SLOVENSKI STANDARD
oSIST prEN 15935:2011

01-januar-2011

Blato, obdelani biološki odpadki, tla in odpadki - Določevanje žarilne izgube

Sludge, treated biowaste, soil and waste - Determination of loss on ignition

Schlamm, behandelter Bioabfall, Boden und Abfall - Bestimmung des Glühverlusts

Boue, biodéchet traité, sol et déchets - Détermination de la perte au feu

Ta slovenski standard je istoveten z: **prEN 15935**

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge

oSIST prEN 15935:2011 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 15935

December 2010

ICS 13.030.01

English Version

Sludge, treated biowaste, soil and waste - Determination of loss on ignition

Boue, biodéchet traité, sol et déchets - Détermination de la perte au feu

Schlamm, behandelter Bioabfall, Boden und Abfall - Bestimmung des Glühverlusts

This draft European Standard is submitted to CEN members for second enquiry. It has been drawn up by the Technical Committee CEN/TC 400.

If this draft becomes a European Standard, 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.

This draft European Standard was established by CEN 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 CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Warning : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

Foreword

This document (prEN 15935:2010) 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 document is currently submitted to the second CEN Enquiry.

This draft European Standard prEN 15935 was completely technically and editorially revised following the comments made during the first CEN-Enquiry in 2009 and the discussions from CEN/TC 400/WG 4 "Inorganic elements and compounds".

This European Standard is part of a modular horizontal approach in which this document belongs to the analytical step.

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.

Until now, test methods determining properties of materials within the environmental area were prepared in Technical Committees (TCs) working on specific products/matrices (e. g. soil, waste, sludge). However, it is understood that many steps within individual test procedures may also be used for the analysis of various other materials. By careful determination of these steps and selection of specific questions within these steps, elements of the test procedure can be described in a way that can be used for a variety of matrices and materials with certain specifications. This optimization is in line with the development among end-users of standards. A majority of routine environmental analyses are carried out by institutions and laboratories working under a scope that is not limited to one single environmental matrix but covers a wide variety of matrices. Availability of standards covering more matrices contributes to the optimization of laboratory procedures and standard maintenance costs, e. g. costs related to accreditation and recognition.

A horizontal modular approach was developed in the project 'Horizontal'. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole ”chain of measurement” (from sampling to analyses). A beneficial feature of this approach is that individual “modules” can be replaced by improved ones without jeopardizing the standard “chain”.

The results of the desk study as well as the evaluation and validation studies have been subject to discussions with all parties concerned in the CEN structure during the development by project 'Horizontal'. The results of these consultations with interested parties in the CEN structure have been presented to and discussed in CEN/BT TF 151.

Based on data from interlaboratory studies and consultations with interested parties within CEN member bodies, it has been concluded that this draft standard prEN 15935 is acceptable for its intended use and is ready for CEN enquiry.

It is recognized that standardization in the environmental field in most national standardization bodies is organized in national standardization committees that mirror the vertical structure of technical committees in the environmental field in CEN. The present CEN enquiry therefore asks for special attention by the NSBs to assure that the relevant and interested parties are consulted during the CEN enquiry, i. e. to assure that one single consolidated enquiry reply on this draft standard prEN 15935 can be presented by the NSB that covers the entire scope of this draft standard.

Introduction

This European Standard is validated for several types of matrices as indicated below (see also Annex A for the results of the validation):

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

Matrix	Validated for
Sludge	Municipal sludge
Biowaste	Compost, Fresh Compost
Soil	Sludge amended soil, Agricultural soil
Waste	Contaminated soil, Dredged sludge, Nickel sludge

WARNING — Persons using this European Standard should be familiar with normal 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. Special measures shall be taken during the ignition process to prevent contamination of the laboratory atmosphere by flammable, explosive or toxic gases.

1 Scope

This European Standard specifies a method for the determination of the loss on ignition (LOI) of dry mass at 550 °C. The dry matter has been determined in accordance with the method of EN 15934.

This method applies to the determination of loss on ignition of sludge, treated biowaste, soil and waste. The LOI of sediments can also be determined with this method.

NOTE The loss on ignition is often used as an estimate for the content of non-volatile organic matter in the sample. It should be noted that inorganic substances or decomposition products (e.g. H_2O, CO_2, SO_2, O_2) are released or absorbed and some inorganic substances are volatile under the reaction conditions.

