



SLOVENSKI STANDARD

SIST EN 13654-2:2002

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Soil improvers and growing media - Determination of nitrogen - Part 2: Dumas method

Soil improvers and growing media - Determination of nitrogen - Part 2: Dumas method

Bodenverbesserungsmittel und Kultursubstrate - Bestimmung von Stickstoff - Teil 2:
Verfahren nach Dumas

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Amendements du sol et supports de culture - Détermination de l'azote - Partie 2:
Méthode de Dumas

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Ta slovenski standard je istoveten z: EN 13654-2:2001

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65.080

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Fertilizers

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13654-2

September 2001

ICS 65.080

English version

Soil improvers and growing media - Determination of nitrogen - Part 2: Dumas method

Amendements du sol et supports de culture -
Détermination de l'azote - Partie 2: Méthode de Dumas

Bodenverbesserungsmittel und Kultursubstrate -
Bestimmung von Stickstoff - Teil 2: Verfahren nach Dumas

This European Standard was approved by CEN on 11 August 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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.

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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.

Annex A is 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.

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EN 13654-2:2001 (E)

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 dry combustion method was developed originally as a manual method by Dumas [5]. Its application is improved greatly due to the use of modern automated equipment and is applicable to all forms of nitrogen.

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 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.*

EN 13654-1, *Soil improvers and growing media - Determination of nitrogen – Part 1: Modified Kjeldahl method.*

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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, pre-treated according to EN 13040, is determined by heating to a temperature of at least 900 °C in the presence of oxygen gas. Mineral and organic nitrogen compounds are oxidized and/or volatilized and determined as described by the manufacturer.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade.

5.2 For reduction, oxidation, removal and/or fixing of combustion gases that interfere with the analysis, refer to the manufacturer's instructions.

5.3 Calibration substances, pure substances of known nitrogen content, for example acetanilide (C₈H₉NO), L-aspartic acid (C₄H₇NO₄), 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 Dumas apparatus, used to determine the total nitrogen content by combustion of the sample at a temperature of at least 900 °C, including a detector for measuring the nitrogen gas formed. Consult the manufacturer's manual for the setting up and use of the apparatus.

6.3 Containers, of various size as applicable to the instrument.

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 as received contains (or is suspected to contain) > 500 mg/l $\text{NH}_4\text{-N}$, as determined by EN 13651 [2] or EN 13652 [3], analyze the fresh material using the method described in EN 13654-1.

8 Procedure

8.1 Calibration of the apparatus

Calibrate the apparatus as described in the manufacturer's manual. For the purpose of calibration or establishing a calibration scale, use one of the substances given in (5.3).

8.2 Determination of the nitrogen content

The amount of test sample for analysis depends on the expected content of nitrogen and on the sample capacity of the apparatus being used. Weigh out m_1 g of the dried ground sample or sub-sample into a container (6.3). Carry out the analyses in accordance with the manufacturer's manual.

Normally the primary results are given as mg N (X_1) per mass of air dry ground test sample used (m_1).

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.

NOTE Pores within the test sample are filled with air, and therefore, with nitrogen gas. Nitrogen gas can also enter the combustion cell when it is opened for sample exchange. Both facts can lead to an overestimation of the total N content of soil improvers or growing medium if there has been insufficient purging by gas before the combustion step.

8.4 Laboratory compacted bulk density

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

9 Calculation

The nitrogen content (w_n) in milligrams per gram dry matter basis, is calculated using the formula:

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$$w_n = \frac{X_1}{m_1}$$

where:

w_n is the content of nitrogen in milligrams per gram of dried ground test sample;

X_1 is the primary result in mg nitrogen;

m_1 is the mass, in grams, of dried ground test sample used for analysis.

10 Expression of results

Report all results on a mass/mass basis calculated to a dry matter 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.

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12 Test Report

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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;
- f) the laboratory compacted bulk density;
- g) moisture content;
- 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.

Annex A (informative)

Results of an interlaboratory trial to determine nitrogen (Dumas method)

An interlaboratory trial was organized 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 Dumas method in six sample types.

All results are reported on a dry matter basis.

Table A.1 - A summary of the results of the interlaboratory trial for the determination of nitrogen (Dumas 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	10	1	19,5	0,45	6,50	1,27	1,32	18,91	3,68
Biowaste	12	0	13,21	0,36	7,70	1,02	1,10	23,40	3,09
Clay peat (fertilized)	11	1	9,2	0,25	7,67	0,71	0,63	19,21	1,78
Coarse peat (fertilized)	11	1	10,7	0,25	6,56	0,70	1,69	44,15	4,72
Composted sludge	15	1	29,90	0,96	8,97	2,68	2,97	27,77	8,30
Composted wood fibre	12	0	12,07	0,37	8,54	1,03	1,72	39,86	4,81

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