



# SLOVENSKI STANDARD

## SIST EN 15360:2007

01-november-2007

BUXca Yý U

SIST-TS CEN/TS 15360:2006

; bcŦU!8c`c Yj UbŦXŦŦUbcXŦUa ŦXU!A YŦcXUg'hŦ\_c Ŧbg\_c`\_fca UŦc[ fUŦc`j Ŧgc\_Y  
`c`Ŧj cghŦfkD@Ŧ

Fertilizers - Determination of dicyandiamide - Method using high-performance liquid chromatography (HPLC)

Düngemittel - Bestimmung von Dicyandiamid - Verfahren mit Hochleistungs-Flüssigchromatographie (HPLC)

Engrais - Détermination de la teneur en dicyandiamide - Méthode par chromatographie liquide a haute performance (CLHP)

Ta slovenski standard je istoveten z: EN 15360:2007

### ICS:

65.080

Gnojila

Fertilizers

SIST EN 15360:2007

en,fr,de

## **iTeh STANDARD PREVIEW** **(standards.iteh.ai)**

SIST EN 15360:2007

<https://standards.iteh.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007>

English Version

## Fertilizers - Determination of dicyandiamide - Method using high-performance liquid chromatography (HPLC)

Engrais - Détermination de la teneur en dicyandiamide -  
Méthode par chromatographie liquide à haute performance  
(HPLC)

Düngemittel - Bestimmung von Dicyandiamid - Verfahren  
mit Hochleistungs-Flüssigchromatographie (HPLC)

This European Standard was approved by CEN on 13 April 2007.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

[SIST EN 15360:2007](https://standards.iteh.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007)

<https://standards.iteh.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007>



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

## Contents

Page

Foreword.....	3
1 Scope .....	4
2 Normative references .....	4
3 Principle .....	4
4 Reagents .....	4
5 Apparatus .....	4
6 Sampling and sample preparation .....	5
7 Procedure .....	5
8 Calculation.....	6
9 Precision .....	6
10 Notes on procedure .....	7
11 Test report .....	7
Annex A (informative) Results of the inter-laboratory trial .....	8
Bibliography .....	9

[SIST EN 15360:2007  
https://standards.itech.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007](https://standards.itech.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007)

## Foreword

This document (EN 15360:2007) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2007, and conflicting national standards shall be withdrawn at the latest by November 2007.

This document supersedes CEN/TS 15360:2006.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

## iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN 15360:2007

<https://standards.iteh.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007>

## 1 Scope

This document specifies a method for the selective determination of dicyandiamide (DCD) in addition to all the other forms of nitrogen fixations, particularly in fertilizers to which DCD has been added as a nitrification inhibiting agent.

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

## 3 Principle

The sample is dissolved or suspended in water using an ultrasonic bath. Methyl dicyandiamide is added to the filtered solution as the internal standard. The solution is then transferred onto a C18 reversed-phase column using a bypass injector and then separated. For detection, a UV-detector is used at a wavelength of 220 nm.

## 4 Reagents

Use only reagents of recognized analytical grade and water conforming to grade 2 of EN ISO 3696.

### 4.1 Dicyandiamide standard solution

Weigh 50 mg of dicyandiamide of known purity into a 1 000 ml volumetric flask and dissolve in water; make up the volume to the mark. Pipette 10 ml of this solution into a 100 ml volumetric flask and, after having added 10 ml of internal standard solution (4.2), make up the volume to the mark with water.

### 4.2 Internal standard solution

Weigh 50 mg of methyl dicyandiamide of known purity into a 1 000 ml volumetric flask, dissolve in water and make up the volume to the mark.

### 4.3 Methanol

HPLC-grade purity

## 5 Apparatus

### 5.1 Ultrasonic bath

### 5.2 Membrane filter

0,45 µm, with the usual filtration equipment.

### 5.3 HPLC apparatus

UV-detector for variable wavelengths and an electronic integrator, sample injection valve equipped with a 20 µl bypass injector.

## 6 Sampling and sample preparation

Sampling is not part of the method specified in this document; however, a recommended sampling method is given in EN 1482-1. Sample preparation shall be carried out in accordance with EN 1482-2.

