SLOVENSKI STANDARD
SIST-TS CEN/TS 17403:2021

01-april-2021

Gnojila - Določevanje v hladni in vroči vodi netopnega dušika, dobljenega s počasnim sproščanjem iz trdnih gnojil urea formaldehida in metilen uree, in določevanje topnosti polimernih hranil v raztopini fosfatnega pufra s pH 7,5 pri 100 °C

Fertilizers - Determination of cold water insoluble nitrogen and hot water insoluble nitrogen in solid urea formaldehyde and methylene urea slow-release fertilizers and determination of the solubility of nutrient polymers in phosphate buffer solution with a pH of 7,5 at 100 °C

Düngemittel - Bestimmung von kalt- und heißwasserunlöslichem Stickstoff in festen langsam freisetzenden Harnstoff-Formaldehyd- und Methylenharnstoff-Düngemitteln sowie Bestimmung der Löslichkeit von Nährstoffpolymeren in Phosphatpufferlösung mit einem pH-Wert von 7,5 bei 100 °C

Engrais - Dosage de l'azote insoluble dans l'eau froide et de l'azote insoluble dans l'eau chaude dans les engrais à libération lente urée-formaldéhyde solides etméthylène-urée, et détermination de la solubilité des polymères nutritifs dans une solution tampon phosphate avec un pH de 7,5 à 100°°C

Ta slovenski standard je istoveten z: **CEN/TS 17403:2021**

ICS:

65.080	Gnojila	Fertilizers

SIST-TS CEN/TS 17403:2021 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 17403

January 2021

ICS 65.080

English Version

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This Technical Specification (CEN/TS) was approved by CEN on 30 November 2020 for provisional application.

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

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European foreword

This document (CEN/TS 17403:2021) 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

Solid urea formaldehyde and methylene urea slow-release fertilizers are non-coated and chemically synthesized nitrogen fertilizers with slow-release effect. In 1924, the first slow-release fertilizer patent in the world was issued to urea formaldehyde (UF) and in 1955, UF was put into commercial production as the oldest slow-release fertilizer.

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 method for the determination of the cold and hot water insoluble nitrogen content in solid urea formaldehyde and methylene urea slow-release fertilizers and for the determination of the solubility of nutrient polymers in a phosphate buffer solution with a pH of 7,5 at 100 °C.

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 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*

ISO 5315, *Fertilizers — Determination of total nitrogen content — Titrimetric method after distillation*

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:

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

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

3.1
urea formaldehyde
UF
slow-release nitrogenous fertilizer obtained by the reaction between urea and formaldehyde to produce molecular chains of general formula NH_2-CO-NH$(CH_2NHCONH)_nH$

3.2
methylene urea
MU
slow-release nitrogenous fertilizer obtained by the reaction between urea and formaldehyde to produce oligomers such as MDU (methylendiurea), DMTU (dimethylentriurea), TMTU (trimethylentetraurea), TMPU (tetramethylenpentaurea) and higher counterparts

3.3
CMC 8 nutrient polymers
component material of EU fertilising products consisting of polymers exclusively made of monomer substances complying with the criteria set out in points 1 and 2 of CMC 1, where the purpose of the polymerisation is to control the release of nutrients from one or more of the monomer substances

Note 1 to entry: See [2], Annex II, Part I and Part II.

3.4
CMC 1
component material of EU fertilising products consisting of virgin material substances and mixtures

Note 1 to entry: See [2], Annex II, Part I and Part II.

Note 2 to entry: Excluded are the following products: waste and by-products according to [3], materials formerly having constituted in waste or by-products, animal by-products or derived products and polymers.

3.5
cold water insoluble nitrogen
CWIN
insoluble nitrogen fractions in urea formaldehyde or methylene urea products that are insoluble in phosphate buffer solution (pH 7,5) or distilled water at 25 °C during a 15 min period

[SOURCE: ISO 19670:2017 [4], 3.2]

3.6
cold water soluble nitrogen
CWSN
soluble nitrogen fractions in urea formaldehyde or methylene urea products that are soluble in phosphate buffer solution (pH 7,5) or distilled water at 25 °C during a 15 min period

Note 1 to entry: CWSN = Total nitrogen - CWIN.

[SOURCE: ISO 19670:2017 [4], 3.3]

3.7
hot water insoluble nitrogen
HWIN

insoluble nitrogen fractions in urea formaldehyde or methylene urea products that are insoluble in phosphate buffer solution (pH 7,5) at 100 °C during a 30 min period

[SOURCE: ISO 19670:2017 [4], 3.4]

3.8
hot water soluble nitrogen
HWSN
soluble nitrogen fractions in urea formaldehyde or methylene urea products and in nutrient polymers that are soluble in phosphate buffer solution (pH 7,5) at 100 °C during a 30 min period

Note 1 to entry: HWSN = Total nitrogen - HWIN.

[SOURCE: ISO 19670:2017 [4], 3.5, modified – “and in nutrient polymers” has been added to the definition.]

