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Fertilizers and soil conditioners — Solid urea aldehyde slow release fertilizer — General requirements

Engrais et amendements — Engrais urée aldéhyde solide à libération lente — Exigences générales

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners.*

Introduction

Solid urea aldehyde slow release fertilizer is a non-coated and chemically synthesized nitrogen fertilizer 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. Solid urea aldehyde slow release fertilizer has the longest history of research, use and production among the slow release fertilizers used in practice. At the same time, it is the most widely used of all slow release fertilizers.

For facilitating international fertilizer trade, it is necessary to have an international and general standard for solid urea aldehyde slow release fertilizers.

Fertilizers and soil conditioners — Solid urea aldehyde slow release fertilizer — General requirements

1 Scope

This document specifies general requirements, analytical methods, sampling and preparation of test sample, marking and labelling, packaging, transport and storage for solid urea aldehyde slow release fertilizer.

This document applies to pure solid urea aldehyde slow release fertilizer, i.e. urea formaldehyde (UF), methylene urea (MU), crotonylidene diurea (CDU), isobutylidene diurea (IBDU). This document does not apply to mixtures of nitrogenous fertilizers containing solid urea aldehyde slow release fertilizer.

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.

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

ISO 7409, *Fertilizers — Marking — Presentation and declarations*

ISO 7410, *Fertilizers and soil conditioners — Final samples — Practical arrangements*

ISO 7742, *Solid fertilizers — Reduction of samples*

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

ISO 8633, *Solid fertilizers — Simple sampling method for small lots*

ISO 19746:2017, *Determination of urea content in urea-based fertilizers by high performance liquid chromatography (HPLC)*

ISO 25705:2016, *Fertilizers — Determination of urea condensates using high-performance liquid chromatography (HPLC) — Isobutylidenediurea and crotonylidenediurea (method A) and methylen-urea oligomers (method B)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8157 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 aldehyde slow release fertilizer
products of reaction between urea and aldehyde(s) that produce a slow release nitrogenous fertilizer

EXAMPLE Urea formaldehyde (UF), methylene urea (MU), crotonylidene diurea (CDU), isobutylidene diurea (IBDU).

3.2
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

3.3
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

3.4
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

3.5
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

3.6
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) at 25 °C during a 15 min period

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

3.7
activity index
AI
percentage of *CWIN* (3.2) that is solubilised in hot water (*HWSN only*) (3.6)

Note 1 to entry: $\mathrm{AI} = \dfrac{\mathrm{CWIN} - \mathrm{HWIN}}{\mathrm{CWIN}} \times 100$

Note 2 to entry: CWIN and HWIN in Note 1 are expressed in the mass fraction (%).

Note 3 to entry: A higher AI indicates better slow release characteristics of urea formaldehyde fertilizer.

4 Requirements

4.1 Visual inspection

The product shall be in powder, granules, prills, pellets, pastilles, chips or other solid forms. Visually inspect for the presence of contaminant and foreign matter.

4.2 Requirement of solid urea aldehyde slow release fertilizer

Solid urea aldehyde slow release fertilizer shall be tested to demonstrate conformance with all the requirements specified in Tables 1 and 2 respectively, and declared values on containers.

Table 1 — Requirements of urea formaldehyde/methylene urea fertilizer

Item		Requirements
Total nitrogen (TN) (mass fraction)	≥	36 %
Ureic nitrogen (mass fraction)	≤	5 %
HWSN (mass fraction)	≥	3/5 of the declared total nitrogen content
AI	≥	40 %
The requirements specified by national/regional legislation shall be followed when urea formaldehyde/methylene ureas are covered by legislation.		

Table 2 — Requirements of IBDU and CDU

Item		Requirements
Total nitrogen (mass fraction)	≥	28 %
Ureic nitrogen (mass fraction)	≤	3 %
Nitrogen from IBDU or CDU (mass fraction)	≥	25 %
The requirements specified by the national/regional legislation shall be followed when IBDU or CDU are covered by legislation.		

5 Analytical methods

5.1 Determination of the appearance

It shall be determined by visual method.

5.2 Determination of the mass fraction of total nitrogen

It shall be determined in accordance with ISO 5315.

5.3 Determination of the mass fraction of ureic nitrogen

It shall be determined in accordance with ISO 19746.

5.4 Determination of the mass fraction of CWIN

5.4.1 Principle

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

5.4.2 Reagents

5.4.2.1 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.

5.4.2.2 Anhydrous ethanol.

5.4.2.3 Reagents listed in ISO 5315.

5.4.3 Apparatus

5.4.3.1 Usual laboratory apparatus.

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

5.4.3.3 **Quantitative filter paper** (intermediate speed).

