SLOVENSKI STANDARD
SIST EN 15169:2007

01-september-2007

Karakterizacija odpadkov - Določ evanje ģarilne izgube odpadka, blata in sedimentov

Characterization of waste - Determination of loss on ignition in waste, sludge and sediments

Charakterisierung von Abfall - Bestimmung des Glühverlustes in Abfall, Schlamm und Sedimenten

Caractérisation des déchets - Détermination de la perte au feu des déchets, des boues et des sédiments

Ta slovenski standard je istoveten z: **EN 15169:2007**

ICS:

13.030.40	Naprave in oprema za odstranjevanje in obdelavo odpadkov	Installations and equipment for waste disposal and treatment

SIST EN 15169:2007 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15169

February 2007

ICS 13.030.40

English Version

Characterization of waste - Determination of loss on ignition in waste, sludge and sediments

Caractérisation des déchets - Détermination de la perte au feu des déchets, des boues et des sédiments

Charakterisierung von Abfall - Bestimmung des Glühverlustes in Abfall, Schlamm und Sedimenten

This European Standard was approved by CEN on 13 January 2007.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, 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.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 15169:2007: E

Contents

Page

Foreword

This document (EN 15169:2007) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, 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 August 2007, and conflicting national standards shall be withdrawn at the latest by August 2007.

The method described in this standard has been derived from EN 12879 which was prepared by CEN/TC 308.

Anyone dealing with waste and sludge analysis should be aware of the risks of that kind of material, irrespective of the parameters to be determined. Waste and sludge samples may contain hazardous (e. g. toxic, reactive, flammable, infectious) substances, which can be prone to biological and/or chemical reaction. Consequently these samples should be handled with special care. Gases which may be produced by microbiological or chemical activity are potentially flammable and will pressurise sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

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, 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.

1 Scope

This European Standard specifies a method for the determination of the loss on ignition.

This procedure is applicable to all kinds of waste, sludge and sediments.

The loss on ignition is often used as an estimate for the content of non-volatile organic matter in waste, sludge and sediments. It should be noted that any content of elementary carbon and volatilisation of organic materials or chemical reactions by inorganic compounds, is included in the loss on ignition.

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.

EN 14346, *Characterisation of 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
change in mass as a result of heating a sample under specified conditions. The loss on ignition is expressed in weight percent of the dry matter

3.2
residue on ignition
mass remaining after heating a sample under specified conditions. The residue on ignition is expressed in weight percent of the dry matter

3.3
dry residue
w_{dr}
remaining mass fraction of a sample after a drying process at 105 °C, as specified in EN 14346

3.4
water content
w_{w}
mass fraction of water in a sample determined by Karl-Fischer titration, as specified in EN 14346

3.5
dry matter
w_{dm}
mass fraction of a sample excluding water expressed as a percentage by mass, calculated by determination of dry residue or water content according to EN 14346

3.6
constant mass
constant mass is 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 test portion is heated in a furnace up to (550 ± 25) °C. The difference in mass before and after the heating process is used to calculate the loss on ignition.

The determination is performed on a beforehand dried sample following EN 14346 or directly on the undried sample including a drying step or by referring to the dry matter.

5 Interference

In principle there are no interferences connected to the determination, as the loss on ignition is an empirical parameter. However, for many purposes the determination is used for assessing the content of organic matter in waste, sludge or sediment samples. It should be noted that any 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 of ignition value.

NOTE 1 Chemically bound water or volatile metals (e. g. in hydroxides from flue gas cleaning processes) could be released during heating, contributing to the loss on ignition with a positive amount.

NOTE 2 Iron or other metals present in the sample in the metallic state could be oxidised during heating, contributing to the loss on ignition with a negative amount.

NOTE 3 Sulphides present in the sample could be oxidised to sulphate during heating, contributing to the loss on ignition with a negative amount.

6 Hazards

It is advised not to store samples in the open laboratory. If samples are to be stored, this has to be done in a well ventilated place.

7 Sample preservation and preparation

7.1 Preservation

When not analysed immediately samples should be stored in tight containers. For biologically inactive samples special preservation may not be necessary. Biologically active samples may need to have the lids loosened to prevent build-up of gas pressure.

If samples are known to be biologically active they should be made inactive e. g. by freezing or air drying.

7.2 Preparation

The samples supplied for analysis should be as homogeneous as possible.

Depending on the nature and appearance of the sample, different procedures can be used according EN 15002, e. g.

a) Solid samples may be directly comminuted (avoiding heat) and reduced to a granular powder, preferably a particle size less than 200 µm. If no volatiles are present samples may be air-dried prior to comminuting. The drying temperature shall not exceed 40 °C.

