SLOVENSKI STANDARD

SIST EN 16640:2017

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Bioizdelki - Delež bioogljika - Ugotavljanje deleža bioogljika z radioogljično metodo

Bio-based products - Bio-based carbon content - Determination of the bio-based carbon content using the radiocarbon method

Biobasierte Produkte - Gehalt an biobasiertem Kohlenstoff - Bestimmung des Gehalts an biobasiertem Kohlenstoff mittels Radiokarbonmethode

Produits biosourcés - Teneur en carbone biosourcé - Détermination de la teneur en carbone biosourcé par la méthode au radiocarbone

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13.020.55	Biološki izdelki	Biobased products
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English Version

Bio-based products - Bio-based carbon content - Determination of the bio-based carbon content using the radiocarbon method

Produits biosourcés - Teneur en carbone biosourcé - Détermination de la teneur en carbone biosourcé par la méthode au radiocarbone

Biobasierte Produkte - Gehalt an biobasiertem Kohlenstoff - Bestimmung des Gehalts an biobasiertem Kohlenstoff mittels Radiokarbonmethode

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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-CENELEC Management Centre has the same status as the official versions.

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European foreword

This document (EN 16640:2017) has been prepared by Technical Committee CEN/TC 411 "Bio-based products", 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of an identical text or by endorsement, at the latest by August 2017, and conflicting national standards shall be withdrawn at the latest by August 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 16640:2014.

This document has been prepared under Mandate M/492 "Mandate addressed to CEN, CENELEC and ETSI for the development of horizontal European Standards and other standardization deliverables for bio-based products".

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Bio-based products from forestry and agriculture have a long history of application, such as paper, board and various chemicals and materials. The last decades have seen the emergence of new bio-based products in the market. Some of the reasons for the increased interest lie in the bio-based products' benefits in relation to the depletion of fossil resources and climate change. Bio-based products may also provide additional product functionalities. This has triggered a wave of innovation with the development of knowledge and technologies allowing new transformation processes and product development.

Acknowledging the need for common standards for bio-based products, the European Commission issued Mandate M/492[1)], resulting in a series of standards developed by CEN/TC 411, with a focus on bio-based products other than food, feed and biomass for energy applications.

The standards of CEN/TC 411 "Bio-based products" provide a common basis on the following aspects:

— Common terminology;

— Bio-based content determination;

— Life Cycle Assessment (LCA);

— Sustainability aspects;

— Declaration tools.

It is important to understand what the term bio-based product covers and how it is being used. The term 'bio-based' means 'derived from biomass'. Bio-based products (bottles, insulation materials, wood and wood products, paper solvents, chemical intermediates, composite materials, etc.) are products which are wholly or partly derived from biomass. It is essential to characterize the amount of biomass contained in the product by, for instance, its bio-based content or bio-based carbon content.

The bio-based content of a product does not provide information on its environmental impact or sustainability, which may be assessed through LCA and sustainability criteria. In addition, transparent and unambiguous communication within bio-based value chains is facilitated by a harmonized framework for certification and declaration.

This European Standard has been developed with the aim to specify the method for the determination of bio-based carbon content in bio-based products using the ^{14}C method. This method using the ^{14}C method is based on the analytical test methods used for the determination of the age of objects containing carbon.

This European Standard provides the reference test methods for laboratories, producers, suppliers and purchasers of bio-based product materials and products. It may be also useful for authorities and inspection organizations.

Part of the research leading to this document has been performed under the European Union Seventh Framework Programme (see http://www.biobasedeconomy.eu/research/kbbpps/).This document is based on EN 15440 [1] prepared by CEN/TC 343, "Solid recovered fuels", EN ISO 13833 [2], prepared by ISO/TC 146 "Air quality" and CEN/TC 264 "Air quality", and CEN/TS 16137 [3], prepared by CEN/TC 249, "Plastics".

The analytical test methods specified in this European Standard are compatible with those described in ASTM D 6866-12 [4].

1) A mandate is a standardization task embedded in European trade laws. Mandate M/492 is addressed to the European Standardization bodies, CEN, CENELEC and ETSI, for the development of horizontal European Standards for bio-based products.

1 Scope

This European Standard specifies a method for the determination of the bio-based carbon content in products, based on the ^{14}C content measurement.

This European Standard also specifies two test methods to be used for the determination of the ^{14}C content from which the bio-based carbon content is calculated:

— Method A: Liquid scintillation-counter method (LSC) ;

— Method B: Accelerator mass spectrometry (AMS).

A third method, Method C: Beta ionization (BI) can also be used for the determination of the ^{14}C content and is described in Annex D (informative)

The bio-based carbon content is expressed by a fraction of sample mass or as a fraction of the total carbon content. This calculation method is applicable to any product containing carbon, including bio composites.

