SLOVENSKI STANDARD
oSIST prEN ISO 25705:2019

01-junij-2019

Gnojila - Določevanje kondenzatov sečnine s tekočinsko kromatografijo visoke ločljivosti (HPLC) - Izobutilidendiurea in krotonilidendiurea (metoda A) in oligomeri metilenuree (metoda B) (ISO 25705:2016)

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

DÃ¼ngemittel - Bestimmung von Harnstoffkondensaten mit Hochleistungs-FlÃ¼ssigchromatographie (HPLC) - Isobutylidendiharnstoff und Crotonylidendiharnstoff (Verfahren A) und Methylenharnstoff-Oligomere (Verfahren B); Deutsche und Englische Fassung prEN ISO 25705:2018

Engrais - Dosage des condensats d'urée par chromatographie liquide haute performance (HPLC) - Isobutylidène diurée et crotonylidène diurée (méthode A) et oligomères de méthylène-urée (méthode B) (ISO 25705:2016)

Ta slovenski standard je istoveten z: **prEN ISO 25705**

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65.080	Gnojila	Fertilizers
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INTERNATIONAL STANDARD

ISO 25705

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Fertilizers — Determination of urea condensates using high-performance liquid chromatography (HPLC) — Isobutylidenediurea and crotonylidenediurea (method A) and methylen-urea oligomers (method B)

Engrais — Dosage des condensats d'urée par chromatographie liquide haute performance (CLHP) — Isobutylidène diurée et crotonylidène diurée (méthode A) et oligomères de méthylène-urée (méthode B)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 25705 was prepared by CEN/TC 260 (as EN 15705:2010) and was adopted by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*. The following modifications were made.

— The references to EN 1482-1 and EN 1482-2 were changed to ISO 14820-1 and ISO 14820-2.

— In 5.2.1 and 6.2.1, the general text (not related to the water) was moved directly under 5.2 and 6.2, respectively.

— In Table 1, the word "approximate" was added to the column headers for IBDU and CDU.

— In 5.4.3 and 6.4.3, "the sample grounded" was changed to "the ground sample".

— In 6.4.3, "pieces of glass" and "pieces" were replaced by "boiling stones".

— In 6.4.2.1, 6.4.2.2, 6.4.2.3, and 6.4.2.4, "by placing the flask in the" was added before "ultrasonic bath".

— In 6.4.2.1, 6.4.2.2, 6.4.2.3, 6.4.2.4, 6.4.2.5 and 6.4.3, "homogenize" was changed to "mix thoroughly".

— In 6.4.2.5, "before transferring into" was changed to "before transferring in" three times.

— In the keys for Figures B.2, B.3, B.4, C.2, C.3, C.4 and C.5, the units for the areas were added.

Introduction

Fertilizers containing the condensates of urea and specified aldehydes (with crotonaldehyde called crotonyliden diurea or CDU, with isobutyraldehyde called isobutylidene diurea or IBDU, with formaldehyde called urea formaldehyde or methylene urea or MU) are covered by Regulation (EC) 2003/2003, Annex I[4] as nitrogenous fertilizers. The methods described in this International Standard enable the quantitative determination of these condensates and the determination of the solubility of the MU-oligomers according to the Regulation.

Fertilizers — Determination of urea condensates using high-performance liquid chromatography (HPLC) — Isobutylidenediurea and crotonylidenediurea (method A) and methylen-urea oligomers (method B)

1 Scope

This International Standard specifies methods for the determination of isobutylidene diurea (IBDU), Crotonylidene diurea (CDU) (method A) and methylene-urea oligomers (MU) (method B) in fertilizers using high-performance liquid chromatography (HPLC).

The method is applicable to all fertilizers which do not contain interfering organic compounds.

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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14820-2, *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*

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 Sampling and sample preparation

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 14820-1.

Sample preparation shall be carried out in accordance with ISO 14820-2.

5 Method A: Determination of CDU and IBDU

5.1 Principle

The sample is extracted with water and, after appropriate dilution, analysed using a suitable HPLC system.

5.2 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.2.1 Water, distilled or demineralized water (grade 3 according to ISO 3696).

