

Institut luxembourgeois de la normalisation de l'accréditation, de la sécurité et qualité des produits et services

ILNAS-EN 15935:2021

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

National Foreword

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ILNAS-EN 15935:2021**EN 15935**

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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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This European Standard exists 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-CENELEC Management Centre has the same status as the official versions.

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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of an identical text or by endorsement, at the latest by February 2022, and conflicting national standards shall be withdrawn at the latest by February 2022.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 $550\,^{\circ}$ 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 $550\,^{\circ}\text{C}$ 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: