SLOVENSKI STANDARD
SIST EN 13266:2002

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Poļ asi delujoļ a gnojila - Doloļ evanje sproġļ anja hranil - Metoda za obloġena gnojila

Slow-release fertilizers - Determination of the release of the nutrients - Method for coated fertilizers

Langsam freisetzende Düngemittel - Bestimmung der Freisetzungsrate von Nährstoffen - Verfahren für umhüllte Düngemittel

Engrais a libération lente - Détermination du mode de libération des éléments nutritifs - Méthode applicable aux engrais enrobés

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English version

Slow-release fertilizers - Determination of the release of the nutrients - Method for coated fertilizers

Engrais à libération lente - Détermination du mode de libération des éléments nutritifs - Méthode applicable aux engrais enrobés

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This European Standard was approved by CEN on 29 September 2001.

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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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

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

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

Annex A is normative and annex B is informative.

This standard includes a Bibliography.

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

1 Scope

This European Standard specifies a method for the determination of the slow release properties of nutrients from coated fertilizers. pH-dependent hydrolysis and degradation by biological or microbial mechanisms are excluded.

The specified method is only applicable to products releasing any nutrients by means of a non-biological process (i.e. those where the nutrients are released by a physical mechanism). Microbial attack on the coating (e.g. sulfur coated fertilizers) and the consequences thereof are not measurable by the technique described.

This method involves a lengthy process which may not be appropriate for day to day testing purposes. Accelerated methods may be used provided they are correlated with this standard. An example of such an accelerated method is described in annex B. Regression analysis may also be used for this purpose.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1482, *Sampling of solid fertilizers and liming materials.*

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

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
release
transfer of a nutrient from the fertilizer to the receiving medium (water)

3.2
slow release
release under the defined conditions meeting each of the criteria set out in annex A

3.3
initial release of a nutrient
the mass fraction in percent of a nutrient released during the first 24 h after the start of the test

3.4
stated release time
time period between the start of the test and the release of a given minimum percentage of the specified nutrient

NOTE This may also be referred to as longevity. See annex A.

3.5
coated fertilizer
fertilizer that is encapsulated, by covering it with a water-insoluble material, in order to reduce its release rate in water

4 Principle

Elution of a test portion of a fertilizer with a specified volume of water. Determination of the concentration of the nutrient(s), that have been dissolved in defined time intervals.

5 Apparatus

Ordinary laboratory apparatus and, in particular, the following:

5.1 Glass beakers, of capacity 800 ml, with a lid

5.2 One mark volumetric flask, 500 ml capacity

5.3 Magnetic stirrer, with a magnetic rod with a size of 25 mm, or any other suitable stirrer

5.4 Temperature-control equipment, capable of maintaining the medium at the temperature of (25 ± 0,5)°C

6 Sampling

Sampling and sample preparation shall be carried out in accordance with EN 1482.

The method of sampling and of sample preparation shall be indicated in the test report. Take care to avoid damage to, or destruction of, the coating.

Do not crush or grind the sample.

7 Procedure

7.1 Preparation of the test solution

Transfer 500 ml of water, conforming to grade 3 of EN ISO 3696, into a beaker (5.1). Weigh, to the nearest 0,01 g, (10 ± 0,1) g of the fertilizer, add it to the water in the beaker and record the time. Weigh the beaker together with its contents: the sample of fertilizer, water and stirring rod. Note the total mass to the nearest 1 g. Start the stirrer (5.3) at a rotational frequency of approximately 300 min^{-1}. Cover the beaker with a lid to avoid evaporation of water and maintain the temperature at (25 ± 0,5)°C with the temperature-control equipment (5.4).

Each time it is desired to make a nutrient determination (see 7.2), decant the solution into another beaker (5.1), taking care to avoid any of the undissolved fertilizer being carried over. Refill the beaker (5.1) with water (conforming to grade 3 of EN ISO 3696) at 25°C so as to achieve the previously recorded mass. Continue extraction immediately.

Continue the extraction procedure until more than 75 % of the nominal quantity of water soluble nutrients has been leached.

NOTE The mass ratio of fertilizer test portion to water is limited to a maximum of 2 % to avoid concentration effects

7.2 Time intervals for periodic determinations of nutrient content

Determine the nutrient content at about ten time intervals until the stated release time (for release condition 3). The first determination shall be made after (24 ± 0,25) h (for release condition 1), another one shall be made at day 28 from the beginning of the elution (for release condition 2). Choose the intervals for taking the samples with respect to the amounts of leached nutrient concentration and their detection limits (for slow release criteria 1, 2, 3 see annex A).