5.4.3.4 Apparatus listed in ISO 5315.

5.4.4 Procedure

Two analyses shall be performed simultaneously for the determination.

Place 1 g to 1,4 g test portion (accurate to 0,000 1 g) in 50 ml beaker wet with ethanol (5.4.2.2). Add 20 ml phosphate buffer solution (5.4.2.1) or distilled water and let stand 15 min in the water bath maintained at 25 °C ± 2 °C and stir at 5 min intervals during standing. Transfer the supernate to a piece of filter paper (5.4.3.3) in a long-stem funnel and wash the residue four or five times by decanting with water of 25 °C ± 2 °C. Finally, transfer all the residue to the filter paper and complete washing until the filtrate measures 250 ml. Determine nitrogen content in the filter paper and the residue in accordance with ISO 5315.

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.

5.4.5 Expression of results

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

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

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 litre, 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.

5.5 Determination of the mass fraction of HWIN and AI

5.5.1 Principle

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

5.5.2 Reagents

5.5.2.1 Reagents listed in 5.4.2.

5.5.2.2 **Celite**.

5.5.3 Apparatus

5.5.3.1 Usual laboratory apparatus.

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

5.5.3.3 Apparatus listed in 5.4.3.3 and 5.4.3.4.

5.5.4 Procedure

Two analyses shall be performed simultaneously for the determination.

Place an accurately weighed test portion containing 0,12 g CWIN (accurate to 0,000 1 g) in a 250 ml tall-form beaker. Add 100 ml 100 °C phosphate buffer solution (5.4.2.1) from a graduated cylinder to the test portion, stir, cover and immerse promptly in a boiling water bath (5.5.3.2) so that liquid in the beaker is below the water level in the water bath. Maintain the water bath at 98 °C to 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 (5.4.3.3). If filtration takes more than 4 min, discard determination. Repeat the determination, adding 1 g celite (5.5.2.2) as filter-aid just before removing the beaker from the water bath, stir and filter.

Wash the insoluble residue completely onto the filter paper with boiling water and continue washing until the total volume used is 100 ml. Complete washing before the filtrate becomes cloudy or its temperature drops to below 60 °C. Determine total nitrogen in the wet filter paper and residue in accordance with ISO 5315.

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

5.5.5 Expression of results

5.5.5.1 HWIN content of the sample

The HWIN content, w_2, expressed as a mass fraction (%), is given by Formula (2):

$$w_2 = \frac{(V_3 - V_4) \times c_2 \times 14{,}01}{m_2 \times 1\,000} \times 100 \tag{2}$$

where

V_3 is the volume, in millilitres, of sodium hydroxide standard solution used for the blank test;

V_4 is the volume, in millilitres, of sodium hydroxide standard solution used for the determination;

c_2 is the concentration, in mol per litres, of sodium hydroxide standard solution used;

m_2 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.

5.5.5.2 Calculation of AI

AI, expressed in %, is given by Formula (3):

$$AI = \frac{w_1 - w_2}{w_1} \times 100 \tag{3}$$

5.6 Determination of the mass fraction of nitrogen from IBDU and CDU

It shall be determined in accordance with ISO 25705:2016, method A or ISO 19746.

6 Sampling and preparation of test sample

6.1 Sampling method

6.1.1 Products in bags

Carry out the sampling operation by following the procedure described in ISO 8633.

6.1.2 Products in bulk

Carry out the sampling operation by following the procedure described in ISO 8633.

6.2 Reduction of samples

Mix all the increments (collected as in 6.1) uniformly and promptly to form a single aggregate sample using a device or by hand. The aggregate sample is reduced to about 1 kg according to the procedure in ISO 7742. Next, divide into two parts for final laboratory samples. The two laboratory samples are put into two clean and dry glass or plastic containers or any other inert material of adequate resistance capable of maintaining the sample in its original condition. The containers shall be fitted with airtight closures. Carry out all the operations described above as rapidly as possible to avoid loss or gain of moisture. Each container shall be secured and sealed following the instruction given in ISO 7410. Each laboratory sample shall be labelled following the instructions given in ISO 7410. The label shall, at minimum, carry the following information:

a) the name of manufacturer;

b) the name of product;

c) the manufacturer's reference and batch number or production date (if available);

d) the lot size;

e) the date of sampling;

f) the place of sampling;

g) the signature of the sampler;

h) the signature and name of the person or his representative on whose premises the sample was taken.

One of the containers is used for further quality analysis, while the other is kept for additional analysis in 6 mos.

6.3 Test sample preparation

Select one of the laboratory samples from the two containers obtained in 6.2. Mix the content of the container according to the procedure in ISO 7742. The test sample used for determining the items