NOTE This European Standard does not provide the methodology for the calculation of the biomass content of a sample see prEN 16785–1 [5] and prEN 16785–2 [6].

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 15400, *Solid recovered fuels - Determination of calorific value*

EN ISO 1716, *Reaction to fire tests for products - Determination of the gross heat of combustion (calorific value) (ISO 1716)*

ISO 1928, *Solid mineral fuels — Determination of gross calorific value by the bomb calorimetric method and calculation of net calorific value*

3 Terms and definitions

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

3.1
organic material
material containing carbon-based compound in which the element carbon is attached to other carbon atoms, hydrogen, oxygen, or other elements in a chain, ring, or three-dimensional structure

3.2
isotope abundance
fraction of atoms of a particular isotope of an element

3.3
percentage modern carbon
pMC
normalized and standardized value for the amount of the ^{14}C isotope in a sample, calculated relative to the standardized and normalized ^{14}C isotope amount of oxalic acid standard reference material, NIST SRM 4990b or NIST SRM 4990c or Sucrose (NIST SRM 8542)

Note 1 to entry: In 2016, the value of 100 % bio-based carbon was set at 101,5 ± 0,5 pMC.

Note 2 to entry: SRM 4990c, SRM 4990b and SRM 8582 are the trade name of products supplied by the US National Institute of Standards and Technology. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

3.4
laboratory sample
sub-quantity of a sample suitable for laboratory tests

3.5
sample
quantity of material, representative of a larger quantity for which the property is to be determined

3.6
sample preparation
all the actions taken to obtain representative analysis samples or test portions from the original sample

3.7
beta- particle
electron emitted during radioactive decay

4 Symbols and abbreviations

^{14}C	carbon isotope with an atomic mass of 14
AMS	accelerator mass spectrometry
BI	beta-ionization
Bq	Bequerel (disintegrations per second)
C	symbol for element carbon
Cpm	counts per minute
dpm	disintegrations per minute
GM	Geiger-Müller
LLD	lower limit of detection
LSC	Liquid Scintillation Counter or Liquid Scintillation Counting
m	dry mass of a sample expressed in grams
MOP	3-methoxy 1-propyl amine
PE	Polyethylene
PLA	poly(lactic acid)
pMC	percentage of modern carbon
REF	reference value, expressed in pMC, of 100 % bio-based carbon depending on the origin of organic carbon
TC	total carbon
x_B	bio-based carbon content by mass, expressed as a percentage of the mass of the sample (dry)
x^{TC}	total carbon content, expressed as a percentage of the mass of the sample (dry)
x_B^{TC}	bio-based carbon content by total carbon content, expressed as a percentage of the total carbon content

5 Principle

The ^{14}C present in products is originating from recent atmospheric CO_2. Due to its radioactive decay, it is almost absent from fossil products older than 20 000 years to 30 000 years. The ^{14}C content may thus be considered as a tracer of products recently synthesized from atmospheric CO_2 and particularly of recently produced bio-products.

The determination of the biomass content is based on the measurement of ^{14}C in bio-based products, which allows the calculation of the bio-based carbon fraction.

A large experience in ^{14}C determination and reference samples is available from dating of archaeological objects, on which the three methods described in this European Standard are based:

— Method A: Proportional scintillation-counter method (LSC),

— Method B: Accelerator mass spectrometry (AMS); or

— Method C: Beta-ionization (BI).

NOTE 1 The advantages and disadvantages of these test methods are given in Table 1.

Table 1 — Advantages and disadvantages of the methods

Method	**Additional requests**	**Duration needed for measurement**	**Relative standard deviation**	**Instrumental costs**
Method A (LSC)	Normal laboratory	4 h to 12 h	0,2 % to 5 %	Low
Method B (AMS)	- Large installation - Graphite Conversion device	10 min to 30 min	0,2 % to 2 %	High
Method C (BI)	- Low background laboratory - Gas purification device	8 h to 24 h	0,2 % to 5 %	Low

For the ^{14}C LSC measurement a Low Level Counter shall be used. The statistical scattering of the radioactive decay sets a limit, both for method A and B. Thereby both methods need a purified carbon dioxide, otherwise oxides of nitrogen from the combustion in the calorific bomb will result in counting losses by quenching and adulteration of the cocktail in case of LSC measurement. When using method A (LSC), samples with low bio-based carbon content (<10 %) can only be measured with sufficient precision using the benzene conversion procedure or, if applicable, direct LSC measurement, as described in Annex B.

NOTE 2 At this moment compact new AMS equipment has become available. In a number of cases, no graphite conversion is required anymore.

6 Determination of the ^{14}C content

6.1 General

A general sample preparation and three test methods for the determination of the ^{14}C content are described in this European Standard. With this modular approach, it will be possible for normally equipped laboratories to prepare samples for the ^{14}C content, and determine the ^{14}C content with own equipment or to outsource the determination of the ^{14}C content to laboratories that are specialized in this technique.

