



**SLOVENSKI STANDARD**  
**oSIST prEN 17864:2022**  
**01-julij-2022**

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**Anorganska gnojila - Določevanje dušika v IBDU (izobutilidendiurea) in CDU ((krotonilidendiurea)**

Inorganic fertilizers - Determination of nitrogen content in IBDU (isobutylidenediurea) and CDU (crotonylidenediurea)

Anorganische Düngemittel - Bestimmung des Stickstoffgehalts in IBDU (Isobutylidendiharnstoff) und CDU (Crotonylidendiharnstoff)

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EUROPEAN STANDARD  
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ICS

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**Inorganic fertilizers - Determination of nitrogen content in  
IBDU (isobutylidenediurea) and CDU  
(crotonylidenediurea)**

Anorganische Düngemittel - Bestimmung des  
Stickstoffgehalts in IBDU (Isobutylidendiarnstoff) und  
CDU (Crotonylidendiarnstoff)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 260.

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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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

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**

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## European foreword

This document (prEN 17864:2022) 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 CEN Enquiry.

This document, together with prEN 15705:2022, will supersede EN 15705:2010.

In comparison with EN 15705:2010, the following technical modifications have been made:

- EN 15705:2010 is split into two parts:
  - Method A of EN 15705:2010 is given in this document.
  - Method B of EN 15705:2010 is transferred to prEN 15705:2022.

This document has been prepared under a Standardization Request given to CEN by the European Commission and the European Free Trade Association.

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**prEN 17864:2022 (E)****1 Scope**

This document specifies a method for the determination of nitrogen content in IBDU (isobutylidenediurea) and CDU (crotonylidenediurea) using high-performance liquid chromatography (HPLC).

**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*

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

**3 Terms and definitions**

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

**4 Principle**

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

**5 Reagents**

Use only reagents of recognized analytical grade and distilled or demineralized water, free from carbon dioxide and all nitrogenous compounds (grade 3 according to EN ISO 3696:1995).

**5.1 Acetonitrile**, HPLC-grade.

**5.2 Isobutylidenediurea (IBDU)**, in pure form, as standard.

**5.3 Crotonylidenediurea (CDU)**, in pure form, as standard.

**5.4 Eluent**, acetonitrile + water (10 + 90, V + V).

Mix 100,0 ml of acetonitrile (5.1) with 900,0 ml of water and homogenize well.

**6 Apparatus and equipment**

Usual laboratory glassware and equipment and, in particular, the following.

**6.1 Analytical balance**, capable for weighing to the nearest 0,1 mg.

**6.2 Ultrasonic bath.**

**6.3 Magnetic stirrer.**

**6.4 Disposable filter**, 0,45  $\mu\text{m}$ .

**6.5 HPLC-system**, with the following components.

**6.5.1 UV-detector.**

**6.5.2 HPLC reversed phase column.**

An example of a suitable column and analytical conditions are reported in Annex C.

An acceptable chromatogram, as shown as example in Annex B, can be achieved using a silica column with C18 reverse phase.

## 7 Sampling and sample preparation

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

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

## 8 Procedure

### 8.1 General

IBDU is able to form urea in aqueous solution. Therefore, the measurement of the calibration solutions 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 (see 8.2) to ensure sufficient reproducibility.

### 8.2 Calibration

#### 8.2.1 Stock solution IBDU, mass concentration $\rho(\text{IBDU}) = 100 \text{ mg/l}$

Weigh  $100/P$  mg of IBDU (5.2), where  $P$  is the purity of IBDU expressed as a pure number, to the nearest 0,1 mg, into a 1 000 ml flask and add about 900 ml of water. Dissolve in an ultrasonic bath (6.2) for about 10 min, followed by stirring on a magnetic stirrer (6.3) for about 1 h. Make up to volume with water. Filtration is not necessary.

#### 8.2.2 Stock solution CDU, $\rho(\text{CDU}) = 100 \text{ mg/l}$

Weigh  $100/P$  mg of CDU (5.3), where  $P$  is the purity of expressed as a pure number, to the nearest 0,1 mg, into a 1 000 ml flask and add about 900 ml of water. Dissolve in an ultrasonic bath (6.2) for about 10 min, followed by stirring on a magnetic stirrer (6.3) for about 1 h. Make up to volume with water. Filtration is not necessary.

