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Priloga A: Postopek določanja izgube pri segrevanju

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

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

Sol, boue et biodéchet traité - Détermination de la perte au feu

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

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/SS S99.

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Foreword

This document (prEN 15935:2009) has been prepared by Technical Committee CEN/SS S99 "Health, environment and medical equipment - Undetermined", the secretariat of which is held by CMC.

The document is currently submitted to the CEN Enquiry.

This draft standard has been prepared by the European project «Horizontal » and presented to CEN/TC BTTF 151 "Horizontal Standards in the Fields of Sludge, Biowaste, and Soil", the secretariat of which is held by DS. Standardisation is carried out under mandate M330 given to CEN by the European Commission, and supports essential requirements of EU Directive(s).

The standard is part of a modular horizontal approach in which this standard belongs to the analytical step.

The results of the desk study as well as the evaluation and validation studies have been subject to discussions with all parties concerned in the CEN structure during the development by project Horizontal. The results of these consultations with interested parties in the CEN structure have been presented to and discussed in CEN BT/TF 151.

Based on data from interlaboratory studies and consultations with interested parties within CEN member bodies, it has been concluded that the draft standard is acceptable for its intended use and is ready for the CEN enquiry. The matrix "waste" was included during the entire process of development of the draft standard and is according to a resolution by TC 292 in 2008 included in the scope of the standard.

It is recognized that standardization in the environmental field in most national standardization bodies is organized in national standardization committees that mirror the vertical structure of technical committees in the environmental field in CEN. The present CEN enquiry therefore asks for a special attention by the NSBs to assure that the relevant and interested parties are consulted during the CEN enquiry, i.e. to assure that one single consolidated enquiry reply on the draft standard can be presented by the NSB that covers the entire scope of the draft standard.

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The standard is applicable and validated for several types of matrices as indicated below:

Material	Validated for (Type of sample, e.g. municipal sludge, compost)	References:
Sludge	Eight sludge samples Two municipal sludge, Germany	EN 12879:2001 – Characterization of sludges - Determination of the loss on ignition of dry mass Horizontal Project Interlab comp.
Soil	Muck soil, andosol, garden soil, loess under forest and sandy soil Sludge amended soil, Germany Agricultural soil, UK	EN 10694:1995 - Soil quality - Determination of organic and total carbon after dry combustion (elementary analysis) Horizontal Project Interlab comp. Horizontal Project Interlab comp.
Waste	Contaminated soil, dredged sludge, nickel sludge	EN 15169:2007 - Characterization of waste - Determination of loss on ignition in waste, sludge and sediments
Biowaste	Compost, Germany Compost, Austria	Horizontal Project Interlab comp. Horizontal Project Interlab comp.

Introduction

This draft standard is one of a number of draft standards developed and validated in the European project 'Horizontal' financed by the EU commission and EU member states. The project "Horizontal" was conducted by a European Consortium under the management by ECN, The Netherlands. This draft standard was presented by the project Horizontal to CEN/BT TF 151 in June 2008 with a view to be formally adopted as European Standard under CEN rules.

The standardisation by CEN is carried out on a mandate by the European Commission (Mandate M/330). The mandate considers standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants. It was the aim of the initiative to develop standards that are suitable for a wide range of environmental materials and lead to equivalent results as far as this is technically feasible.

Until now test methods determining properties of materials within the environmental area were prepared in Technical Committees (TCs) working on specific products/matrices (soil, waste, sludge etc). However, it is recognized that many steps in test procedures can be used in test procedures for other products/matrices. By careful determination of these steps and selection of specific questions within these steps, elements of the test procedure can be described in a way that can be used for more matrices and materials with certain specifications. This optimization is in line with the development among end-users of standards. A majority of routine environmental analyses are carried out by institutions and laboratories working under a scope which is not limited to one single environmental matrix but covers a wide variety of matrices. Availability of standards covering more matrices contributes to the optimization of laboratory procedures and standard maintenance costs, e.g. cost related to accreditation and recognition.

A horizontal modular approach was developed in the project 'Horizontal'. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole "chain of measurement" (from sampling to analyses). A beneficial feature of this approach is that "modules" can be replaced by better ones without jeopardizing the standard "chain".

The modules that relates to this standard are specified in section 2 - Normative references.

1 Scope

This European Standard specifies a method for the determination of the loss on ignition of dry mass at 550 °C after the dry matter has been determined in accordance with the method of WI CSS99023.

This method applies to the determination of loss on ignition of:

- sludges, including liquid, paste-like or solid sludges;
- all types of soil samples;
- sediments;
- waste and
- treated biowaste.

NOTE The loss on ignition is often used as an estimate for the content of non-volatile organic matter in the sample. It should be noted that inorganic substances or decomposition products (e.g. H₂O, CO₂, SO₂, O₂) are released or absorbed and some inorganic substances are volatile under the reaction conditions.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

CSS99031 Sludge, treated biowaste, and soils in the landscape – Sampling – Framework for the preparation and application of a sampling plan

CSS99058 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 1: Guidance on selection and application of criteria for sampling under various conditions

CSS99057 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 2: Guidance on sampling techniques

CSS99032 Sludge, treated biowaste, and soils in the landscape – Sampling - Part 3: Guidance on sub-sampling in the field

CSS99059 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

CSS99060 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 5: Guidance on the process of defining the sampling plan

CSS99035 Soil, sludge and treated biowaste – Pre-treatment for inorganic and organic characterization

CSS99022 Soil, sludge, waste, and treated biowaste – Determination of dry matter – Gravimetric method

3 Terms and definitions

For the purpose of this European Standard, the following terms and definitions apply.

