

Gnojila - Določevanje v hladni in vroči vodi netopnega dušika, dobljenega s počasnim sproščanjem iz trdnih gnojil urea formaldehida in metilen uree in iz polimerov hranil

Fertilizers - Determination of cold water insoluble nitrogen and hot water insoluble nitrogen in solid urea formaldehyde and methylene urea slow-release fertilizers and in nutrient polymers

Düngemittel - Bestimmung von kalt- und heißwasserunlöslichem Stickstoff in festen langsam freisetzenden Harnstoff-Formaldehyden und Methylenharnstoff sowie in Nährstoffpolymeren

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 solide et méthylène-urée et dans les polymères nutritif

Ta slovenski standard je istoveten z: FprCEN/TS 17403

ICS:

65.080 Gnojila Fertilizers

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FprCEN/TS 17403

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ICS 61.080

English Version

**Fertilizers - Determination of cold water insoluble
nitrogen and hot water insoluble nitrogen in solid urea
formaldehyde and methylene urea slow-release fertilizers
and in nutrient polymers**

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 solide et
méthylène-urée et dans les polymères nutritif

Düngemittel - Bestimmung von kalt- und
heißwasserunlöslichem Stickstoff in festen langsam
freisetzenden Harnstoff-Formaldehyden und
Methylenharnstoff sowie in Nährstoffpolymeren

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

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Contents	Page
European foreword.....	3
Introduction	4
1 Scope.....	5
2 Normative references.....	5
3 Terms and definitions	5
4 Determination of the mass fraction of CWIN.....	7
4.1 Principle	7
4.2 Reagents	7
4.3 Apparatus.....	7
4.4 Procedure.....	7
4.5 Calculation and expression of results.....	8
5 Determination of the mass fraction of HWIN.....	8
5.1 Principle	8
5.2 Reagents	8
5.3 Apparatus.....	8
5.4 Procedure.....	8
5.5 Calculation and expression of results.....	9
6 Sampling and sample preparation.....	9
6.1 Sampling of products in bags and in bulk	9
6.2 Reduction of samples.....	9
7 Test report.....	9
Bibliography.....	10

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European foreword

This document (FprCEN/TS 17403:2019) 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.

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FprCEN/TS 17403:2019 (E)**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.

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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 hot water soluble nitrogen content in nutrient polymers (see Component Material Category CMC 9 as specified in the Regulation (EC) No xxxx/2019 on Fertilizing Products [2]).

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 — Vocabulary — Part 1: General terms*

EN 12944-2, *Fertilizers and liming materials — Vocabulary — Part 2: Terms relating to fertilizers*

prEN ISO 14820-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

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

ISO 19670:2017, *Fertilizers and soil conditioners — Solid urea aldehyde slow release fertilizers — General requirements*

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 <http://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 $\text{NH}_2\text{-CO-NH}(\text{CH}_2\text{NHCONH})_n\text{H}$

[SOURCE: EN 12944-2:1999, 3.1.12 modified]

3.2

methylene urea

MU

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

3.3

CMC 9 nutrient polymers

component material of EU fertilizing products consisting of polymers exclusively made of monomer substances complying with the description in 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.

FprCEN/TS 17403:2019 (E)**3.4****CMC 1**

component material of EU fertilizing products consisting of virgin material substances and mixtures

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

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, 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, 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, 3.4]

3.8**hot water soluble nitrogen****HWSN**

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

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

[SOURCE: ISO 19670:2017, 3.5]

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]

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4 Determination of the mass fraction of CWIN

4.1 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.

4.2 Reagents

4.2.1 Distilled water.

4.2.2 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.

4.2.3 Anhydrous ethanol.

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

4.3 Apparatus

4.3.1 Usual laboratory apparatus.

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

4.3.3 Quantitative filter paper (intermediate speed).

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

4.4 Procedure

4.4.1 Two analyses shall be performed simultaneously for the determination.

4.4.2 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 (4.2.3). Add 20 ml phosphate buffer solution (4.2.2) or distilled water (4.2.1) and let stand for 15 min in the water bath maintained at (25 ± 2) °C (4.3.2) and stir at 5 min intervals during standing. Transfer the supernate to a piece of filter paper (4.3.3) in long-stem funnel and wash the residue four or five times by decanting with water at (25 ± 2) °C. Finally, transfer all the residue to the filter paper and complete washing until the filtrate measures 250 ml. Determine the nitrogen content in the filter paper and the residue in accordance with ISO 5315.

4.4.3 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.