## 7 Procedure

### 7.1 Preparing the analytical solution

Weigh to the nearest 0,001 g, between 0,8 g and 1,5 g of the ground and thoroughly homogenized test sample (corresponding to approximately 50 mg of DCD) and mix with 750 ml of water into a 1 000 ml volumetric flask and dissolve using an ultrasonic bath (5.1). Those portions that have not dissolved after 5 min are disregarded. Make up the volume to the mark with water. Filter one part of the homogenized sample solution (approximately 50 ml) through the membrane filter (5.2) into a dry vessel.

Pipette 10 ml of this filtrate into a 100 ml volumetric flask and, after having added 10 ml of the internal standard solution (4.2), make up the volume to the mark with water.

### 7.2 HPLC conditions

Eluent:	Mixture of water and methanol (4.3), (99 + 1) parts by volume
Separation column and packing:	250 mm x 4,6 mm C18 reversed-phase column
Column temperature:	Room temperature
Flow rate:	1,0 ml/min
Wavelength:	220 nm

### 7.3 HPLC determination

Alternately, transfer the standard solution (4.1) and the test solution (7.1) onto the separation column three times, applying the standard solution before the test solution. Measure the peak areas for DCD and methyl dicyandiamide.

## 8 Calculation

Calculate the proportion in the standard solution (4.1),  $P_{x'}$  according to the following equation:

$$P_{x'} = \frac{A_1}{A_2} \quad (1)$$

where

$A_1$  is the peak area for DCD (4.1);

$A_2$  is the peak area of the internal standard.

Calculate the proportion in the test solution (7.1),  $P_x$  according to the following equation:

$$P_x = \frac{A_3}{A_2} \quad (2)$$

where

$A_2$  is the peak area of the internal standard;

$A_3$  is the peak area for DCD (of 7.1).

Take from each of the groups three values for  $P_x$  and  $P_{x'}$ , the mean values  $P_{x'_M}$  and  $P_{x_M}$ , and calculate the DCD content of the sample, expressed in g/100 g using the following equation:

$$w_{\text{DCD}} = \frac{P_{x_M} \times m'}{P_{x'_M} \times m} \quad (3)$$

SIST EN 15360:2007  
<https://standards.iteh.ai/catalog/standards/sist/4bb92268-8c54-47cf-84b9-0fc6202ab301/sist-en-15360-2007>

where

$P_{x_M}$  is the mean value of the proportions for the sample;

$P_{x'_M}$  is the mean value of the proportions for the standard;

$m'$  is the mass of DCD in the standard solution (4.1) (50 mg), in milligrams;

$m$  is the mass of the sample in the aliquot part of the test solution (7.1) used, in milligrams.

If necessary, correct the final result depending on the purity of the DCD used for the standard solution.

## 9 Precision

### 9.1 General

The precision of the method has been determined in an inter-laboratory trial, carried out and evaluated in 1997 according to ISO 5725-1. A summary of the results is given in Annex A. The values derived from this inter-laboratory trial might not be applicable to concentration ranges and matrices other than those given.



## 9.2 Repeatability

The absolute difference between two independent single test results obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within short time intervals, will exceed the values for the repeatability limit  $r$ , given in Table 1, on average in no more than 5 % of cases.

## 9.3 Reproducibility

The absolute difference between two independent single test results obtained using the same method on identical test material in different laboratories by different operators using different equipment will exceed the values for the reproducibility limit  $R$ , given in Table 1, on average in no more than 5 % of cases.

Table 1 — Precision data

Sample	Level g/100 g	$r$ g/100 g	$R$ g/100 g
No 1 (Alzon 27/1)	2,41	0,089 98	0,466 7
No 2 (Alzon 27/2)	2,18	0,073 7	0,329

## 10 Notes on procedure

10.1 As described, the method covers a range between 2 g and 10 g of DCD per 100 g.

By variation of the weighed portion and the extent of dilution, DCD contents of between (0,1 g and 100 g) per 100 g can be determined.

10.2 The method may also be carried out using an external standard. In this case the equation for the calculation has to be modified accordingly.

## 11 Test report

The test report shall include at least the following information:

- information necessary for complete identification of the sample;
- test method used, making reference to this document, i.e. EN 15360;
- test results together with the units used to express them;
- date the test was finished;
- statement as to whether the requirement for the repeatability limit has been fulfilled;
- procedural steps not specified in this document or carried out optionally, as well as details of any circumstances that occurred while carrying out the method that might have influenced the result(s).