 17043:2018

01-september-2018

Gnojila - Določevanje molibdena v koncentracijah, manjših ali enakih 10 %, s spektrometrijo kompleksa z amonijevim tiocianatom

Fertilizers - Determination of molybdenum in concentrations ≤ 10 % using spectrometry of a complex with ammonium thiocyanate

Düngemittel - Bestimmung von Molybdän in Konzentrationen ≤ 10 % durch Spektrometrie eines Komplexes mit Ammoniumthiocyanat

Engrais - Dosage du molybdène dans des concentrations ≤ 10 % par spectrométrie d'un complexe avec le thiocyanate d'ammonium

Ta slovenski standard je istoveten z: **EN 17043:2018**

ICS:

65.080	Gnojila	Fertilizers

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English Version

Fertilizers - Determination of molybdenum in concentrations ≤ 10 % using spectrometry of a complex with ammonium thiocyanate

Engrais - Dosage du molybdène dans des concentrations ≤ 10 % par spectrométrie d'un complexe avec du thiocyanate d'ammonium

Düngemittel - Bestimmung von Molybdän in Konzentrationen ≤ 10 % durch Spektrometrie eines Komplexes mit Ammoniumthiocyanat

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

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European foreword

This document (EN 17043:2018) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2018, and conflicting national standards shall be withdrawn at the latest by December 2018.

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.

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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 of fertilizers in the framework of Regulation (EC) No 2003/2003 [1].

This European Standard 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 measurement.

The determination of molybdenum in fertilizers can be executed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to EN 16963. The spectrophotometric determination of the complex with ammonium thiocyanate is more labour intensive and skill demanding but it is an option when ICP-AES is not available. More attention should be paid to organic matter removal and interferences from extract colour. The procedure for removal of organic matter from the extracts is specified in EN 16962, which also describes the modification of the extraction procedure using acidification with hydrochloric acid instead of nitric acid.

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

1 Scope

This European Standard specifies a method for determination of total and water extractable molybdenum in mineral fertilizers containing less than or equal to 10 % molybdenum.

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

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*

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

EN 16964, *Fertilizers - Extraction of total micro-nutrients in fertilizers using aqua regia*

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 apply.

4 Principle

Molybdenum Mo(V) forms a complex $[MoO(SCN)_5]$ in an acid medium with SCN^- ions. The complex is extracted into *n*-butyl acetate. Interfering ions such as iron remain in the aqueous phase. The yellow-orange coloured complex is determined spectrophotometrically at 470 nm.

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 [2].

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

6 Reagents

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

6.1 Water, grade 2 according to EN ISO 3696 and free from molybdenum.

6.2 Hydrochloric acid, (HCl) 37 %, ρ = 1,18 g/ml.

6.2.1 Diluted hydrochloric acid solution, with a concentration of about 6 mol/l.

Mix one volume of hydrochloric acid (6.2) with one volume of water (6.1).

6.2.2 Diluted hydrochloric acid solution, with a concentration of about 0,5 mol/l.

Add 42 ml of hydrochloric acid (6.2) to approximately 600 ml of water (6.1) in a 1 000 ml volumetric flask (7.4). Fill to the mark with water (6.1) after cooling.

6.3 Copper solution, ρ = 70 mg/l in 1,5 mol/l hydrochloric acid.

Dissolve 275 mg of copper sulfate ($CuSO_4$ $5H_2O$) weighed to within 0,1 mg in 250 ml of the 6 mol/l hydrochloric acid solution (6.2.1) in a 1 000 ml volumetric flask (7.4). Make up to volume with water (6.1) and mix thoroughly.

6.4 Ascorbic acid solution, ρ = 50 g/l.

Dissolve 50 g of ascorbic acid ($C_6H_8O_6$) in water (6.1) in a 1 000 ml volumetric flask (7.4). Make up to volume with water (6.1), mix thoroughly and keep in a refrigerator.

6.5 *n*-butyl acetate.

6.6 Ammonium thiocyanate solution, c = 0,2 mol/l.

Dissolve 15,224 g of ammonium thiocyanate (NH_4SCN) in water (6.1) in a 1 000 ml volumetric flask (7.4). Make up to volume with water (6.1), mix thoroughly and store in a dark-coloured bottle.

