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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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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

ISO 21263 was prepared by CEN/TC 260 as EN 13266:2001 and was adopted by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, with the following modifications:

— in Clause 1, two instances of “may” (permission) have been changed to “can” (possibility);

— in 7.2, the wording of the last sentence has been changed so that normative Annex A is cited normatively.

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

1 Scope

This document 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 can be used provided they are correlated with this document. An example of such an accelerated method is described in Annex B. Regression analysis can also be used for this purpose.

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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

3 Terms and definitions

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

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

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

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
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 1 to entry: 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 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 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 10 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 (slow release criteria 1, 2, 3 are specified in 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 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 Formula (1).

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

The longevity $d_{75\,\%}$, expressed in days, is given by the interpolation Formula (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 Formula (3).

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

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 interlaboratory 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.

1) Now withdrawn.

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 Formula (3)]:

$$CrD_{95}\left(\left|\bar{y}-m_0\right|\right)=\frac{0,84}{\sqrt{2}}\cdot\sqrt{\left(R^2-r^2/2\right)} \quad (3)$$

where

$|\bar{y}-m_0|$ is the absolute difference between the arithmetic mean of the test result and the limit;

r is the repeatability limit;

R is the reproducibility limit.

10 Test report

The test report shall include the following information:

a) all information necessary for the complete identification of the sample;

b) the methods of sampling and of sample preparation;

c) a reference to this document;

d) a short description of the principle of the determination, including references to standardized analytical methods used;

e) the results of the determinations giving at least the results for the three criteria of Annex A and the method of expression used;

f) details of any unusual features noted during the determinations (e.g. changes in temperature, formation of a precipitate, appearance of microorganism);

g) any operation, not specified in this document, or in any other standard to which reference is made, or regarded as optional, together with reference to any circumstances which may be considered to have affected the results of the test.

Annex A
(normative)

Criteria for release conditions

For “slow release”, the criteria to be taken into account are:

1) not more than a mass fraction of 15 % of a nutrient released in 24 h;
2) not more than a mass fraction of 75 % of a nutrient released in 28 days;
3) at least a mass fraction of 75 % of a nutrient released at the stated release time.

Annex B
(informative)

Accelerated test methods based on an elevated temperature

B.1 Relationship of 'accelerated test method' to 'standard method'

Any attempt to compare results taken from “accelerated” measurements to results from the standard method should be made with caution. Make both standard and accelerated test measurements. Adjust the results taken from an accelerated test to standard time intervals on the basis of setting an equal percentage leached.

It should be considered that

— raising the temperature of extraction from (25 ± 0,5) °C may have a different effect on the relative solubility of different chemical compounds;

— the stability of the coating at elevated temperatures can be different;

— the behaviour of fertilizers containing urea at higher temperatures may cause problems.

B.2 Apparatus

B.2.1 For a cumulative determination according to B.3.1

B.2.1.1 Three-necked flask with a reflux condenser, 1 000 ml capacity.

B.2.1.2 Graduated measuring cylinder, 800 ml capacity.

B.2.1.3 Boiling aids.

B.2.1.4 Stop-watch, with a range of at least 24 h.

B.2.1.5 Temperature-control equipment, capable of maintaining the medium at the desired temperature (e.g. for 100 °C: heater set to boil water permanently).

B.2.1.6 Gauze fleece, resistant to boiling water, 15 cm × 15 cm.

B.2.1.7 Sensor system, to measure the nutrient content directly under the desired temperature.

B.2.2 For a periodic determination according to B.3.2

The apparatus according to B.2.1.3 to B.2.1.6 and the following.

B.2.2.1 Conical flask, with a conical joint (socket) and a suitable reflux condenser, 800 ml capacity.

B.2.2.2 One mark volumetric flask, 500 ml capacity.