2 Normative references

The following referenced documents are indispensable for the application 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.

prEN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter by determination of dry residue or water content*

3 Terms and definitions

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

3.1
loss on ignition
LOI
change in mass as a result of igniting a sample under specified conditions

NOTE The LOI is expressed as a weight percentage of the dry mass.

3.2
residue on ignition
mass remaining after igniting a sample under specified conditions

NOTE The residue on ignition is expressed as a weight percentage of the dry mass.

4 Principle

A dried test sample is heated in a furnace to constant mass at (550 ± 25) °C. The difference in mass before and after the ignition process is used to calculate the loss on ignition.

The determination is performed on a dried sample or directly on the un-dried sample including a drying step or by referring to the dry matter.

5 Interferences and sources of errors

LOI is an empirical parameter, thus in principle there is no interference connected to the determination. However, for some purposes the determination of LOI is used for the assessment of the content of organic matter in the sample. It should be noted that elementary carbon in the sample will be included in the loss on ignition value. Furthermore, any volatilisation or chemical reactions of inorganic compounds will also be included in the loss on ignition value.

Chemically bound water could be released during heating, thereby contributing to the loss on ignition.

Iron or other metals present in the sample in metallic state could be oxidised during heating, thereby producing lower results.

Sulfides present in the sample could be oxidised to sulfate during heating, thereby producing lower results.

Explosive ignition is likely to result in loss of residue from the crucible, thereby contributing to the loss on ignition.

Calcium hydroxide or calcium oxide present in large amounts (e.g. sludge conditioned with lime) may combine with sulfuric oxides liberated during ignition or with carbon dioxide formed during ignition producing lower results.

6 Apparatus

6.1 Crucible, typically 50 mm to 70 mm in diameter, suitable for ignition at 550 °C, e. g. made of nickel, platinum, porcelain, or silica.

6.2 Muffle furnace, or equivalent equipment, capable of maintaining a temperature of (550 ± 25) °C.

6.3 Metal plate, or comparable plate, suitable for the initial cooling of crucibles.

6.4 Desiccator, with an active drying agent, such as silica gel.

6.5 Precision balance, with an accuracy of 1 mg or greater.

7 Procedure

7.1 Samples with low content of volatiles

If the determination of dry matter and the determination of loss on ignition are carried out in successive operations in the same crucible refer to prEN 15934 for the initial crucible weighing. If not, the sample is a representative test portion of the dry mass obtained according to prEN 15934. Every necessary precaution shall be taken to avoid absorption of atmospheric humidity by the sample until weighed.

Place a crucible (6.1) in the furnace (6.2) and heat to (550 ± 25) °C for at least 30 min. Transfer the crucible from the furnace after initial cooling on a metal plate (6.3) to a desiccator (6.4) and finish cooling to ambient temperature. Weigh the empty crucible using a precision balance (6.5) to the nearest 1 mg.

Weigh into the crucible 0,5 g to 5 g of the dried test portion to the nearest 1 mg, and place the crucible into the furnace. Raise the furnace temperature to (550 ± 25) °C and hold this temperature for at least 1 h.

If the dry mass has high organic matter content, losses may occur as a result of rapid ignition or deflagration of the sample. In this case heat the sample slowly until ignition. For certain wastes (e.g. paper wastes and demolition wood) a step-wise heating process can be used: the crucible is inserted in a cold furnace; the temperature is raised slowly to 250 °C over a period of 50 min, allowing pyrolysis of the sample. Then the temperature is raised slowly to 550 °C and the 550 °C is kept for at least 2 h.

If the sample contains higher amounts of moisture, insert the crucible in a cold furnace and raise the furnace temperature evenly to (550 ± 25) °C over a period of 1 h and hold this temperature for at least 1 h.

Place the hot crucible containing the residue on ignition on a metal plate (6.3) for a few minutes. While still warm, transfer the crucible to a desiccator (6.4) and leave to cool to ambient temperature.

As soon as ambient temperature is reached, weigh the crucible containing the dry residue to the nearest 1 mg.

The crucible is weighed immediately after removal from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition and thus the loss on ignition shall be regarded as constant if the mass obtained after further 30 min of ignition at (550 ± 25) °C in the preheated furnace, differs not more than 0,5 % of the previous value or 2 mg, whichever is the greater. Otherwise repeat the ignition process.

In cases when even after the third ignition period constant mass is not obtained, record the value determined in the last of the three measurements. The lack of constant mass shall be reported together with the result.