6.7 Stannous chloride solution, ρ = 50 g/l in 2 mol/l hydrochloric acid.

This solution shall be perfectly clear and prepared immediately before use. Very pure stannous chloride shall be used otherwise the solution will not be clear.

For the preparation of 100 ml of solution, dissolve 5 g of ($SnCl_2$ $2H_2O$) in 35 ml of 6 mol/l hydrochloric acid solution (6.2.1). Add 10 ml of the copper solution (6.3). Make up to volume with water (6.1) and mix thoroughly.

6.7.1 Molybdenum stock solution, ρ = 1 000 mg/l.

Dissolve 1,840 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}$ $4H_2O$] weighed to within 0,1 mg in the 0,5 mol/l hydrochloric acid (6.2.2) in a 1 000 ml volumetric flask (7.4). Make up to volume with that solution and mix thoroughly.

Commercially available stock solutions with adequate specification may be used. The solution is considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer shall be considered.

6.7.2 Molybdenum intermediate solution, ρ = 100 mg/l.

Place 10 ml of the stock solution (6.7.1) in a 100 ml volumetric flask (7.3). Make up to volume with 0,5 mol/l hydrochloric acid (6.2.2) and mix thoroughly.

6.7.3 Molybdenum working solution, ρ = 10 mg/l.

Place 10 ml of the intermediate solution (6.7.2) in a 100 ml volumetric flask (7.3). Make up to volume with 0,5 mol/l hydrochloric acid (6.2.2) and mix thoroughly.

7 Apparatus

7.1 Spectrophotometer, set to a wavelength of 470 nm with a cuvette with a 10 mm optical path.

7.2 Separating funnels, capacity 200 ml or 250 ml.

7.3 Volumetric flasks, capacity 100 ml.

7.4 Volumetric flasks, capacity 1 000 ml.

7.5 Precision pipette.

7.6 Test tube.

8 Procedure

8.1 Preparation of calibration solutions

Prepare a series of at least six calibration solutions of increasing concentration corresponding to the optimum response range of the spectrometer.

Pipette 0 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of working solution (6.7.3) into 100 ml volumetric flasks (7.3), fill to the mark with diluted hydrochloric acid (6.2.2) and mix well. The calibration solutions contain mass concentrations of respectively Mo = 0 mg/l; Mo = 0,1 mg/l; Mo = 0,2 mg/l; Mo = 0,3 mg/l; Mo = 0,4 mg/l and Mo = 0,5 mg/l.

Pipette 25 ml of calibration solutions into the separating funnels (7.2) and add 25 ml of 6 mol/l hydrochloric acid (6.2.1).

8.2 Preparation of the test solutions

Dilute an aliquot portion of the aqua regia extracts prepared according to EN 16964 five times with water (6.1) to achieve final concentration of acids approximately 0,5 mol/l. The final concentration of hydrochloric acid in water extracts prepared according to EN 16962 is 0,5 mol/l. Dilute these solutions with 0,5 mol/l hydrochloric acid (6.2.2) if necessary to obtain a molybdenum concentration in the range specified in (8.1). Pipette 25 ml of the test solutions into the separating funnels (7.2) and add 25 ml of 6 mol/l hydrochloric acid (6.2.1).

Prepare a blank solution following the same procedure, omitting only the test sample of fertilizer.

8.3 Development and separation of the complex

To the separating funnels containing the calibration solutions (8.1), test solutions (8.2) and blank (8.2), add in the following order:

10 ml of the copper solution (6.3); 20 ml of the ascorbic acid solution (6.4); mix thoroughly and wait for 2 min to 3 min.

Then add 10 ml of *n*-butyl acetate (6.5), using a precision pipette (7.5); 20 ml of the thiocyanate solution (6.6). Shake for 1 min to extract the complex in the organic phase; allow to precipitate; after the separation of the two phases, draw off the entire aqueous phase and discard it.

Then wash the organic phase with 10 ml of the stannous chloride solution (6.7). Shake for 1 min. Allow to precipitate and draw off the entire aqueous phase. Collect the organic phase in a test tube (7.6); this will make it possible to collect the drops of water in suspension.