SLOVENSKI STANDARD
kSIST FprEN 15705:2009

01-november-2009

Gnojila - Določanje kondenzatov sečnine s tekočinsko kromatografijo visoke ločljivosti (HPLC) - Izobutilidendiurea in krotonilidendiurea (metoda A) in oligomeri metilenuree (metoda B)

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

Düngemittel - Bestimmung von Harnstoffkondensaten mit Hochleistungs-Flüssigchromatographie (HPLC) - Isobutylidendiharnstoff und Crotonylidendiharnstoff (Verfahren A) und Methylenharnstoff-Oligomere (Verfahren B)

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

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Ta slovenski standard je istoveten z: **FprEN 15705**

ICS:

65.080 Gnojila Fertilizers

kSIST FprEN 15705:2009 **en,fr,de**

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

FINAL DRAFT
FprEN 15705

August 2009

ICS 65.080

Will supersede CEN/TS 15705:2009

English Version

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

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If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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

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Foreword

This document (FprEN 15705:2009) 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 Unique Acceptance Procedure.

This document will supersede CEN/TS 15705:2009.

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

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 Annex I of the Regulation (EC) 2003/2003 [1] as nitrogenous fertilizers. The methods described in this European Standard enable the quantitative determination of these condensates and the determination of the solubility of the MU-oligomers according to the Regulation.

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1 Scope

This European Standard specifies methods for the determination of isobutylidenediurea (IBDU), crotonylidenediurea (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 referenced documents are indispensable for the application 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 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

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4 Sampling and sample preparation

Sampling is not part of the method specified in this European Standard. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2.

5 Method A: Determination of CDU and IBDU

5.1 Principle

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

5.2 Reagents

5.2.1 Reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

5.2.2 **Acetonitrile,** p.a., HPLC-grade.

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5.2.3 Isobutylidenediurea and crotonylidenediurea, 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 Disposible 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

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5.4.2 Calibration

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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).

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).

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[1] E.g. LiChrosorb RP-18 7µm 250/4 mm or equivalent.

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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	Content of CDU mg/l
Standard1	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 sample grounded to < 0,2 mm to the nearest 0,1 mg and flush 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

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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.

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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 the following equations:

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

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

where

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0,322 is the conversation factor for the content of IBDU in the fertilizer into nitrogen content;

0,326 is the conversation factor for the content of CDU in the fertilizer into nitrogen content.

6 Method B: Determination of methylen-urea oligomers (MU)

NOTE By the condensation of urea and formaldehyde developed no only one compound, as it is by the reaction of urea and crotonaldehyde or isobutylaldehyde, but oligomers like methylen–diurea (MDU), dimethylen-triurea (DMTU), trimethylen-tetraurea (TMTU) and higher oligomers. These three molecules are the most soluble in water, the higher compounds are insoluble in hot water, but their nitrogen is available for plants by microbiological decomposition. Also urea is always a companion of MU – oligomers.

6.1 Principle

The sample is extracted with boiling water and analyzed using a suitable HPLC system.

The methylen-urea soluble oligomers are measured and detected by the HPLC-method.

In the HPLC-diagram methylen-urea oligomers are represented by different peaks: urea, methylen-diurea, dimethylen-triurea; trimethylen-tetraurea are, in the mean time, the most soluble and important.

6.2 Reagents

6.2.1 Reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

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6.2.2 Acetonitrile, p.a., HPLC-grade.

6.2.3 Urea, p.a., 46,6 % of total nitrogen.

6.2.4 Methylen-diurea (MDU), synthesized and purified by a special laboratory, 42,4 % of total nitrogen.

6.2.5 Dimethylen-triurea (DMTU), synthesized and purified by a special laboratory, 41,2 % of total nitrogen.

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6.2.6 Trimethylen-tetraurea (TMTU), synthesized and purified by a special laboratory, 40,6 % of total nitrogen.

6.3 Apparatus

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

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

6.3.3 Technical balance, capable of weighing to an accuracy of ± 0,01 g.

6.3.4 HPLC-system, equipped with an UV-detector.

6.3.5 Ultrasonic bath

6.3.6 Magnetic stirrer

6.3.7 Disposable filter, 0,45 µm.