SLOVENSKI STANDARD
oSIST prEN 15934:2009

01-julij-2009

Tla, blato, odpadki in bioodpadki - Določ evanje suhe snovi - Gravimetrijska metoda

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

Schlamm, Boden, Abfall und behandelter Bioabfall - Bestimmung der Trockenmasse - Gravimetrisches Verfahren

Sol, boue et biodéchet traité - Détermination de la teneur pondérale en matière sèche - Méthode gravimétrique

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Ta slovenski standard je istoveten z: **prEN 15934**

ICS:

13.030.01	Odpadki na splošno	Wastes in general

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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 15934

April 2009

ICS

English Version

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

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

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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Foreword

This document (prEN 15934:2009) 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.

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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.

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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.

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)**	**Reference:**
Sludge	Eight sludge samples Municipal sludge, North Rhine Westphalia	EN 12880:2000, Annex Horizontal Project Interlab comp.
Soil	Soil samples Sludge amended soil, Barcelona	ISO 11465:1993 Horizontal Project Interlab comp.
Biowaste	Fresh compost, Vienna	Horizontal Project Interlab comp.
Waste	Contaminated soil, dredged sludge, nickel sludge	EN 14346:2006

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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.

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1 Scope

This European Standard specifies a method for the determination of dry matter on a mass basis of samples of:

— sludges, including liquid, paste-like or solid sludges,

— all types of air-dried soil samples and field moist soil samples,

— sediment

— waste and

— treated biowaste.

NOTE Determination of water content of a sample using this method is possible provided that other compounds other than water do not contribute significantly to the weight loss by heating to 105 ºC.

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).

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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, S*oil, sludge, and treated biowaste – Pre-treatment for inorganic and organic characterization*

CSS99023, S*oil, sludge and treated biowaste – Determination of loss on ignition*

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3 Terms and definitions

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

3.1
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

3.2
water content w_{wc}
mass fraction determined as the loss on mass after the specified drying process. It is expressed as a percentage or in grams per kilogram

3.3
constant mass
mass reached when, during the drying process, the difference between two successive weighings of the sample at an interval of minimum 1 h, first heated, then cooled to room temperature, does not exceed 0,5 % (m/m) of the last determined mass or 2 mg, whichever is the greater

NOTE 1 These definitions do not - for technical reasons - apply to samples containing volatile substances.

NOTE 2 Usually 16 h to 24 h are sufficient for most soil, sludge, sediment and waste samples, but certain sample types and large samples may require longer drying periods.

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4 Safety remarks

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Samples of sludge, waste, bio-waste or contaminated soils are liable to ferment and usually contain harmful microorganisms. It is essential to keep them away from any food or drink, and to protect any cuts. Bursting bottles containing e.g. sludge can produce microorganisms-contaminated shrapnel and/or infectious aerosols.

When handling sludge, waste and bio-waste samples, it is necessary to wear gloves, face and eye protection, and sufficient body protection to guard against bottles bursting. Gasses evolved may be flammable.

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

5 Principle

Samples are dried to constant mass in an oven at (105 ± 5) °C. The difference in mass before and after the drying process is used to determine the dry matter and the water content.

6 Interferences and sources of errors

The samples may change chemically during the drying process (e.g. by absorption of carbon dioxide in the case of basic samples or of oxygen caused by reducing substances).

NOTE 1 When determining the water content, volatile substances (such as organic solvents or substances deriving from the decomposition of organic or inorganic substances) are also included either completely or partially.

NOTE 2 In case of samples with a high content of solids (e.g. dry matter $w_{dr} \geq 30\%$) there is the risk of water still remaining trapped in the sample after drying.

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NOTE 3 Decomposition of organic matter can, in general, be neglected at this temperature. However, for soil samples with a high content of organic matter (> 10% (m/m)), for example peaty soils, the method of drying should be adapted. In this case, the sample should be dried to constant mass at 50°C. Use of a vacuum will speed up the operation.

NOTE 4 Some minerals similar to gypsum lose water of crystallisation at a temperature of 105 °C.