3.1

loss on ignition (LOI)

change in mass as a result of heating a sample under specified conditions

NOTE The loss on ignition (LOI) is expressed as a weight percentage of the dry mass.

3.2

residue on ignition

mass remaining after heating a sample under specified conditions. The residue on ignition is expressed as a weight percentage of the dry mass.

3.3

dry matter, w_{dm}

dry residue after drying according to the specified drying process. It is expressed as a percentage or in grams per kilogram

3.4

constant mass

constant mass is reached when the change in dry mass during a further period of heating of 1 hour is within 0,5 % (m/m) or 2 mg, whichever is the greater

4 Safety remarks

Samples of sludge, waste, bio-waste or contaminated soil are liable to ferment and may contain harmful microorganisms. Consequently it is recommended that these samples should be handled with special care. The

gases, which may be produced by microbiological activity, are potentially inflammable and will pressurise sealed bottles. Exploding bottles are likely to result in infectious shrapnel and/or pathogenic aerosols.

Special measures must be taken during the ignition process to prevent contamination of the laboratory atmosphere by flammable, explosive or toxic gasses.

5 Principle

A dried test sample is heated in a furnace to constant mass at $(550 \pm 25)^\circ\text{C}$.

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 un-dried sample including a drying step or by referring to the dry matter.

6 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.

NOTE 1 Chemically bound water could be released during heating, thereby contributing to the loss on ignition.

NOTE 2 Iron or other metals present in the sample in metallic state could be oxidised during heating, thereby producing lower results.

NOTE 3 Sulphides present in the sample could be oxidised to sulphate during heating, thereby producing lower results.

NOTE 4 Explosive ignition is likely to result in loss of residue from the crucible, thereby contributing to the loss on ignition.

NOTE 5 Calcium hydroxide or calcium oxide present in large amounts (e.g. sludge conditioned with lime) may combine with sulphuric oxides liberated during ignition or with carbon dioxide formed during ignition producing lower results.

7 Apparatus

7.1 Crucible

Typically 50 mm to 70 mm in diameter, suitable for ignition at 550°C , e.g. made of nickel, platinum, porcelain, or silica.

7.2 Muffle furnace

Or equivalent equipment, capable of maintaining a temperature of $(550 \pm 25)^\circ\text{C}$.

7.3 Metal plate

Suitable for the initial cooling of crucibles.

7.4 Desiccator

With an active drying agent, such as silica gel.

7.5 Analytical balance

With an accuracy of 1 mg or greater.

8 Sampling and sample pre-treatment

8.1 Sampling

Sampling should be carried out in accordance with relevant international standards – See Clause 2.

During storage, samples may be subject to changes (e.g. uptake or liberation of water, carbon dioxide and other volatiles), which are liable to falsify the results. Biological active samples should be analysed within 3 days. If analysed within this period, the samples should be stored at about 4 °C; or otherwise stored directly at maximum -18°C. Other samples may be stored in a closed container in a well-ventilated place.

8.2 Sample pre-treatment

Sampling and sample pre-treatment shall be carried out in accordance with the relevant methods as specified in Clause 2.

8.3 Sludge, sediment, soil, and waste with low content of volatiles

If the determination of dry matter and the determination of loss on ignition are carried out in successive operations in the same crucible refer to CSS 99022 for the initial crucible weighing. If not, the sample is a representative portion of the dry mass obtained according to CSS 99022. Every necessary precaution shall be taken to avoid absorption of atmospheric humidity by the sample until weighed.

Place a crucible (see 7.1) in the furnace (see 7.2) and heat at (550 ± 25) °C for at least 30 minutes. Transfer the crucible from the furnace (see 7.2) after initial cooling on a metal plate (see 7.3) to a desiccator (see 7.4) and finish cooling to ambient temperature. Weigh the empty crucible to the nearest 1 mg, (m_a).

Weigh into the crucible 0.5 g to 5 g of the dried sample to the nearest 1 mg, (m_b), and raise the furnace temperature to (550 ± 25) °C and hold this temperature for at least 1 hour.

NOTE 1 If the dry mass 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 minutes (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.

NOTE 2 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 hour and hold this temperature for at least 1 hour.

Place the hot crucible containing the residue on ignition on a metal plate (see 7.3) for a few minutes. While still warm, transfer the crucible to a desiccator (7.4) and leave to cool to ambient temperature.

As soon as ambient temperature is reached, weigh the crucible containing the dry residue to the nearest 1 mg (m_c).

The crucible is weighed immediately after removal from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition and thus the loss on ignition shall be regarded as constant if the mass obtained after a further half-hour period of ignition at (550 ± 25) °C in the preheated furnace, ($m_c - m_a$), differs max. 0,5 % of the previous value or 2 mg, whichever is the greater (see 3.5).

NOTE 3 In cases when even after the third ignition period constant mass is not obtained, record the value determined in the last of the three measurements. The lack of constant mass should be reported together 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 an 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 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. Both the value of loss on ignition obtained after the third ignition period and the value of loss on ignition obtained after addition of ammonium nitrate shall be given in the test report.