SLOVENSKI STANDARD
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Izboljġevalci tal in rastni substrati - Doloļ evanje duġika - 1. del: Spremenjena Kjeldahlova metoda

Soil improvers and growing media - Determination of nitrogen - Part 1: Modified Kjeldahl method

Bodenverbesserungsmittel und Kultursubstrate - Bestimmung von Stickstoff - Teil 1: Modifiziertes Verfahren nach Kjeldahl

Amendements du sol et supports de culture - Détermination de l'azote - Partie 1: Méthode de Kjeldahl modifiée

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This European Standard was approved by CEN on 11 August 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.

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 223 "Soil improvers and growing media", the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by March 2002, and conflicting national standards shall be withdrawn at the latest by March 2002.

The annexes A and B are informative.

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.

SAFETY PRECAUTIONS — Care should be taken when handling samples that may contain sharps or is of a dusty nature.

1 Scope

This European Standard specifies a method for the determination of nitrogen in soil improvers and growing media. The Kjeldahl method determines ammonium-N, nitrate-N, nitrite-N and organic N content of soil improvers and growing media. Nitrogen in N-N-linkages, N-O-linkages and some heterocyclics (especially pyridine) is only partially determined. [6], [7], [8]

The method is not applicable to liming materials and preformed materials such as mineral wool slabs and foam slabs.

NOTE The requirements of the standard may differ from the national legal requirements for the declaration of the products concerned.

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

EN 13040:1999, *Soil improvers and growing media - Sample preparation for chemical and physical test, determination of dry matter content, moisture content and laboratory compacted bulk density.*

3 Terms and definitions

For the purposes of this standard the terms and definitions given in EN 13040 apply.

4 Principle

The nitrogen content of the sample is determined using a method based on a sulfuric acid/potassium sulfate digestion. Copper sulfate is used as the catalyst.

5 Reagents

5.1 General

All reagents shall be of recognized analytical grade. Use water of grade 2 complying with EN ISO 3696.

5.2 Salicylic acid/sulfuric acid, dissolve 25 g of salicylic acid ($HO.C_6H_4COOH$) in 1000 ml of concentrated sulfuric acid (ρ = 1,84 g/ml).

5.3 Potassium sulfate catalyst mixture, grind and thoroughly mix 200 g of potassium sulfate, 6 g of copper (II) sulfate pentahydrate. (This mixture may be available commercially).

5.4 Sodium thiosulfate pentahydrate, crush the crystals to form a powder that passes through a sieve with an aperture of 0,25 mm.

5.5 Calibration substances, pure substances of known nitrogen content, for example acetanilide (C_8H_9NO), L-aspartic acid ($C_4H_7NO_4$), or amino acids of known composition.

NOTE The total content of nitrogen of the calibration substance should be as similar to the sample nitrogen content as possible.

6 Apparatus

Usual laboratory apparatus, and in particular the following :

6.1 Analytical balance, capable of weighing accurately to 1,0 mg.

6.2 Kjeldahl digestion flasks or **tubes,** of nominal value 50 ml, suitable for the digestion system (**6.3**)

NOTE Larger flasks (300 ml to 500 ml) may be required for bulky materials or where a larger sample mass is used.

6.3 Digestion or mineralisation system with variable heating controls.

6.4 Sieve, aperture 5 mm

6.5 Stand, capable of holding the digestion flasks or tubes.

7 Test sample

Prepare the test sample in accordance with EN 13040:1999, clause 9.

Losses of nitrogen can occur with samples of high ammonium-N content in alkaline situations and particularly when these samples are dried. If the sample contains (or is suspected to contain) > 500 mg/l NH_4 -N fresh basis, as determined by prEN 13651:2001 [4] or prEN 13652:2001 [5], analyze the fresh material as soon as possible.

8 Procedure

8.1 WARNING — Digestions with sulfuric acid are potentially hazardous and laboratory coats, gloves and safety spectacles or goggles shall be used.

The digestion shall be carried out in a well-ventilated fume cupboard with the reflux digestion on a temperature - controlled heating apparatus. Antibumping granules (or roughened glass beads) may be added both to the blank and the samples to prevent bumping and loss of solution. It is important to maintain gentle reflux, both of the blank and the test samples, to avoid temperature fluctuations, which could cause local superheating.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound needs to be treated as a potential health hazard. From this viewpoint, reduce exposure to these chemicals to the lowest possible level by whatever means available.

