SLOVENSKI STANDARD
oSIST prEN 15936:2009

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Tla, blato, odpadki in bioodpadki - Določevanje celotnega organskega ogljika (TOC) s suhim sežigom

Soil, sludge, waste, and treated biowaste - Determination of total organic carbon (TOC) by dry combustion

Schlamm, Boden, Abfall und behandelter Bioabfall - Bestimmung des gesamten organischen Kohlenstoffs (TOC) durch trockene Verbrennung

Sol, boue et biodéchet traité - Détermination de la teneur en carbone organique total (COT) par combustion sèche

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13.030.01	Odpadki na splošno	Wastes in general

oSIST prEN 15936:2009 **en**

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ICS

English Version

Soil, sludge, waste, and treated biowaste - Determination of total organic carbon (TOC) by dry combustion

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Contents

Foreword

This document (prEN 15936:2009) 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.

The document is technical equivalent to EN 13137 (covering the matrices waste, sludge and sediment), however, the matric soil is included. A new method is included in Annex C describing the suspension method. Annex C is so far informative, as a decision on including the principle in the final standard is recommended to depend on the availability of validation.

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	Two samples of municipal sludge, Germany	EN 13137:2001, Characterisation of waste – Determination of total organic carbon (TOC) in waste, sludges and sediments
Soil	Sludge amended soil, Germany Agricultural soil, UK	JRC report and report on validation by project Horizontal
Biowaste	Compost, Vienna, Austria Compost, Germany	JRC report and report on validation by project Horizontal
Sediment	See Annex A	EN 13137:2001, Characterisation of waste – Determination of total organic carbon (TOC) in waste, sludges and sediments
Waste	See Annex A	EN 13137:2001, Characterisation of waste – Determination of total organic carbon (TOC) in waste, sludges and sediments

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 two methods for the determination of total organic carbon (TOC) in sludge, sediment, waste, biowaste and soil samples containing more than 1 g carbon per kg of dry matter (0,1%).

Coal and charcoal (elemental carbon) and inorganic carbon compounds except carbonates will be determined as organic carbon when present in the sample.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the 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).

ISO 8466-1, *Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics - Part 1: Statistical evaluation of the linear calibration function*

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

CSS99034 Soil, sludge, and treated biowaste – Pre-treatment for solid samples

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

ISO 10693, Soil quality – Determination of carbonate content – Volumetric method

ISO 3733, Petroleum products and bituminous materials - Determination of water - Distillation method

ISO 6296, Petroleum products - Determination of water - Potentiometric Karl Fischer titration method

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply:

3.1
Total Carbon (TC)
quantity of carbon present in the sample in the form of organic, inorganic and elemental carbon according to this standard

3.2
Total Inorganic Carbon (TIC)
quantity of carbon that is liberated as carbon dioxide by acid treatment according to this standard

3.3
Total Organic Carbon (TOC)
quantity carbon that is converted into carbon dioxide by combustion according to this standard and which is not liberated as carbon dioxide by acid treatment according to this standard

4 Safety remarks

Samples may be liable to fermentation and may be infectious. Due to this it is recommended to handle these samples with special care. The gases, which may occur due to the microorganisms activities, are potentially flammable. Excessive pressure build-up may cause the sample container to burst, potentially resulting in the formation of infectious aerosols and contaminated shrapnel.

Harmful compounds may arise during the combustion process and during the acid treatment. The user has to take appropriate precautions (e.g. activated carbon filters) to avoid these getting into the laboratory environment.

Samples with a high organic content may explode at introduction into the furnace. Using less sample material or covering the sample with inert material can reduce this risk.

5 Principle

5.1 General

The TOC can be measured either by Method A (indirect procedure) or by Method B (direct procedure).

5.2 Method A (indirect procedure)

In this procedure the TOC is obtained by the difference between the results of the measurements of TC and TIC.

