SLOVENSKI STANDARD
SIST EN 15935:2012

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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

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English Version

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

Boues, bio-déchets traités, sols et déchets - Détermination de la perte au feu

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

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Foreword

This document (EN 15935:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.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	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 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. 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 matter at 550 °C. The dry matter is determined according to 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 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, 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*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15934 and the following apply.

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

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

4 Principle

A weighed test portion is burned up 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 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.

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

Sulfides present in the sample could be oxidised 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 too low a content of the loss on ignition. Such reactions can be avoided using the stepwise heating procedure stated in 7.1 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 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 at least 1 mg.

7 Procedure

7.1 Samples with low content of volatiles

If the determination of dry matter fraction and the determination of loss on ignition are carried out in successive operations in the same crucible refer to EN 15934 for the initial crucible weighing. If the determinations are performed as separate operations, representative portions of the sample shall be used for the determination of loss on ignition and dry matter fraction according to EN 15934. Alternatively the sample material may be dried according to EN 15934 before the determination of the loss on ignition. 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 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 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.

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 according to EN 15934 method B, and the loss on ignition is always performed directly 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 assured. 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):

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

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