SLOVENSKI STANDARD
SIST EN 15935:2021

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Nadomešča:
SIST EN 15169:2007
SIST EN 15935:2012

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: EN 15935:2021

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge

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EUROPEAN STANDARD

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EN 15935

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Supersedes EN 15169:2007, EN 15935:2012

English Version

Soil, waste, treated biowaste and sludge - Determination of loss on ignition

Sols, déchets, biodéchets traités et boues - Détermination de la perte au feu

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

This European Standard was approved by CEN on 4 July 2021.

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

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European foreword

This document (EN 15935:2021) has been prepared by Technical Committee CEN/TC 444 "Environmental characterization of solid matrices", the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by February 2022, and conflicting national standards shall be withdrawn at the latest by February 2022.

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

This document supersedes EN 15169:2007 and EN 15935:2012.

The following technical modifications have been made:

— two existing standards have been combined;

— the scope defines more sample types;

— the criteria for the heating time are specified.

This document specifies a method for the determination of the loss on ignition (LOI) at 550 °C of sediment, sludge, treated biowaste, soil and waste and combines the methods previously described in EN 15935:2012 and EN 15169.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The method described in this document has been developed from EN 15169 (derived from EN 12879), which was prepared by CEN/TC 292 “Characterization of waste” and from EN 15935:2012, which was prepared by CEN/TC 400 “Horizontal standards in the field of sludge, biowaste and soil”.

This document specifies a method for the determination of the loss on ignition (LOI) at 550 °C of sediment, sludge, treated biowaste, soil and waste and combines the methods previously described in EN 15935:2012 and EN 15169. Both dried and undried samples which are pretreated prior to determination can be used. The determination is ended after a predefined timeframe or when constant mass is reached.

This document 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 document 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) 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 https://www.electropedia.org/

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

3.1
loss on ignition
LOI
mass fraction lost by incineration of a dried sample to constant mass (3.3) at a specified temperature

3.2
residue on ignition
mass fraction remaining after incineration of a dried sample to constant mass (3.3) at a specified temperature

3.3
constant mass
mass obtained when the change in mass during a further period of heating of 1 h is within 0,5 % (m/m) or 2 mg, whatever is greater

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 can be oxidized during heating, thereby contributing to the loss on ignition with a negative amount.

Sulphides present in the sample could be oxidized to sulphate 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 with a positive amount. If such loss of residue is observed during the removal of the crucible from the furnace, the test shall be discharged and repeated taken the necessary precautions, e.g. using a smaller test portion.

Calcium hydroxide or calcium oxide present in large amounts (e.g. sludge conditioned with lime) can combine with sulphuric 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 7.4 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 Precision scale, 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.

For biological inactive samples special preservation may not be necessary. Biological active samples should be made inactive e.g. by freezing or air-drying.

7.2 Pretreatment

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

Depending of the origin, nature and appearance of the sample different procedures stated in e.g. EN 15002 or EN 16179 can be used:

a) Solid waste samples to be comminute and reduced to a granular powder, preferably a particle size less than 200 µm. If no volatile substances are present in the sample, sample may be air-dried prior to comminuting. The drying temperature of the air-drying shall not exceed 40 °C.

b) Moist or pasty-like waste samples may be mixed with aluminium oxide until a granular material is obtained and then comminuted, preferably to a particle size less than 200 µm. In this case the ratio of aluminium to sample shall be considered in the calculation of the loss on ignition.

c) Liquid samples shall be homogenized immediately prior to the weighing of the test portion.

Foreign bodies or non comminutable material (as metallic parts such as nuts, blots, scrap) should be separated from the sample and the weight and nature of the material recorded.

7.3 Determination

The determination of loss on ignition can be performed using

— an undried sample (see 7.4);

— an air-dried sample;

— a sample after determination of dry matter at (105 ± 5) °C, in successive operations using the same crucible.

The dry matter is determined according to EN 15934.

NOTE This procedure can be combined with the determined according to EN 15934 by using a crucible (6.1) dried at (105 ± 5) °C.

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 scale (6.5) to the nearest 1 mg.

Weigh into the crucible a test portion of 0,5 g to 5 g of the test sample to the nearest 1 mg, and place the crucible into the furnace. Larger masses may be taken if complete combustion can be ensured.

Raise the furnace temperature to (550 ± 25) °C 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 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 for at least 1 h.

NOTE Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml distilled or demineralized water.

The mass after at least 2 h heating at (550 ± 25) °C is considered constant.

7.4 Samples containing a high content of volatile or organic substances or undried samples

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 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 scale (6.5) to the nearest 1 mg.

Weigh into the crucible a test portion of 0,5 g to 5 g of the test sample 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.

Place the crucible into the cold furnace.

If the test portion has high organic matter content, losses can 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 temperature is raised slowly to 250 °C over a period of 50 min, allowing pyrolysis of the sample.

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 for at least 1 h.

NOTE Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml distilled water.

Ignition can be regarded as complete when a constant mass is obtained. If even after a third heating at (550 ± 25) °C constant mass is not obtained, use the value determined as the last measurement. This shall be recorded along with the result.

8 Calculation and expression of results

8.1 Loss on ignition, based on dry mass according to EN 15934 (7.3)

If the loss on ignition is performed in successive operation with the determination of dry residue (in the same crucible) the result shall be calculated from Formula (1):

$$w_{LOI} = \frac{m_c - m_d}{m_c - m_a} \times 100 \quad (1)$$

where

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

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

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

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