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BSI Standards Publication

Sludge, treated biowaste and soil — Determination of total nitrogen using dry combustion method

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee H/-/4, Environmental testing programmes.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Sludge, treated biowaste and soil - Determination of total nitrogen using dry combustion method

Boues, biodéchets traités et sols - Détermination de la teneur totale en azote par combustion sèche

Schlamm, behandelter Bioabfall und Boden - Bestimmung des Gesamt-Stickstoffgehalts mittels trockener Verbrennung

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Foreword

This document (EN 16168:2012) has been prepared by Technical Committee CEN/TC 400 “Project Committee - Horizontal standards in the fields of sludge, biowaste and soil”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the validation).

Table 1 — Matrices for which this European Standard is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Fresh compost Compost
Soil	Sludge amended soil Agricultural soil

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this European Standard be carried out by suitably trained staff.

1 Scope

This European Standard specifies the determination of total nitrogen (organic and inorganic) according to the procedure of Dumas in sludge, treated biowaste and soil. A typical limit of detection is 0,02 % nitrogen, and a typical limit of quantification is 0,08 % nitrogen.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

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

EN ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples (ISO 5667-15)*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

3 Terms and definitions

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

3.1
total nitrogen
amount of nitrogen that is released after Dumas combustion of the sample

4 Principle

The total nitrogen content of the material is determined by heating it to a temperature of at least 850 °C in the presence of oxygen. Mineral (inorganic) and organic nitrogen compounds are oxidized and/or volatilized. The combustion products are oxides of nitrogen (NO_x) and molecular nitrogen (N_2). After transforming all nitrogen into molecular nitrogen, the content of the nitrogen gas is measured using thermal conductometry or other device specific detectors.

5 Interferences and sources of errors

Pores in the material to be analysed are filled with air and therefore with nitrogen. Nitrogen also enters the combustion cell when it is opened to exchange the sample. Purging of the cell and the material to be analysed by inert gas is sufficient in leaving no nitrogen gas behind. Moist samples shall be used only in special cases and handled with care, as they can leach out of sample vials during the process or contaminate the device by spattering.

Fluctuations of total nitrogen may be caused by differences in nitrogen content of carrier gases used. Therefore a blank determination shall be performed after changing gas bottles and each day before starting the analytical series.

6 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

6.1 Water, grade 2 as specified in EN ISO 3696.

6.2 Combustion gas (oxygen), free of nitrogen.

6.3 Inert gas, carrier gas, free of nitrogen, e.g. helium.

6.4 Ethylenediaminetetraacetic acid (EDTA), or other calibration substances with known content of nitrogen, e.g. acetanilide (C_8H_9NO), L-aspartic acid ($C_4H_7NO_4$), sulfanilic acid or other amino acids or reference standards (e.g. NIST) which contain the matrix materials of investigations.

7 Apparatus

Usual laboratory apparatus, and in particular the following:

7.1 Dumas apparatus, suitable and calibrated for determination of total nitrogen by combustion of the sample at a temperature of at least 850 °C, and equipped with a detection device for measurement of nitrogen gas.

7.2 Crucibles, adapted to the Dumas apparatus (7.1) of variable sizes, e.g. 1 ml to 20 ml of nominal volume or special foil.

8 Sample storage and sample pretreatment

Store soil samples according to ISO 18512 and sludge samples according to EN ISO 5667-15.

For the purpose of this European Standard biowaste may be stored like soil.

Pretreat the sample according to EN 16179, if not otherwise specified.

During the drying procedure or a milling process, care shall be taken to avoid losses of ammonium-N and/or nitrate-N. Prolonged drying at 40 °C or at room temperature may cause losses of nitrogen due to microbial activity within the sample. Therefore rapid drying methods should be used.

If necessary, moist samples can be used. In this case determine the dry mass on a special sample, so that the result can be referred to dry mass.

Determine the dry mass of the sample according to EN 15934.

9 Procedure

9.1 General

The total nitrogen contribution from water (6.1), reagents and gases (6.2, 6.3) shall be significantly less than the lowest total nitrogen content to be determined. The overall total nitrogen content of water (6.1), reagents and gases (6.2, 6.3) shall be checked by performing a blank test (see 9.3).

Ascertain homogeneity of the laboratory sample and the air-dried test sample.

In special cases the use of moist samples is necessary. Take care that the sample is homogeneous and avoid spattering during the combustion process in the Dumas apparatus.