EXAMPLE Determination intervals for a slow-release fertilizer intended to release nutrients over eight months:

1) D_1 after 24 h (day 1);

2) D_7 at day 7 after start, period from day 1 to day 7;

3) D_{14} at day 14, period from day 7 to day 14;

4) D_{21} at day 21, period from day 14 to day 21;

5) D_{28} at day 28 (week 4, 1 month), period from day 21 to day 28;

6) D_{56} at day 56 (week 8, 2 months), period from day 28 to day 56;

7) D_{84} at day 84 (week 12, 3 months), period from day 56 to day 84;

8) D_{112} at day 112 (week 16, 4 months), period from day 84 to day 112;

9) D_{168} at day 168 (week 24, 6 months), period from day 112 to day 168;

10) D_{224} at day 224 (week 32, 8 months), period from day 168 to day 224.

7.3 Determination of the nutrient content

Take aliquots as appropriate to determine the concentrations of the nutrients, using standard analytical methods, and calculate the total extracted amount of nutrients.

Note the result as "D_n + nutrient symbol“ for each nutrient under examination.

7.4 Determination of the total water-extractable nutrient content

Transfer 500 ml of water (conforming to grade 3 of EN ISO 3696) into a beaker (5.1). Weigh, to the nearest 0,01 g, (10 ± 0,1) g of the fertilizer, ground or treated in some other appropriate manner to destroy any coating and add it to the water in the beaker. Start the stirrer (5.3) at a rotational frequency of approximately 300 min^{-1}, cover the beaker with a lid to avoid evaporation of water and maintain the temperature at (25 ± 0,5)°C with the temperature-control equipment (5.4) for about 24 h. Take aliquots as appropriate without any undissolved part to determine the concentrations of the nutrients extracted as desired, using standard analytical methods. Note the result as "C_∞ + nutrient symbol" for each nutrient under examination.

8 Expression of results

The initial release, m_1, expressed as mass fraction in percent of total water extractable nutrient(s) is given by equation (1).

$$m_1 = \frac{100 \cdot D_1}{C_\infty} \tag{1}$$

The longevity $d_{75\%}$, expressed in days, is given by the interpolation equation (2):

$$d_{75\%} = d_l + \frac{(d_h - d_l) \cdot (75 - m_l)}{(m_h - m_l)} \tag{2}$$

The released mass fraction within n days, m_n, expressed as a percentage, is given by equation (3).

$$m_n = 100 \cdot \sum_{(i=1\ to\ n)} \frac{D_i}{C_\infty} \tag{3}$$

where

m_n is the released mass fraction within n days;

C_∞ is the total water extractable nutrient content (7.4), given as mass fraction in percent;

D_n is the nutrient content of the sample at day n (released between day n-1 and day n);

n is the number of days;

d_l is the day until less than 75 % of nutrient(s) are released;

d_h is the day until more than 75 % of nutrient(s) are released;

m_l is the released mass fraction until d_l;

m_h is the released mass fraction until d_h.

9 Precision

9.1 General

The precision data of this method has been established by an international inter-laboratory test (see ISO 5725:1986[1)]).

In this test, eight samples of different origin, nutrient compositions, coating and longevity were investigated and slow release properties were determined by measurement of the electrical conductivity and determination of the nitrogen content by 11 laboratories in six countries.

For the values obtained for repeatability limit and reproducibility limit, a probability level of 95 % holds.

9.2 Repeatability

The absolute difference between two single test results, obtained under repeatability conditions, shall not exceed the value of r given in table 1.

9.3 Reproducibility

The absolute difference between two single test results, obtained under reproducibility conditions, shall not exceed the value of R given in table 1.

Table 1

value	arithmetic mean	standard deviation
r	4,6	1,2
R	12,3	4,5
CrD_{95}	6,6	3,0

CrD_{95} is the critical difference on the 95 % level (see equation (3)):

$$CrD_{95}\left(\left|\bar{y}-m_o\right|\right)=\frac{0{,}84}{\sqrt{2}}\cdot\sqrt{\left(R^2-r^2/2\right)} \qquad (3)$$

1) Now withdrawn