SLOVENSKI STANDARD
SIST EN 16169:2013

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Blato, obdelani biološki odpadki in tla - Določevanje dušika po Kjeldahlu

Sludge, treated biowaste and soil - Determination of Kjeldahl nitrogen

Schlamm, behandelter Bioabfall und Boden - Bestimmung des Kjeldahl-Stickstoffs

Boues, bio-déchets traités et sols - Détermination de l'azote Kjeldahl

Ta slovenski standard je istoveten z: **EN 16169:2012**

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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English Version

Sludge, treated biowaste and soil - Determination of Kjeldahl nitrogen

Boues, bio-déchets traités et sols - Détermination de l'azote Kjeldahl

Schlamm, behandelter Bioabfall und Boden - Bestimmung des Kjeldahl-Stickstoffs

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Foreword

This document (EN 16169:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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

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 Kjeldahl nitrogen according to the Kjeldahl procedure in sludge, treated biowaste and soil.

Nitrate and nitrite are not included.

Compounds with nitrogen bound in N-N, N-O linkages and some heterocycles (pyridines) are only partially determined.

The limit of detection (LOD) is usually 0,03 % nitrogen, and the limit of quantification (LOQ) is 0,1 % nitrogen (using 0,25 mol/l sulfuric acid for titration).

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 purpose of this document, the following terms and definitions apply.

3.1
Kjeldahl nitrogen
nitrogen that is contributed by free ammonia, inorganic ammonia compounds and those types of organic nitrogen compounds that are converted to ammonium sulfate by the digestion process described in this standard (catalytic sulfuric acid digestion)

[SOURCE: EN 13342:2000, 3.1]

4 Principle

The dried and homogenized, moist or liquid material is digested in a suitable Kjeldahl tube with sulfuric acid, thus converting most nitrogen compounds present to ammonium sulfate. To raise the temperature, potassium sulfate is added and e.g. a mixture of titanium dioxide and copper sulfate is used as a catalyst. After adding sodium hydroxide to the digestion solution the produced ammonium is evaporated by distillation as ammonia. This is condensed in the cooling system and flows into a conical flask with boric acid solution (or diluted sulfuric acid). This solution is analyzed for ammonia by titration with sulfuric or hydrochloric acid.

5 Interferences and sources of errors

The Kjeldahl method in principle does not capture all nitrogen compounds. The nitrogen that occurs in N-N and N-O linkages (e.g. azo-, nitro- and nitroso compounds, hydrazines, hydrazones, oximes, pyrazolones, isooxazoles, dia- and triazines) is not completely recorded. Furthermore the inorganic fraction (nitrate and nitrite) is not determined. Another source of error includes contamination of the apparatus. Therefore the apparatus shall be rinsed after each analytical series and blank determinations shall be carried out. The amount of sulfuric acid used in digestion process depends on the composition of the sample (see Table 2). A ratio of sample to acid of at least 1:10 (ratio weight to volume) shall be used for samples with high content of organic matter. Digestion block temperature shall not rise above 400 °C to avoid analyte loss.

Table 2 — Amounts of sulfuric acid consumption by various materials during Kjeldahl digestion

Material	Consumption of sulfuric acid (36 mol/l) during digestion ml/g
Soil, organic C	10,0
Soil, organic matter	5,8
Al_2O_3	1,63
Fe_2O_3	1,04
Clay	0,60
$CaCO_3$	0,55
Silt	0,33
Sand	0
Salicylic acid	6,76
$Na_2S_2O_3$	0,58
Reduced Fe	1,50

6 Reagents

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

6.1 Water, complying with grade 2 according to EN ISO 3696.

6.2 Sulfuric acid, H_2SO_4, ρ = 1,84 g/ml.

6.3 Catalyst mixture

Grind and thoroughly mix 200 g of potassium sulfate (K_2SO_4), 20 g of copper sulfate pentahydrate ($CuSO_4 \cdot 5\,H_2O$) and 20 g of titanium dioxide (TiO_2), with the crystal structure of anatase, to prepare a mixture: 10:1:1 = K_2SO_4:$CuSO_4$:TiO_2.

This catalyst mixture is commercially available.

6.4 Sodium hydroxide, c(NaOH) = 10 mol/l.

6.5 Boric acid solution, H_3BO_3, ρ = 20 g/l.

6.6 Mixed indicator

Dissolve 0,1 g of bromocresol green and 0,02 g of methyl red in 100 ml ethanol.

Mixed indicators are commercially available and may be used.

6.7 Sulfuric or hydrochloric acid solution, $c(H^+)$ = 0,01 mol/l to 0,50 mol/l.

7 Apparatus

Usual laboratory apparatus, and in particular the following:

7.1 Kjeldahl digestion flasks or tubes, suitable for digestion stand (7.2).

7.2 Digestion stand, suitable for digestion of samples with sulfuric acid at a temperature near to 400 °C.

7.3 Distillation apparatus, e.g. of the Parnas-Wagner-type or other suitable distillation apparatus with steam generator.

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 samples according to EN 16179, if not otherwise specified. Usually, they are dry, homogeneous and of a defined grain size, liquid or moist. Results are referred to dry mass, so that in case of liquid or moist samples a special sample shall be used for the determination of dry mass.

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

9 Procedure

9.1 General

Homogeneity of the test sample shall be ensured.

9.2 Digestion

Place a test portion of the dried sample, of about 0,2 g to 1 g, or an undried sample with the corresponding dry matter, to the nearest of 0,1 % accuracy in the digestion flask or tube (7.1). Larger test portions are possible; the mass should be chosen according to the nitrogen content.

To use the semi-micro or the macro method respective flasks or tubes shall be used with suitable volumes. Add 10 ml sulfuric acid (6.2).

NOTE 1 The amount of sulfuric acid may be adapted to the size of the flask or tube.

Swirl until the acid is thoroughly mixed with the sample. Allow the mixture to stand and cool. Add 2,5 g of the catalyst mixture (6.3).