CEN/TC 223/WG 4 "Analytical methods"
WG Secretariat: **ASI**
Convenor: **Baumgarten Andreas Mr**

1stWD_WI00223106_EN13654-2rev

Document type	Related content	Document date	Expected action
Project / Other		2021-07-26	**INFO**

Description

Please find enclosed the 1st working draft on the revision of EN 13654-2. As agreed during the last meeting of CEN/TC 223/WG 4, this version is circulated to CEN/TC 223 as first working draft.

CEN/TC 223

Date: 2021-06

prEN 13654-2:2021

Secretariat: NEN

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

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

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

ICS:

CCMC will prepare and attach the official title page.

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European foreword

This document (prEN 13654-2:2021) 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 13654-2:2001.

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

- Adaptation of scope and applicability with respect to the EU – Fertilizer Product Regulation

This document has been prepared under a standardisation request given to CEN by the European Commission and the European Free Trade Association.

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 dry combustion method was developed originally as a manual method by Dumas [4]. Its application is improved greatly due to the use of modern automated equipment and is applicable to all forms of nitrogen.

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], *Soil improvers and growing media — Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 00223137:—[2] apply.

4 Principle

The nitrogen content of the sample 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 (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.

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

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

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:—, 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 as received contains (or is suspected to contain) > 500 mg/l NH_4–N, as determined by EN 13651 [2] or EN 13652 [3], prepare the sample as it is described in EN 13040:—, clause 10.2, c).

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

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.

9 Calculation

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

$$w_n = \frac{X_1}{m_1} \tag{1}$$

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.

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;

f) moisture content;

g) 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 (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

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

Bibliography

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

[2] EN 13651, *Soil improvers and growing media — Extraction of calcium chloride/DTPA (CAT) soluble nutrients and elements*

[3] EN 13652, *Soil improvers and growing media — Extraction of water soluble nutrients and elements*

[4] Dumas, J.B.A. (1831), Procédés de l'analyse organique. Ann. Chim. Phys. 247, 198-213

[5] CEN/TS 00223137:—, *Soil improvers and growing media — Terminology*