The total carbon (TC) present in the un-dried sample or dried sample is converted to carbon dioxide by combustion in an oxygen-containing gas flow free of carbon dioxide. For soil dried samples are used. To ensure complete combustion, catalysts and/or modifiers can be used. The released amount of carbon dioxide is measured by infrared spectrometry, gravimetry, coulometry, conductometry, thermal conductivity detection, flame ionisation detection after reduction to methane, or other suitable techniques.

The TIC is determined separately from another sub-sample by means of acidification and purging of the released carbon dioxide. The carbon dioxide is measured by one of the techniques mentioned above. Alternatively, for soil the total organic carbon content may be calculated by determining the total carbon content and subtracting the carbon present as carbonate, which can be determined according to ISO 10693 (volumetric method).

5.3 Method B (direct procedure)

In this procedure the carbonates present in the un-dried or dried sample are previously removed by treating the sample with acid. The carbon dioxide released by the following combustion step is measured by one of the techniques mentioned in 5.2 and indicates the TOC directly.

5.4 Applicability of Methods A or B

Methods A and B have the same applicability in the terms of TOC content and/or TIC to TOC ratio. In samples with relatively high inorganic carbon contents method B is preferred.

Method B may lead to incorrect results in the following cases:

— the sample contains volatile substances that evaporate during the acidification (e.g. volatile hydrocarbons from sludge of oil separators);

— side reactions between the sample and the acid take place (e.g. decarboxylation, volatile reaction products).

The quality of results of Method B is dependent on experience and practise, especially regarding the steps before the determination of TOC. Use of automatic dispensing units regarding removal of carbonates prior to determination of TOC may improve the performance of Method B.

6 Interferences and sources of errors

Volatile organic substances may be lost during sample preparation. If necessary, the carbon content resulting from volatile organic substances shall be determined separately.

Depending on the laboratory experience with samples containing high amounts of carbonate the procedures may lead to unreliable TOC results if the TIC to TOC ratio is very high (e.g. ≥ 10).

Depending on the detection method used, different interferences may occur, for instance:

— the presence of cyanide can interfere with the coulometric detection of TIC by modifying the pH value (dissolution of HCN);

— high content of halogenated compounds may lead to an overestimation of TOC when coulometric detection is used; in some cases the classical silver or copper trap can be insufficient to absorb all halides.

When present, elemental carbon, carbides, cyanides, cyanates, isocyanates, isothiocyanates and thiocyanates are determined as organic carbon using the methods described in this standard. An interpretation of the measured value may therefore be problematic in cases where the sample contains relevant levels of the above-mentioned components. If needed, these components shall be determined separately by means of a suitable validated procedure and be recorded in the test report.

Elementary carbon, determined separately, may be subtracted if required for the sample. If this is done this shall be reported by the laboratory.

7 Reagents

7.1 General

All reagents used shall be at least of analytical grade and suitable for their specific purposes.

Hygroscopic substances shall be stored in a desciccator.

7.2 Calcium carbonate

Calcium carbonate, $CaCO_3$.

7.3 Sodium carbonate

Sodium carbonate Na_2CO_3, anhydrous.

7.4 Tetrasodium ethylenediamine tetraacetate-tetra-hydrate

Na_4-EDTA·$4H_20$ - $C_{10}H_{12}N_2O_8Na_4 \cdot 4H_2O$, heated at 80°C for two hours.

NOTE Other forms of Na_4-EDTA hydrates may be used if the water content is exactly known. Then the composition of the control mixtures has to be recalculated accordingly (see also 7.11 and7.12).

7.5 Potassium hydrogen phthalate

Potassium hydrogen phthalate, $C_8H_5O_4K$

7.6 Acetanilide

Acetanilide, C_8H_9NO.

7.7 Atropine

Atropine, $C_{17}H_{23}NO_3$.

7.8 Spectrographic graphite powder

Spectrographic graphite powder, C.

7.9 Sodium salicylate

Sodium salicylate, $C_7H_50_3Na$.

7.10 Aluminium oxide

Aluminium oxide, Al_2O_3, neutral, granular size < 200 µm, annealed at 600°C.