The Dumas apparatus (7.1) is used to combust the material at a minimum temperature of 850 °C in the presence of oxygen (6.2), to reduce the nitrogen oxides, to eliminate the interfering gases and to detect the content of molecular nitrogen gas formed.

9.2 Confirmation of calibration

Confirm the calibration of the apparatus by analyzing calibration substances (6.4) to control the combustion and the apparatus on the day of use as follows:

Weigh an adequate amount of EDTA (6.4) or any calibrating substance and measure the amount of nitrogen. If necessary, check the linearity of the analyser with different amounts of EDTA (6.4) or the calibrating substance used.

If the confirmation value derives by more than 3 %, the calibration should be updated.

9.3 Blank test

Carry out blank determinations (empty crucibles) in each series.

If a reproducible blank value is measured, it shall be considered during calibration. Otherwise, the reason shall be determined and removed.

NOTE Fingerprints on sample containers may lead to blank values.

9.4 Determination of total nitrogen content

Weigh a portion of the dried or moist sample to be analyzed to the nearest of 0,1 % accuracy into the crucible (7.2). The amount depends on the expected total nitrogen content and on the size of the crucible.

Carry out the analysis in accordance with the manufacturer's instructions. Use oxygen (6.2) as combustion gas. For reduction, oxidation, removal and/or fixing of combustion gases that interfere with the analysis, refer to the manufacturer’s instructions.

10 Calculation

Calculate the content of nitrogen w_N, in milligrams per kilogram, using Formula (1):

$$w_N = \frac{X \times 100}{m \times w_{dm}} \quad (1)$$

where

w_N is the content of nitrogen on the basis of dry matter, expressed in milligrams per kilogram (mg/kg);

X is the primary result in milligram (mg) nitrogen;

m is the mass of the test sample in the crucible, expressed in kilograms (kg);

w_{dm} is the dry matter fraction, expressed in percent (%), determined according to EN 15934.

11 Expression of results

The result shall be expressed in milligrams per kilogram (mg/kg) dry matter or in percent (%) on the basis of dry matter and reported to two significant figures.

12 Precision

The performance characteristics of the method have been evaluated (see Annex A).

13 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 16168);

b) complete identification of the sample;

c) the results calculated according to Clause 10 and expressed according to Clause 11;

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

Annex A
(informative)

Repeatability and reproducibility data

A.1 Materials used in the interlaboratory comparison study

The interlaboratory comparison of the determination of total nitrogen – dry combustion method in sludge, treated biowaste and soil was carried out with 12 to 13 European laboratories on six materials. Detailed information can be found in the final report on the interlaboratory comparison study mentioned in [6].

Table A.1 lists the types of materials tested.

Table A.1 — Materials tested in the interlaboratory comparison of the determination of total nitrogen – dry combustion method in sludge, treated biowaste and soil

Grain size	Sample	Material
Sludge (< 0,5 mm)	Sludge 1	Mix of municipal waste water treatment plant sludges from North Rhine Westphalia, Germany
	Sludge 2	Mix of municipal waste water treatment plant sludges from North Rhine Westphalia, Germany
Fine grained (< 2,0 mm)	Compost 1	Fresh compost from Vienna, Austria
	Compost 2	Compost from Germany
	Soil 4	Sludge amended soil from Hohenheim, Germany
	Soil 5	Agricultural soil from Reading, United Kingdom

A.2 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table A.2).

Table A.2 — Results of the interlaboratory comparison studies of the determination of total nitrogen – dry combustion method in sludge, treated biowaste and soil

Matrix	l	n	n_o	$\bar{\bar{x}}$ g/kg	s_R g/kg	$C_{V,R}$ %	s_r g/kg	$C_{V,r}$ %
Sludge 1	11	51	2	39,15	0,70	1,80	0,33	0,84
Sludge 2	12	57	1	35,88	1,81	5,03	0,55	1,54
Compost 1	12	57	1	18,07	1,01	5,61	0,37	2,02
Compost 2	14	64	2	13,79	1,32	9,59	0,53	3,86
Soil 4	12	58	1	1,79	0,40	22,52	0,11	5,89
Soil 5	11	52	2	1,92	0,29	15,30	0,13	6,52

Explanation of symbols

l	number of participating laboratories
n	number of analytical results after outlier rejection
n_o	number of outliers
$\bar{\bar{x}}$	total mean of results (without outliers)
s_R	reproducibility standard deviation
$C_{V,R}$	coefficient of variation of reproducibility
s_r	repeatability standard deviation
$C_{V,r}$	coefficient of variation of repeatability

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