SLOVENSKI STANDARD
oSIST prEN 15935:2020

01-april-2020

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 15935:2020 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 15935

March 2020

ICS

Will supersede EN 15169:2007, EN 15935:2012

English Version

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

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

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

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European foreword

This document (prEN 15935:2020) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 15169:2007and EN 15936:2012.

Introduction

The method described in this document has been developed from EN 15169 (derived from EN 12879) which was prepared by CEN/TC 292 and from EN 15935:2012 which was prepared by CEN/TC 400.

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	Compost, Fresh Compost
Soil	Sludge amended soil, Agricultural soil
Waste	Contaminated soil, Dredged sludge (sediment), Nickel sludge

1 Scope

This document specifies a method for the determination of the loss on ignition (LOI) of dry matter at 550 °C. The dry matter is determined according to EN 15934.

This method applies to the determination of loss on ignition of sediment, sludge, treated biowaste, soil and waste.

NOTE The loss on ignition is often used as an estimate for the content of organic matter in the sample. 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 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 15934, *Sludge, treated biowaste, soil and waste - Calculation of dry matter fraction after determination of dry residue or water content*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
loss on ignition
LOI
mass fraction lost by incineration of a dried sample to constant mass at a specified temperature

3.2
residue on ignition
mass fraction remaining after incineration of a dried sample to constant mass at a specified temperature

4 Principle

A weighed test portion is incinerated 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 undried 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 elemental 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.

Samples containing iron or other metals in low bonding state or in metallic state could be oxidized during heating, thereby contributing to the loss on ignition with a negative amount.

Sulfides present in the sample could be oxidized to sulfate during heating, thereby contributing to the loss on ignition with a negative amount.

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 resulting in a too low content of the loss on ignition. Such reactions can be avoided using the stepwise heating procedure stated in 7.3 combined with a sufficient ventilation rate in the furnace and a height of the sample layer in the crucible not exceeding 5 mm.

6 Apparatus

6.1 Crucible, preferably flat bottom type and 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 Furnace, 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 Balance, with an accuracy of at least 1 mg

7 Procedure

7.1 Preservation

Preserve samples according to e.g. EN ISO 5667-15 or ISO 18512, as appropriate.

7.2 Pretreatment

Pretreat samples according to e.g. EN 15002 or EN 16179, as appropriate.

7.3 Determination

The determination of loss on ignition can be preformed using a

— wet sample;

— air-dried sample;

— sample after determination of dry matter.

The dry matter is determined according to EN 15934.

Place a crucible (6.1) in the furnace (6.2) and heat to (550 ± 25) °C and hold for at least 20 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, and place the crucible into the furnace. Raise the furnace temperature to (550 ± 25) °C and hold this temperature for at least 2 h. The sample layer in the crucible should not exceed a height of 5 mm.

If the test portion 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 2 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 ignition residue.

If black carbon particles are still present (some organic substances burn slowly at 550 °C), moisten the residue using a few drops of an ammonium nitrate solution. Insert the crucible into the furnace and slowly heat to avoid losses by the steam development 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 or demineralized water.

7.4 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 according to EN 15934 method B, and the loss on ignition is always performed on the undried 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 ensured. All necessary precautions should be taken to avoid loss of volatiles from the samples until it has been weighed.

To avoid splashing caused by 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 place the crucible into the cold furnace. Raise the furnace temperature to (550 ± 25) °C 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 ignition residue to the

nearest 1 mg.

If black carbon particles are still present (some organic substances burn slowly at 550 °C), moisten the residue using a few drops of an ammonium nitrate solution. Insert the crucible into the furnace and slowly heat to avoid losses by the steam development 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.

8 Calculation and expression of results

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

$$w_{\mathrm{LOI}} = \frac{m_{\mathrm{c}} - m_{\mathrm{d}}}{m_{\mathrm{c}} - m_{\mathrm{a}}} \times 100 \tag{1}$$

If the loss on ignition is performed directly on the undried or air-dried sample the result shall be calculated from Formula (2):

$$w_{\mathrm{LOI}} = \left(100 - \left(\frac{m_{\mathrm{d}} - m_{\mathrm{a}}}{m_{\mathrm{b}} - m_{\mathrm{a}}} \times 100\right)\right) \times \frac{100}{w_{\mathrm{dm}}} \tag{2}$$

The residue on ignition of the dry matter of a solid sample expressed in percent shall be calculated from Formula (3):

$$w_{\mathrm{R}} = 100 - w_{\mathrm{LOI}} \tag{3}$$

where

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

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

$w$dm is the dry matter fraction of the sample, in percent (%);

ma is the mass of the empty crucible, in grams (g);

mb is the mass of the crucible containing the undried sample, in grams (g);

mc is the mass of the crucible containing the dried sample, in grams (g);

md is the mass of the crucible containing the ignited sample, in grams (g).

9 Performance characteristics

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

10 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 15935);

b) complete identification of the sample;

c) details of sample pretreatment, if carried out;

d) expression of results, according to Clause 8;

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