5.2.2 Acetonitrile, HPLC-grade.

5.2.3 Isobutylidene diurea and Crotonylidene diurea, in their pure form.

5.3 Apparatus

5.3.1 Laboratory equipment and glassware, for preparation of solutions and dilutions.

5.3.2 Analytical balance, capable of weighing to an accuracy of ±0,1 mg.

5.3.3 HPLC-system, with UV-detector.

5.3.4 Ultrasonic bath.

5.3.5 Magnetic stirrer.

5.3.6 Disposable filter, 0,45 µm.

5.4 Procedure

5.4.1 System parameters of HPLC

Analytical/separating column: silica column with C18 reverse phase[1)]

Detection wavelength:	200 nm
Eluent:	acetonitrile/water: 10/90 (volume fraction)
Flow rate:	1 ml/min
Temperature:	ambient temperature
Injection volume:	20 µl

5.4.2 Calibration

5.4.2.1 Stock solution IBDU ρ(IBDU) = 100 mg/l

Weigh 100/R mg of IBDU (5.2.3), where R is the purity of IBDU, into a 1 000 ml flask and add about 900 ml of water (5.2.1). Dissolve in an ultrasonic bath (5.3.4) for about 10 min, followed by stirring on a magnetic stirrer (5.3.5) for about 1 h. Make up to volume. Filtration is not necessary.

5.4.2.2 Stock solution CDU ρ(CDU) = 100 mg/l

Weigh 100/R mg of CDU (5.2.3), where R is the purity of CDU, into a 1 000 ml flask and add about 900 ml of water (5.2.1). Dissolve in an ultrasonic bath (5.3.4) for about 10 min, followed by stirring on a magnetic stirrer (5.3.5) for about 1 h. Make up to volume. Filtration is not necessary.

5.4.2.3 Calibration solution

For calibration, prepare three solutions according to Table 1 using one-mark (bulb) pipettes and dilute to the mark with water (5.2.1).

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1) LiChrosorb RP-18 7 µm 250/4 mm is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

For the determination of the retention time, dilute 10 ml of the stock solution (5.4.2.1) or respectively (5.4.2.2) into two 100 ml flasks and make up to volume with water (5.2.1).

The evaluation of calibration is carried out manually or by means of a suitable PC-aided (computerized) calculation method.

Table 1 — Preparation of calibration solutions

Parameter	Amount of stock solution IBDU/CDU ml (to be added to the 100 ml flask)	Content of IBDU mg/l (approximate)	Content of CDU mg/l (approximate)
Standard 1	10	10,0	10,0
Standard 2	25	25,0	25,0
Standard 3	50	50,0	50,0

5.4.3 Preparation of the test portion

Weigh 1 g of the ground sample to <0,2 mm to the nearest 0,1 mg and quantitatively transfer into a 1 000 ml volumetric flask with water (5.2.1). Fill the flask to an amount of approximately 900 ml and treat it for 10 min in the ultrasonic bath (5.3.4). Then make up to the mark and stir for 1 h at room temperature on a magnetic stirrer (5.3.5). Dilute 10 ml of the solution in a 100 ml volumetric flask and filter into the HPLC injection vial through a disposable filter (5.3.6).

5.4.4 Measurement

Measurement is performed manually or by means of an automatic sample loading system (autosampler).

5.4.5 Important annotations

IBDU is able to form urea in aqueous solution. Therefore, the measurement of the calibration and sample solutions shall be completed within one working day.

The concentrations of CDU and IBDU in the sample solutions shall be kept within the calibration limits (5.4.2) to ensure sufficient reproducibility.

5.5 Calculation

The calculation can be performed manually or by means of a PC using the calibration parameters in respect to the amount used.

In the case of PC-aided (computerized) calculation and application of Table 1 regarding the amounts of stock solution, the content of IBDU/CDU in milligrams per litre will be calculated by the system. The calculated values are equal to the percentage mass concentration of IBDU/CDU in the analysed sample of fertilizer.

Following general rules for declaration in regulations to declare the content of the compounds as percentage mass fraction of nitrogen, calculate the contents, $w_{N(IBDU)}/w_{N(CDU)}$ in percent (g/100 g), according to Formulae (1) and (2):

$$w_{N(IBDU)} = w_{IBDU} \times 0{,}322 \quad (1)$$

$$w_{N(CDU)} = w_{CDU} \times 0{,}326 \quad (2)$$

where