#### 8.2.3 Calibration solutions

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

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For the determination of the retention time, dilute 10 ml of the stock solution (see 8.2.1) or respectively (see 8.2.2) into two 100 ml flasks and make up to volume with water.

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**

Calibration solution	Amount of stock solution IBDU and CDU <sup>a</sup>	Equivalent concentration of IBDU	Equivalent concentration of CDU
	ml	mg/l	mg/l
1	10,0	10,0	10,0
2	25,0	25,0	25,0
3	50,0	50,0	50,0

<sup>a</sup> To be added to the 100 ml flask.

### 8.3 Preparation of the test solution

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. Fill the flask to an amount of approximately 900 ml and treat it for approximately 10 min in the ultrasonic bath (6.2). Then make up to the mark with water and stir for approximately 1 h at room temperature on a magnetic stirrer (6.3). Dilute 10 ml of the solution in a 100 ml volumetric flask and filter into the HPLC injection vial through a 0,45 µm disposable filter (6.4).

### 8.4 Measurement

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

## 9 Calculation and expression of the result

The calculation is 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 and CDU in milligram per litre is calculated by the system. The calculated values are equal to the percentage mass concentration of IBDU ( $w_{(IBDU)}$ ) and CDU ( $w_{(CDU)}$ ) in the sample.

Calculate the content  $w_{N(IBDU)}$  and  $w_{N(CDU)}$  in percentage mass fraction of nitrogen according to the following Formulae:

$$w_{N(IBDU)} = w_{(IBDU)} \times F_1 \quad (1)$$

$$w_{N(CDU)} = w_{(CDU)} \times F_2 \quad (2)$$

where

$F_1$  is the conversion factor from the content of IBDU to the nitrogen content of IBDU, i.e. 0,322;

$F_2$  is the conversion factor from the content of CDU to the nitrogen content of CDU, i.e. 0,326.



## 10 Precision

### 10.1 Inter-laboratory test

Inter-laboratory tests have been carried out in 2006 with 11 participating laboratories. Repeatability and reproducibility were calculated according to ISO 5725-2.

The values derived from this inter-laboratory tests may not be applicable to concentration ranges and matrices other than those given in Annex A.

### 10.2 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in no more than 5 % of the cases exceed the values of  $r$  given in Table 2.

### 10.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in no more than 5 % of the cases exceed values of  $R$  given in Table 2.

**Table 2 — Mean values, repeatability and reproducibility limits**

Sample	$\bar{x}$ %	$r$ %	$R$ %
IBDU 1	17,838	1,312	3,222
IBDU 2	35,411	1,297	3,874
CDU	38,264	0,922	3,257

## 11 Test report

The test report shall contain at least the following information:

- all information necessary for the complete identification of the sample;
- a reference to this document, including its year of publication;
- the test results obtained;
- date of sampling and sampling procedure (if known);
- date when the analysis was finished;
- whether the requirement of the repeatability limit has been fulfilled;
- all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method, which might have influenced the test result(s).

## Annex A (informative)

### Results of the inter-laboratory tests

The precision has been determined in the year 2006 in an inter-laboratory trial with 11 laboratories participating and carried out on three samples of fertilizer (two for IBDU and one for CDU). The statistical results are given in Table A.1.

**Table A.1 — Statistical results of the inter-laboratory tests**

Parameter	IBDU 1	IBDU 2	CDU
Year of the test	2006	2006	2006
Number of participating laboratories	11	11	11
Number of laboratories after eliminating outliers	11	9	10
Level mean value, (g/100 g)	17,838	35,411	38,264
Repeatability standard deviation $s_r$ , (g/100 g)	0,473	0,468	0,333
Coefficient of variation $CV_r$ (%)	2,65	1,32	0,87
Repeatability limit $r$ ( $2,77 s_r$ ) (g/100 g)	1,312	1,297	0,922
Reproducibility standard deviation, $s_R$ (g/100 g)	1,163	1,399	1,176
Coefficient of variation $CV_R$ (%)	6,52	3,95	3,07
Reproducibility limit $R$ ( $2,77 s_R$ ) (g/100 g)	3,222	3,874	3,257