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

c) Liquid samples shall be homogenized immediately prior to weighing.

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

8 Apparatus

8.1 Flat bottom crucible, typically 50 mm to 70 mm in diameter, suitable for heating to 550 °C e. g. made of porcelain, silica, nickel or platinum.

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

8.3 Desiccator, containing a desiccant.

8.4 Analytical balance with an accuracy of 1 mg or better.

8.5 Metal plate.

9 Procedure

9.1 Samples not containing volatiles

The determination of loss on ignition and the determination of dry matter are performed as separate operations on different portions of the same sample. For some materials, however, it is convenient to carry out the determination of both dry residue and loss on ignition in successive operations in the same crucible. Procedure for dry residue see EN 14346.

In all cases necessary precautions should be taken to avoid absorption of atmospheric humidity by the sample until it is weighed.

Prepare a crucible (8.1) by placing it in the furnace (8.2) and heat at (550 ± 25) °C for at least 20 min. Transfer the crucible from the furnace (8.2) after initial cooling on a metal plate (8.5) to a desiccator (8.3) and finish cooling to ambient temperature. Weigh the empty crucible to constant mass to the nearest 1 mg *(m(a))* on an analytical balance (8.4).

Transfer 0,5 g to 5 g of the sample to the crucible and weigh to the nearest 1 mg *(m(b))*. Larger masses may be taken if appropriate.

NOTE 1 Masses larger than 5 g should not be used, unless it has been proven that a complete combustion of the actual sample material can be obtained at a higher loading.

If the determination of loss on ignition and determination of dry matter are performed in successive operations, weigh the crucible with the dried sample to the nearest 1 mg (m(d)). Insert the crucible into the furnace (8.2) and heat at (550 ± 25) °C for at least 1 h.

NOTE 2 If the dry matter is of high organic content, losses may occur as a result of rapid ignition or deflagration of the sample. In this case heat the sample slowly.

Remove the hot crucible from the furnace (8.2) and allow cooling on a clean metal plate (8.5) for a few min. While still warm, transfer the crucible to a desiccator (8.3) and finish cooling to ambient temperature. Weigh the crucible containing the ignition residue to the nearest 1 mg (*m(c)*) as soon as ambient temperature is reached.

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, record the value determined as the last measurement. This shall be recorded along with the result.

NOTE 3 If black carbon particles are still present (some organic substances burn slowly at 550 °C), wet the residue using a few drops of 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 to (550 ± 25) °C. Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml of demineralised water.

9.2 Samples containing volatiles

For samples containing significant amounts of volatile components dry matter can not be determined as dry residue. In this case the dry matter is calculated from the water content. For procedure see EN 14346. In this case the loss on ignition is always performed directly on the undried sample.

Prepare a crucible (8.1) by placing it in the furnace (8.2) and heat to (550 ± 25) °C for at least 20 min. Transfer the crucible from the furnace (8.2) after initial cooling on a metal plate (8.5) to a desiccator (8.3) and finish cooling to ambient temperature. Weigh the empty crucible to constant mass to the nearest 1 mg *(m(a))* on an analytical balance (8.4).

Transfer 0,5 g to 5 g of the sample to the crucible and weigh to the nearest 1 mg *(m(b))*. Larger masses may be taken if appropriate. All necessary precautions should be taken to avoid loss of volatiles from the sample until it is weighed.

NOTE 1 Masses larger than 5 g should not be used unless it has been proven that a complete combustion of the actual sample material can be obtained at a higher loading.

NOTE 2 To avoid splashing from escaping vapours or sudden fire it is recommended to carefully remove most of the volatile components from the sample into a drying oven or at ambient temperature into a fume hood prior to ignition.

NOTE 3 Samples of highly flammable waste e. g. solvents or waste oil should be ignited in the crucible and allowed to burn in a fume hood before being inserted into the furnace.

When ready the crucible is inserted into a cold furnace (8.2) and heated to temperature for 1 h.

Remove the hot crucible from the furnace (8.2) and allow it to cool on a clean metal plate (8.5) for a few minutes. While still warm, transfer the crucible to a desiccator (8.3) and finish cooling to ambient temperature. Weigh the crucible containing the ignition residue to the nearest 1 mg (*m(c)*) as soon as ambient temperature is reached.

Ignition is complete when constant mass is obtained. In cases when even after a third heating at (550 ± 25) °C constant mass is still not obtained, record the value determined as the last of the 3 measurements. This shall be reported along with the result.

NOTE 4 If black carbon particles are still present (some organic substances burn slowly at 550 °C), wet the residue using a few drops of 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 to (550 ± 25) °C. Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml of demineralised water.