For the collection from the sample of the ^{14}C content, generally accepted methods for the conversion of the carbon present in the sample to CO_2 are described.

For the measurement of the ^{14}C content, methods are selected that are already generally accepted as methods for the determination of the age of objects.

6.2 Principle

The amount of bio-based carbon in the bio-based product is proportional to this ^{14}C content.

Complete combustion (see Annex B) is carried out in a way to comply with the requirements of the subsequent measurement of the ^{14}C content and shall provide the quantitative recovery of all carbon present in the sample as CO_2 in order to yield valid results.

This measurement shall be carried out according to one of the two following methods:

— Method A: Liquid scintillation-counter method (LSC): indirect determination of the isotope abundance of ^{14}C, through its emission of beta-particle (interaction with scintillation molecules), specified in Annex C, or

— Method B: Accelerator mass spectrometry (AMS): direct determination of the isotope abundance of ^{14}C, specified in Annex E.

Possibly, this measurement can also be carried out according to Method C: Beta-ionization (BI): indirect determination of the isotope abundance of ^{14}C, through its emission of beta-particle (Geiger-Müller type detector), described in Annex D.

6.3 Sampling

In Annex A sampling methods for products that are mentioned in the scope are given.

For any sampling procedure, the samples shall be representative of the material or product and the quantity or mass of sample shall be accurately established.

6.4 Procedure for the conversion of the carbon present in the sample to a suitable sample for ^{14}C determination

The conversion of the carbon present in the sample to a suitable sample for the determination of the ^{14}C content shall be carried out according to Annex B.

6.5 Measurements

The measurement of the ^{14}C content of the sample shall be performed according to one of the methods as described in Annexes C, D or E.

When collected samples are sent to specialized laboratories, the samples shall be stored in a way that no CO_2 from air can enter the absorption solution. A check on the in leak of CO_2 from air shall be performed by preparing laboratory blanks during the sampling stage.

For the determination of the 0 % biomass content the combustion of a coal standard may be used.

For validation of the 100 % biomass content, oxalic acid standard reference material, NIST SRM 4990b or NIST SRM 4990c or Sucrose (NIST SRM 8542) may be used. Mixing reference material NIST 4990 with a known amount of fossil combustion aid improves its combustion behaviour, as oxalic acid is difficult to combust due to its low calorific value. For routine checks, a fresh wood sample calibrated against the standard reference material is sufficient.

7 Calculation of the bio-based carbon content

7.1 General

The calculation of the bio-based carbon content includes the following steps:

a) the determination of the total carbon content of the sample, x^{TC}, expressed as a percentage of the total dry mass;

b) the calculation of the bio-based carbon content by mass, x_B, using the ^{14}C content value, determined by calculation from one of the test methods specified in 7.3, and applying the correction factors detailed in 7.2, and

c) the calculation of the bio-based carbon content as a fraction of the total carbon content, x_B^{TC} (see 7.3.2).

7.2 Reference value for 100 % bio-based carbon

Before the above-ground hydrogen bomb testing (started around 1955 and terminated in 1962) the atmospheric ^{14}C level had been constant to within a few percent, for the past millennium. Hence, a sample grown during this time has a well-defined “modern” activity, and the fossil contribution could be determined in a straightforward way. However, ^{14}C created during the weapons testing increased the atmospheric ^{14}C level to up to 200 pMC in 1962, with a decline to 102 pMC in 2015. The ^{14}C activity of a sample grown since year 1962 is elevated according to the average ^{14}C level over the growing interval. In addition, the large emission of fossil C during the last decades contributes to the decrease of the atmospheric $^{14}C/^{12}C$ ratio.

In ASTM D 6866-16 [4] the 100 % bio-based C value of 101.5 pMC (for year 2016) is used. This value shall be the base of calculations. Other values are only acceptable if evidence can be given on the pMC value of the biogenic part of the material. From the 102 pMC value the correction factor of 0,98 (1/1,02) is derived. It is considered that such correction factor is now stable during a period of a few years.

For the calculation of the bio-based carbon content, a ^{14}C content of 100/0,98 pMC or 13,56/0,98 dpm per gram C is considered as a 100 % bio-based carbon content for biomass that is grown in year 2015.

NOTE 1 This correction factor of 0,98 is in accordance with the value that is given in ASTM D 6866–16 [4].

The fraction of biomass content by dry mass shall be calculated using the biomass carbon in the bio-based product as for other organic carbon materials. Table 2 lists typical values for such common materials.

NOTE 2 The reference value is based on the measurements of CO_2 in air in a rural area in the Netherlands (Lutjewad, Groningen), performed by CIO (Centre for Isotope Research, university Groningen). New reference values for 2016 and later will be available on the CIO internet site (http://www.rug.nl/research/isotope-research/klanten/14cbio-values-standards).