CEN/TC 223/WG 4 "Analytical methods"
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Document type	Related content	Document date	Expected action
Project / Draft		2022-01-24	**INFO**

Description

Please find enclosed the 1st working draft for WI 00223108, Soil improvers and growing media — Determination of nitrogen — Part 1: Modified Kjeldahl method.

As agreed during the last meeting of WG 4 on 2021-11-09, the enclosed 1st working draft and the forms for the activation of the work item will be circulated to CEN/TC 223 as soon as the call for tender for the project leaders is completed and the project leaders are known.

CEN/TC 223

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Secretariat: NEN

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

Bodenverbessungsmittel 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

ICS:

CCMC will prepare and attach the official title page.

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European 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 NEN.

This document is a working document.

This document will supersede EN 13654-1:2001.

In comparison with the previous edition, the following technical modifications have been made:

- normative references have been adapted;
- the procedure has been described in more detail.

This document has been prepared under a standardisation request given to CEN by the European Commission and the European Free Trade Association. The annexes A and B are informative.

SAFETY PRECAUTIONS — Care should be taken when handling samples that may contain sharps or are 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. [7], [8], [9].

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

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.

EN 13040-1:—[1], *Soil improvers and growing media – Sample preparation – Part 1: Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory bulk density*

3 Terms and definitions

For the purposes of this standard the terms and definitions given in CEN/TS 00223137:—[2] and EN 13040-1:— 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 with a specific conductivity not higher than 0,2 mS/m at 25 °C, free from the elements to be determined.

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 Verification 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.

[1] Under preparation. Stage at the time of publication: prEN 13040-1:20**.

[2] Under preparation. Stage at the time of publication: prCEN/TS 00223137:20**.

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, 5 mm square aperture size.

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

7 Test sample

Prepare the test sample in accordance with EN 13040-1: —, clause 10.

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 EN 13651:—[3] [5] or EN 13652:2001 [6], 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

[3] Under preparation. Stage at the time of publication: EN 13651:20xx

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 the crystalline mixture (5.3) just above the acid if a tube is used. Heat the mixture cautiously on the digestion stand (6.3) until frothing has ceased.

Heat until there are no more organic particles visible in the digestion mixture. 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.

8.3 Residual moisture

The moisture in the analyzed sample (8.2) is determined in accordance with EN 13040-1:—, clause 11, using 10 g of the dried sample obtained from EN 13040-1:—, clause 10.

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 or the crystalline mixture (5.3) just above the acid if a tube is used. Heat the mixture cautiously on the digestion stand (6.3) until frothing has ceased.

NOTE Usually 20ml of salicylic/sulfuric acid and two tabs of the crystalline mixture (5.3) are sufficient.

Heat until there are no more organic particles visible in the digestion mixture. 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 13040-1:—, clause 11.

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 Verification

Weigh out a quantity of the verification substance (**5.5**) equivalent in nitrogen content to the samples being analysed 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 bulk density in accordance with EN 13040-1: —, 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 [2], 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 this 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 bulk density;

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

Annex A
(informative)

Results of an interlaboratory trial to determine nitrogen (modified Kjeldahl method)

An interlaboratory trial was organised in 1997 under the auspices of the European Committee for Standardization, to test the procedures specified in this European Standard.

In this trial the number of laboratories given in Table A.1 determined the nitrogen content using the modified Kjeldahl method in six sample types.

All results are reported on a dry matter basis.

The number of matrices has to be enlarged for the future ring test

Table 1 — Summary of the results of the interlaboratory trial for the determination of nitrogen (modified Kjeldahl method)

Sample	**No. of labs after Eliminating outliers**	**No. of outliers (labs)**	**Mean value** Mg/g	**Repeatability** s_r	**Repeatability** Std Dev %	**Repeatability limit** $r = 2{,}8\ s_r$	**Reproducibility** s_R	**Reproducibility** Std Dev %	**Reproducibility limit** $R = 2{,}8\ s_R$
Composted bark	15	0	19,41	0,63	9,11	1,77	2,70	38,98	7,57
Biowaste	15	2	12,88	0,32	7,03	0,91	0,93	20.27	2.61
Clay peat (fertilized)	15	0	9,44	0,31	9,21	0,87	0,66	19,61	1,85
Coarse peat (fertilized)	15	1	11,17	0,22	5,51	0,62	1,10	27,57	3,08
Composted sludge	15	1	29,90	0,96	8,97	2,68	2,97	27,77	8,30
Composted wood fibre	14	1	11,77	0,24	5,69	0,67	0,61	14,62	1,72

Annex B
(informative)

Methods of analysis used in the interlaboratory trial

ELEMENT METHOD

Methods based on or used in the interlaboratory trial

1 ISO 11261: 1995 Distillation method [3]

2 ISO 11732: 2005 Flow analysis (CFA and FIA) and spectrometric detection [4]

Alternative methods may be suitable for the concentration range and extract used.

Bibliography

[1] CEN/TS 00223137:—, Soil improvers and growing media — Terminology.

[2] ISO 5725:1994, Accuracy (trueness and precision) of measurement methods and results.

[3] ISO 11261: 1995, Soil Quality - Determination of total nitrogen - Modified Kjeldahl method.

[4] ISO 11732:2005, Water quality — Determination of ammonium nitrogen — Method by flow analysis (CFA and FIA) and spectrometric detection.

[5] EN 13651:—, Soil improvers and growing media — Extraction of calcium chloride/DTPA (CAT) soluble nutrients.

[6] EN 13652:2001, Soil improvers and growing media — Extraction of water soluble nutrients and elements.

[7] Hoffmann, G., 1997 Die Untersuchung von Böden, 4, Auflage, VDLUFA - Verlag, Darmstadt A2.2.1: Bestimmung von Gesamt-Stickstoff nach Kjeldhal (determination of total N according to Kjeldhal).

[8] Page, A.L. et. al. Methods of soil analysis, Part 2(1982). American Society of Agronomy and Soil Science of America, Madison, WI.

[9] Williams, P.C. J. Sci. Fd. Agric. 24 (1973), p, 343.