7 Apparatus

7.1 Drying oven

Thermostatically controlled with forced air ventilation, maintaining a temperature of (105 ± 5) °C.

7.2 Desciccator

With active drying agent such as silica gel.

7.3 Temperature tolerant evaporating dish or crucible

Withstanding at least 105°C for dry matter analyses or 550°C for further analyses of loss on ignition is required (WI CSS 99023). Suitable materials are nickel, porcelain, silica, and platinum.

7.4 Analytical balance

With an accuracy of 1 mg or better. For samples with dry residue of 10 g or higher, an analytical balance with an accuracy of 10 mg may be used.

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8 Sampling and sample pre-treatment

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

9 Procedure

Place an evaporating dish or crucible (see 7.3) in the drying oven (see 7.1) set at (105 ± 5) °C for a minimum of 30 minutes and then cool to ambient temperature in a desiccator (see 7.2), with the lid closed. After cooling, weigh the dish or crucible to the nearest 1 mg, m_a.

If the same crucible is to be used for the subsequent loss on ignition measurement (WI CSS 99023), it shall be pre-ignited at 550°C for a minimum of 30 min.

Depending on the expected water content, weigh into the evaporating dish or crucible (see 7.3) a suitable amount of sample, m_b, so that the dry matter obtained has a mass of not less than 0,5 g.

NOTE 1 Sample amounts of 30 g to 50 g are suitable for field-moist soil samples, paste-like and solid sludge and solid waste. A larger test portion may be needed to assure a representative sample for e.g. composted bark samples. For air-dried soil samples 10 g to 15 g are suitable.

NOTE 2 Determination of dry matter shall be determined on samples identical to those used for determination of parameters that relates to dry matter.

Place the evaporating dish or crucible (see 7.3) containing the sample in the drying oven (see 7.1) set at 105 °C until the residue appears dry, typically overnight.

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NOTE 3 There is a risk of a cake surface forming. The formation of such cake surface impedes even drying. To avoid this, a glass rod can be weighed along with the crucible. If cake formation occurs during drying, the glass rod is used to stir the sludge to break up the cake and bring the liquid surface into contact with hot air. This is repeated as necessary.

NOTE 4 In the case of samples containing considerable amounts of water careful evaporation of the major part of the water is preferred (e.g. in a water bath) in order to avoid loss of substances by splashing. Alternatively freeze-drying may be used as a first step for the determination of dry matter.

After cooling in the desiccator (see 7.2), weigh the evaporating dish or crucible and contents for the first time, m_c.

The mass ($m_c - m_a$) shall be regarded as constant if the mass obtained after another hour of drying does not differ by more than 0.5% of the previous value or 2 mg, whichever is the greater (see 0).

Otherwise, repeat drying until constant mass is reached.

NOTE 5 In cases when even after the third drying process it is not possible to obtain a constant value, record the value determined after at further 2 h together with a remark on the unfinished process.

NOTE 6 20 hours of drying and omission of re-drying/re-weighing can be applied for sample types with documented evidence that the necessary drying time is less than 20 hours.

10 Quality control

At least one duplicate analysis should be carried out in each batch of analyses. Where uncertainty exists about the homogeneity or behaviour of the sample it is recommended that the analysis be carried out in duplicate.

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11 Calculation and expression of results

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Calculate the dry matter (w_{dm}) or the water content (w_{wc}) expressed as a percentage of mass or grams per kilogram using the following equations:

$$w_{dm} = \frac{m_c - m_a}{m_b - m_a} \times f$$

$$w_{wc} = \frac{m_b - m_c}{m_b - m_a} \times f$$

where:

w_{dm} = is the dry matter of the sample, in percentages or grams per kilogram;

w_{wc} = is the water content of the sample, in percentages or grams per kilogram;

m_a = is the mass of the empty dish or crucible in grams;

m_b = is the mass of the dish or crucible containing the sample in grams;

m_c = is the mass of the dish or crucible containing the dry matter in gram

f is a conversion factor, f = 100 for expression of results as a percentage and factor f = 1 000 for expression in grams per kilogram.

Values should be rounded to the nearest 0,1% (w/w) or alternatively to the nearest 1 g/kg.