Toxic fumes are evolved by boiling sulfuric acid. Always use the concentrated acid in a fume cupboard.

8.2 Dried sample

Place a portion of the dried ground sample (**clause 7**) of between 0,2 g (expected nitrogen content ≈ 0,5 %) and 1,0 g (expected nitrogen content ≈ 0,1 %) in the digestion flask (**6.2**). Add 4 ml of salicylic/sulfuric acid (**5.2**) and swirl until the acid is thoroughly mixed with the sample. Allow the mixture to stand for several hours (overnight). Add 0,5 g of sodium thiosulfate (**5.4**) through a dry funnel with a long stem that reaches down into the bulb, or just above the acid if a tube is used. Heat the mixture cautiously on the digestion stand (**6.3**) until frothing has ceased.

Cool the flask; add 1,1 g of the crystalline mixture (**5.3**) and heat until the digestion mixture becomes clear. Boil the mixture gently for up to 5 h so that the sulfuric acid condenses about 1/3 of the way up the neck of the flask. Ensure that the temperature of the solution does not exceed 400 °C.

NOTE In many cases a boiling time of about 2 h is sufficient.

8.3 Residual moisture

The moisture in the analyzed sample (**8.2**) is determined in accordance with EN 13040:1999, clause 10, using 10 g of the dried sample obtained from EN 13040:1999, clause 9.

8.4 Fresh samples

Fresh samples shall be reduced in size to pass a 5 mm sieve or milled in the presence of dry ice (solid carbon dioxide). Weigh up to 20 g sample into a large Kjeldahl flask. Add sufficient salicylic/sulfuric acid (**5.2**) until the acid is thoroughly mixed with the sample. Allow the mixture to stand for several hours (overnight). Add sodium thiosulfate (**5.4**) (0,5 g for every 4 ml of acid used) through a dry funnel with a long stem that reaches down into the bulb. Heat the mixture cautiously on the digestion stand (**6.3**) until frothing has ceased.

Cool the flask, add the crystalline mixture (**5.3**) (1,1 g for every 4 ml of acid used) and heat until the digestion mixture becomes clear. Boil the mixture gently for up to 5 h so that the sulfuric acid condenses about 1/3 of the way up the neck of the flask. Ensure that the temperature of the solution does not exceed 400 °C.

8.5 Determination of ammonium nitrogen in the digestion

The nitrogen in the digest, as ammonium, may be determined by distillation, spectrophotometrically or any other suitable technique (See annex B).

8.6 Moisture content

Determine the moisture content of the fresh sample in accordance with EN 13040:1999, clause 10.

8.7 Blank determination

The reagent blank test shall be carried out in parallel with the determination, by the same procedure, outlined in (**8.2**) and (**8.4**) above using the same quantities of all the reagents as in the determination but omitting the test portion.

NOTE The measurement of a blank is introduced to determine the contribution of the extracting solution and glassware used.

8.8 Calibration

Weigh out a quantity of the calibration standard (**5.5**) equivalent in nitrogen content to the samples being analyzed and proceed as in (**8.2**)**.** The nitrogen determined should exceed 95 % of the calculated value.

8.9 Laboratory compacted bulk density

Determine the laboratory compacted bulk density in accordance with EN 13040:1999, annex A.

9 Calculation

The nitrogen content in milligrams per gram dry matter or milligrams per litre as received basis is calculated according to the final method of determination used.

10 Expression of results

Subtract the values determined for reagent blank from those obtained for the samples. Report all results on a mass/mass basis calculated to a dry matter basis or mass/volume on as received basis.

11 Precision

The repeatability and reproducibility of the nitrogen content in separately prepared samples should be in accordance with Table A.1.

A summary of the results of an interlaboratory trial to determine the precision of the method, in accordance with ISO 5725 [1], is given in annex A.

NOTE The values derived from this interlaboratory trial may not be applicable to concentrations and matrices other than those tested.

12 Test Report

The test report can be prepared separately or in conjunction with the test report of the subsequent analytical method.

The test report shall include the following information:

a) a reference to the European Standard;

b) a complete identification of the sample;

c) all the analytical methods used;

d) a statement whether ground or un-ground sample has been used;

e) the results of the determination expressed as mass/mass on dry matter basis for dried samples; for fresh samples on a mass/mass or mass/volume basis as received or calculated to mass/mass dry matter basis;

f) moisture content;

g) the laboratory compacted bulk density;

h) any details not specified in the European Standard, or which are optional, as well as any other factor, which may have affected the results.