If black carbon particles are still present (some organic substances burn slowly at 550 °C), wet the residue using a few drops of an ammonium nitrate solution. After repeated drying insert the crucible into the furnace and slowly heat to avoid losses by deflagration and continue heating the residue at (550 ± 25) °C. Ammonium

nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml distilled water. Both the value of loss on ignition obtained after the third ignition period and the value of loss on ignition obtained after addition of ammonium nitrate shall be given in the test report.

7.2 Samples containing volatile substances

For samples containing significant amounts of volatile substances the dry matter cannot be determined as dry residue. In this case the dry matter shall be calculated from the water content and the loss on ignition is always performed directly on the un-dried sample.

Place a crucible (6.1) in the furnace (6.2) and heat at (550 ± 25) °C for at least 30 min. Transfer the crucible from the furnace after initial cooling on a metal plate (6.3) to a desiccator (6.4) and finish cooling to ambient temperature. Weigh the empty crucible using a precision balance (6.5) to the nearest 1 mg.

Weigh into the crucible 0,5 g to 5 g of the test portion to the nearest 1 mg. Larger masses may be taken if complete combustion can be assured. All necessary precautions should be taken to avoid loss of volatiles from the samples until it has been weighed.

To avoid splashing from escaping vapours or sudden fire most of the volatile components should be removed from the sample at ambient temperature in a fume hood prior to ignition.

Samples containing highly flammable components e. g. solvents or waste oil should be ignited and allowed to burn in a fume hood before being inserted into the furnace.

When ready the crucible is inserted into a cold furnace and the temperature of the furnace is raised to (550 ± 25) °C and hold for at least 1 h.

Place the hot crucible containing the residue on ignition on a metal plate for a few minutes. While still warm, transfer the crucible to a desiccator (6.4) and leave to cool to ambient temperature. As soon as ambient temperature is reached, weigh the crucible containing the dry residue to the nearest 1 mg.

The crucible is weighed immediately after removal from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition and thus the loss on ignition shall be regarded as constant, if the mass obtained after further 30 min of ignition at (550 ± 25) °C in the pre-heated furnace, does not differ by more than 0,5 % of the previous value or 2 mg, whichever is the greater. Otherwise repeat the ignition process.

In cases when even after the third ignition period constant mass is not obtained, record the value determined in the last of the three measurements. The lack of constant mass should be reported together with the result.

If black carbon particles are still present (some organic substances burn slowly at 550 °C), wet the residue using a few drops of an ammonium nitrate solution. After repeated drying insert the crucible into the furnace and slowly heat to avoid losses by deflagration and continue heating the residue at (550 ± 25) °C. Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml distilled water. Both the value of loss on ignition obtained after the third ignition period and the value of loss on ignition obtained after addition of ammonium nitrate shall be given in the test report.

8 Calculation and expression of results

The loss on ignition of the dry mass of a solid sample is expressed in percent of the dry mass.

If the loss on ignition is performed on a dried sample the result shall be calculated from Equation (1):

$$w_{\mathrm{LOI}} = \frac{m_{\mathrm{c}} - m_{\mathrm{d}}}{m_{\mathrm{c}} - m_{\mathrm{a}}} \cdot 100 \qquad (1)$$

If the loss on ignition is performed directly on the un-dried sample the result shall be calculated from Equation (2):

$$w_{LOI} = 100 - \left(\frac{m_d - m_a}{m_b - m_a} .100 \right) \cdot \frac{100}{w_{dm}} \quad (2)$$

The residue on ignition of the dry mass of a solid sample expressed in percentage shall be calculated from Equation (3):

$$w_R = 100 - w_{LOI} \quad (3)$$

where

w_{LOI} is the loss on ignition of the dry mass of a solid sample, in percent (%);

w_R is the residue on ignition of the dry mass of a solid sample, in percent (%);

w_{dm} is the dry matter of the sample, in percent (%);

m_a is the mass of the empty dish or crucible in grams (g);

m_b is the mass of the dish or crucible containing the un-dried sample in grams (g);

m_c is the mass of the dish or crucible containing the dry matter in grams (g);

m_d is the mass of the dish or crucible containing the ignited sample in grams (g).

The results shall be rounded to the nearest 0,1 percent.

9 Precision data

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

10 Test report

The test report shall contain the following information:

a) a reference to this European Standard;

b) complete identification of the sample;

c) particular characteristics of the sample;

d) details of sample pre-treatment, if carried out;

e) expression of results, according to clause 8;

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