



SLOVENSKI STANDARD
SIST-TS CEN/TS 15360:2006

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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 (HPLC)

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Ta slovenski standard je istoveten z: **CEN/TS 15360:2006**

ICS:

65.080 Gnojila Fertilizers

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ICS 65.080

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 Technical Specification (CEN/TS) was approved by CEN on 30 January 2006 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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

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Foreword

This Technical Specification (CEN/TS 15360:2006) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

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 announce this Technical Specification: Austria, Belgium, 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 the United Kingdom.

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1 Scope

This Technical Specification 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 Technical Specification. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

prEN 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. To the filtered solution, methyl dicyandiamide is added as the internal standard, the solution is transferred onto a C18 reversed-phase column using a bypass injector and then separated. For the detection an UV-detector is used at a wavelength of 220 nm.

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4 Reagents

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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, and 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

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

6 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in prEN 1482-1. Sample preparation shall be carried out in accordance with prEN 1482-2.

7 Procedure

7.1 Preparation of the analytical solution

Weigh to the nearest 0,001 g an amount of test sample 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 SIST-TS CEN/TS 15360:2006
Separation column and packing:	250 mm x 4,6 mm C18 reversed-phase column (Nucleosil 5 C18, ET 250/8/4, Macherey & Nagel) or equivalent)
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, with the standard solution being applied before the test solution. Measure the peak areas for DCD and methyl dicyandiamide.

8 Calculation

Calculate the proportion of the standard solution (4.1), P_x , according to the following equation:

1) Nucleosil by Macherey&Nagel is an example of a suitable product available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can show to lead to the same results.

$$P_{x'} = \frac{A_1}{A_2}$$

where

A_1 is the peak area for DCD (of 4.1)

A_2 is the peak area if the internal standard

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

$$P_x = \frac{A_3}{A_2}$$

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 of three values for P_x and $P_{x'}$, respectively, 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:

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

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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 in the aliquot part of the test solution (7.1) used, in milligrams.

If necessary, the final result shall be corrected 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 the year 1997 according to ISO 5725-1. A summary of the results obtained 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 not more than 5 % of the 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 not more than 5 % of the 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 and 10) g of DCD per 100 g.

By variation of the weighed portion and the extent of dilution, also 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

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The test report shall include at least the following information:

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