3.9
hot water soluble nitrogen only
HWSN only
soluble nitrogen fractions in urea formaldehyde or methylene urea products that are soluble in phosphate buffer solution (pH 7,5) at 100 °C during a 30 min period and insoluble in phosphate buffer solution (pH 7,5) or distilled water at 25 °C during a 15 min period

Note 1 to entry: HWSN only = HWSN – CWSN = CWIN - HWIN.

[SOURCE: ISO 19670:2017, 3.6]

4 Sampling and sample preparation

4.1 Sampling of products in bags and in bulk

Sampling is not part of the method specified in this document. Recommended sampling methods are given in EN 1482-1 [1].

4.2 Reduction of samples

Reduction, labelling and sample preparation shall be carried out in accordance with EN 1482-2.

For the size reduction of samples to be used for the determinations according to Clause 5 and Clause 6 the laboratory sample shall be reduced to 100 g. Grind this test sample in the grinder. Avoid any warming of the sample on grinding. Take the portion of the grinded sample that passed at 0,500 mm sieve and pass it through a 0,212 mm sieve; use only the portion of the grinded sample that does not pass the 0,212 mm sieve. Put the prepared samples into clean and dry bottles to be used for further analysis.

5 Determination of the mass fraction of CWIN

5.1 Principle

Extraction of the test portion in phosphate buffer solution (pH 7,5) or distilled water at 25 °C. Filtration of the insoluble residue, washing and determination of the nitrogen content in the insoluble residue.

5.2 Reagents

5.2.1 Distilled water.

5.2.2 Phosphate buffer solution, pH 7,5.

Dissolve 14,3 g KH_2PO_4 and 91,0 g K_2HPO_4 in water and dilute to 1 l. Dilute 100 ml of this solution to 1 l.

5.2.3 Anhydrous ethanol.

5.2.4 Reagents listed in ISO 5315 for the determination of the nitrogen content.

5.3 Apparatus

5.3.1 Usual laboratory apparatus.

5.3.2 Water bath, capable of being maintained at (25 ± 2) °C.

5.3.3 Quantitative filter paper (intermediate speed).

5.3.4 Apparatus listed in ISO 5315 for the determination of the nitrogen content.

5.4 Procedure

5.4.1 Two analyses shall be performed simultaneously for the determination.

5.4.2 Place 1 g to 1,4 g test portion (accurate to 0,000 1 g) in a 50 ml beaker and wet the test portion with ethanol (5.2.3). Add 20 ml phosphate buffer solution (5.2.2) or distilled water (5.2.1) and let stand for 15 min in the water bath maintained at (25 ± 2) °C (5.3.2) and stir at 5 min intervals during standing. Transfer the supernate to a piece of filter paper (5.3.3) in long-stem funnel and wash the residue four or five times by decanting with water (5.2.1) at (25 ± 2) °C. Finally, transfer all the residue to the filter paper

and complete washing with water (5.2.1) at (25 ± 2) °C until the filtrate measures 250 ml. Determine the nitrogen content in the filter paper and the residue in accordance with ISO 5315.

5.4.3 Carry out a blank test at the same time as the determination, using the same procedure, using the same reagents, but omitting the test portion.

5.5 Calculation and expression of results

The CWIN content, w_1, expressed as a mass fraction in percent, is given by Formula (1):

$$w_1 = \frac{\left(V_1 - V_2\right) \times c_1 \times 14,01}{m_1 \times 1\,000} \times 100 \quad (1)$$

where

V_1 is the volume, in millilitres, of sodium hydroxide standard solution used for the blank test;

V_2 is the volume, in millilitres, of sodium hydroxide standard solution used for the determination;

c_1 is the concentration, in mol per litres, of sodium hydroxide standard solution used;

m_1 is the mass, in grams, of the test portion.

The calculation results are accurate to two digits after the decimal point. The determination result is the arithmetic average of the duplicate determination results.

6 Determination of the mass fraction of HWIN

6.1 Principle

Extraction of the test portion in phosphate buffer solution (pH 7,5) (5.2.2) at 100 °C. Filtration of the insoluble residue, washing and determination of the nitrogen content in the insoluble residue.

6.2 Reagents

6.2.1 Reagents listed in 5.2

6.2.2 Celite

6.3 Apparatus

6.3.1 Usual laboratory apparatus

6.3.2 Water bath, capable of being maintained at (100 ± 2) °C.

6.3.3 Apparatus listed in 5.3.3 and 5.3.4

6.4 Procedure

6.4.1 Two analyses shall be performed simultaneously for the determination.

6.4.2 Place 0,5 g of the test portion (accurate to 0,000 1 g) in a 250 ml tall-form beaker. Add 100 ml of the phosphate buffer solution (5.2.2) at 100 °C using a graduated cylinder to the test portion, stir, cover with a watch glass and immerse promptly in a boiling water bath (6.3.2) so that the liquid in the beaker is below the water level in the water bath. Maintain the water bath at 100 °C, check with a thermometer and stir at 10 min intervals. After exactly 30 min, remove the beaker from the water bath and filter promptly through a piece of filter paper (5.3.3). If filtration takes more than 4 min, discard determination: