SLOVENSKI STANDARD
SIST EN 16718:2016

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Les in lesni proizvodi - Delež celotnega organskega ogljika (TOC) v lesu in lesnih proizvodih

Wood and wood based products - Dosage of the total organic carbon (TOC) in wood and wood based products

Holz und Holzprodukte - Bestimmung des gesamten organischen Kohlenstoffs (TOC) in Holz und Holzprodukten

Produits de préservation du bois et matériaux à base de bois - Dosage du carbone organique total (COT) dans les bois et matériaux à base de bois

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Wood and wood based products - Dosage of the total organic carbon (TOC) in wood and wood based products

Produits de préservation du bois et matériaux à base de bois - Dosage du carbone organique total (COT) dans les bois et matériaux à base de bois

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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European foreword

This document (EN 16718:2015) has been prepared by Technical Committee CEN/TC 38 “Durability of wood and wood-based products”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

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Introduction

Bio-based products from forestry and agriculture have a long history of application. The last decades have seen the emergence of new bio-based products in the market. Acknowledging the need for common standards for bio-based products, the European Commission issued mandate M/492, resulting in a series of standards developed by CEN/TC 411.

For business to business transactions, claims which are relevant to describe characteristics of bio-based products in a business to business environment will be given in the near future. Data are by consequence required to generate and transfer information in the industrial chain and/or as an input for product specific standards and certification schemes.

The work to be done by the CEN/TC 411/WG 3 concerns the determination of the bio-based carbon in order to determine the level of bio-based content of a product or materials. A document (CEN/TR 16721) has been prepared by Technical Committee CEN/TC 411 "Bio-based products", and describes a list of methods and an "overview of methods to determine the bio-based content and related methods" for Bio-based products.

As part of the task force of CEN/TC 175, devoted to carbon foot printing and LCA, a European Standard was published on the simplified calculation of the amount of biomass carbon stored in wood (using 50 % of the anhydrous wood mass): EN 16449.

This standard EN 16718 describes the methods based on analytical measurements. These methods can be considered as complementary to the radiocarbon based method and methods based on evaluation by calculation (mass balance approaches). One of these analytical methods is a method based on measurement of stable isotopic ratio present in biomass in order to determine the biomass content of the product.

The development of this method described in this report is ongoing with close collaboration between FCBA and the "Institute des Sciences Analytiques" CNRS in order to determine the bio-based content of wood raw materials, glues and panels made with these raw materials for end use manufactured products with this new method. The objective is to propose correlated analysis (with the TOC method proposed by FCBA) to determine the carbon content to purpose a quick and low cost method easy to handle.

References:

— http://www.biobasedeconomy.eu/standardisation/cen-tc411/

— http://www2.afnor.org/espace_normalisation/structure.aspx?commid=86489

The tests that have resulted in the specification of this document were performed in the context of work conducted by the FCBA [timber certification body] Technological Institute aimed at determining a method for supplying data on organic carbon contents that could be used to calculate carbon balances.

The storage of biomass carbon in wood-based products is the preservation of the carbon absorbed by the tree from atmospheric CO_2 through photosynthesis.

The carbon thus captured in the material is of benefit to the climate throughout the lifespan of the product, which can be several dozen years for a construction product, for example. The French Standard NF P01-010 (2004), which lays out the format of environmental and health statements (FDES) for construction products, provides the option of indicating the following supplementary information, in addition to the "Climate change" indicator, which is calculated from the flows of greenhouse gases associated with the product life cycle: "for some construction products (e.g. plant-based products), CO2 storage during the "service life" stage can be given if measurements are taken based on standardized test methods."

Furthermore, the Guide to Best Practices on environmental labelling of mass-market consumer products (BP X30-323) includes in Annex G: “Carbon accounting integrating time lag” which also requires knowledge of the biomass carbon contents of the products.

The purpose of this document is therefore to propose a laboratory measurement method of the amount of biomass carbon that will provide values of carbon or CO_2 equivalent stored in wood-based products, with the aim to integrate this information in the environmental statements of these products according to the texts referenced above.

While measurement is not systematically necessary for solid wood products, for example, given the common knowledge on the densities of the various wood species and on the proportion of carbon contained in wood, this experimental measurement may prove to be necessary for products made of wood-based composite materials.

The organic carbon contained in wood and wood-based materials is found in several different forms. Cumulative measurements, such as total organic carbon (TOC), need to be used. Isotopic ratio enables the differentiation between synthetic and natural products. IRMS (Isotope Ratio Mass Spectrometer) is a complementary method to the TOC method by an identification of the isotope ^{13}C: both techniques are necessary to give reliable data on a bio-based content on a wood based material such as panel, board, and woods containing chemicals in general. A study is currently in progress in France on wood based materials: the results will enable to improve this present document and to give data with multi-isotopic determinations (^{13}C, ^{15}N, ^{2}H, ^{18}O).

1 Scope

This European Standard describes a method for determining total organic carbon by calculating the difference between the results of measurements of total carbon (TC) and total inorganic carbon (TIC). The identification of the bio-based content given by the stable isotopes such as ^{13}C is described also.

This method is applicable to all wood species, wood-based materials (panels, plywood, wood-polymer, etc.) and woods containing chemicals in general.

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 13183-1, *Moisture content of a piece of sawn timber — Part 1: Determination by oven dry method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
total carbon
TC
amount of carbon found in waste, in organic, inorganic and elemental-state forms

3.2
total inorganic carbon
TIC
amount of carbon released as carbon dioxide through acidification

3.3
total organic carbon
TOC
carbon that is transformed into carbon dioxide through combustion and not released through acidification as carbon dioxide

Note 1 to entry: The definitions given above are only applicable in this document and only partly overlap the scientific definitions of TC, TOC and TIC.

4 Principle

In this procedure, TOC is obtained by subtraction between the measurement results of TC and TIC.

The total carbon (TC) present in the undried sample is transformed into carbon dioxide through combustion in a flow of gas that contains oxygen and is free of carbon dioxide. To ensure combustion is total, catalysers and/or modifiers can be used. The amount of carbon dioxide released is measured using infrared spectrometry, gravimetry, coulometry, conductometry, thermal conductivity detection or flame ionization detection after reduction to methane, or any other appropriate technique.

The TIC is determined separately using another sub-sample, through acidification and purging of the released carbon dioxide, which is then measured using one of the techniques mentioned above.

The ^{13}C identification by IRMS is described in 7.7. This protocol is able also to work on other isotopes: ^{15}N, ^{2}H and ^{18}O, which could be useful for complex materials containing wood.

5 Reagents and products

All the reagents used shall be of analytical grade at least and suitable for their specific uses. Hygroscopic products shall be kept in a dessicator.

5.1 Glucose, $C_6H_{12}O_6$.

5.2 Anhydrous sodium carbonate, Na_2CO_3.

5.3 Non-oxidizing mineral acid used to release the carbon dioxide, e.g. phosphoric acid H_3PO_4, (m = 85 %).

5.4 Synthetic air, nitrogen, oxygen, argon, free of carbon dioxide and organic impurities, according to the instructions supplied by the machine manufacturer.

6 Apparatus

6.1 Homogenization device, such as mixers, stirrers.

6.2 Analytical balance accurate to at least 0,5 % of test portion weight.

6.3 Apparatus for dosing carbon in solid matter, along with its accessories.

6.4 Purging device for dosing TIC.

6.5 Mixer mill.

7 Procedure

7.1 Preparation of the sample

Before the sample preparation, the sampling program shall be properly designed in accordance with the context of the testing and objectives.

The samples to be analysed should be as homogeneous as possible and undried.

The samples of wood or wood-based materials can be directly ground (avoiding any heating) and reduced to powder, preferably with a particle size below 500 µm. The samples are ground in their entire thickness.

The samples that contain negligible concentrations (taking into account the accuracy of the method used) of volatile compounds other than water can be dried at 105 °C before they are homogenized.

7.2 Water content

The determination of moisture content shall be carried out using a different test portion. It can be calculated from the dry matter mass, determined according to EN 13183-1.

NOTE Any other determination method (e.g. with a desiccator's balance) can be used if it has been previously validated.

7.3 Dosage

7.3.1 General

This document does not give any recommendations for the apparatus and its mode of operation. The operating conditions should be selected and verified according to the instructions supplied by the manufacturer.

It is advisable to select a test portion with the greatest mass possible, making sure that the amount of carbon dioxide released is within the apparatus measurement range and the calibration range.

7.3.2 TC dosage

The sample, prepared according to 7.1, is weighed in an appropriate container, i.e. inert and not liable to interact during the carbon content analysis reaction or to contain carbon in any form whatsoever (scoop or crucible made of e.g. ceramic, silica glass, platinum or tin). The container can be previously conditioned by heating (in a muffle furnace or in the analyser itself) to minimize blank carbon values.

The sample is burned or broken down in a carrier gas current that contains oxygen.

The combustion temperature shall be sufficiently high to transform all the carbon into carbon dioxide. For samples containing carbonates that are difficult to break down, such as barium carbonate, carbon dioxide release can be improved by increasing the temperature or using modifiers (e.g. tin, copper).

The temperature range of commercially available devices is between 900 °C and 1 500 °C.

During combustion of the reactive samples, any detonation or production of smoke can be avoided by covering the sample with an inert material (e.g. siliceous sand).

The carbon dioxide released while gas is being discharged is measured using the detection method described in chapter 4, and expressed as carbon.

7.3.3 TIC dosage

The sample prepared according to 7.1 is weighed in the purging device.

The system is closed so as to be impermeable to gases and purged using the carrier gas until the carbon dioxide from the ambient air is eliminated. Then, acid is added and the carbon dioxide is carried out through purging or stirring and/or heating. The carbon dioxide released is transferred to the detector by means of the carrier gas.

The carbon dioxide released while gas is being discharged is measured using the detection method described in Clause 4, and expressed as carbon.

7.4 Calibration

If detection is carried out using a relative method, e.g. infrared detection, calibration is necessary.

Glucose is an example of a standard substance that is appropriate for TC dosage.

Sodium carbonate or calcium carbonate can be used for TIC calibration.

Other standard substances can be used, provided their suitability has been verified.

During calibration, the procedure below should be followed:

— set the preliminary measurement range;

— analyse a series of four calibration samples minimum at least twice, at three different times. The concentration of these master samples shall be regularly distributed over the entire measurement range;

— calculate the mean values for each concentration;

— perform a linear regression analysis using the mean values.

This function should be linear. If this is not the case, the measurement range needs to be reduced to the linear range.

This calibration should not be implemented for initial validation purposes or when major modifications of the apparatus are carried out.

7.5 Control measurements

Control measurements shall be taken to make sure the apparatus is functioning properly. They should be taken every working day. It is deemed sufficient to perform the dosage three times from a point located in the middle of the respective measurement ranges. For TC and TIC, the mean recovery rate shall be between 90 % and 110 % with a variation coefficient ≤ 5 %.

7.6 Evaluation

The TC and TIC masses contained in the samples prepared according to 7.1 are calculated using:

— The calibration function and the sample mass where relative detection methods are used;

— Specific constants and the sample mass where absolute detection methods are used.

TC and TIC contents are the means of at least two measurements each. The respective differences between the two values should be less than or equal to 10 % of the mean value. If this is not the case, at least one other additional dosage needs to be carried out; in such a case, the variation coefficient should be less than or equal to 10 %. If this is not the case, the coefficient shall be recorded along with the result obtained.

The TOC content is calculated using the difference between the mean TC and TIC values with the following formula:

$$\omega_{TOC} = \omega_{TC} - \omega_{TIC} \quad (1)$$

where

ω_{TOC} is the TOC content in carbon dioxide form in the original sample, in grams per kilogram (g/kg);

ω_{TC} is the mean value of the TC content in carbon dioxide form in the sample, in grams per kilogram (g/kg);

ω_{TIC} is the mean value of the TIC content in carbon dioxide form in the sample, in grams per kilogram (g/kg).

The TOC content yielded by Formula (1) is correlated to the dry matter using Formula (2). To do so, the water content, determined separately, is used:

$$\omega_{TOCdm} = \omega_{TOC} \frac{100}{100 - w} \quad (2)$$

where

ω_{TOCdm} is the TOC content in carbon dioxide form, correlated to the dry matter base, in grams per kilogram (g/kg);

ω_{TOC} is the TOC content in carbon dioxide form in the original sample, in grams per kilogram (g/kg);

w is the water content of the original sample, expressed as a mass fraction in percentage (%).