SLOVENSKI STANDARD
SIST-TS CEN/TS 16640:2014

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Bioizdelki - Ugotavljanje deleža biogljika v izdelkih z radiogljično metodo

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

Biobasierte Produkte - Bestimmung des biobasierten Kohlenstoffanteils von Produkten mittels Radiocarbonmethode

Produits biosourcés - Détermination de la teneur en carbone biosourcé des produits par la méthode du carbone radioactif

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English Version

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This Technical Specification (CEN/TS) was approved by CEN on 9 December 2013 for provisional application.

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Foreword

This document (CEN/TS 16640:2014) has been prepared by Technical Committee CEN/TC 411 “Bio-based products”, the secretariat of which is held by NEN.

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

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 organizations of the following countries are bound to announce this Technical Specification: 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, 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

Iit 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 Technical Specification 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 Technical Specification 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.kbbpps.eu).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 Technical Specification 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 Technical Specification specifies a method for the determination of the bio-based carbon content in products, based on the ^{14}C content measurement.

It also specifies three 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: Beta-ionization (BI);

— Method C: Accelerator mass spectrometry (AMS).

The bio-based carbon content is expressed by a fraction of sample mass, as a fraction of the total carbon content or as a fraction of the total organic carbon content.

This calculation method is applicable to any product containing organic carbon, including biocomposites.

NOTE This Technical Specification does not provide the methodology for the calculation of the biomass content of a sample.

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
bio-based carbon content
fraction of carbon derived from biomass in a product

Note 1 to entry: There are several approaches to express the bio-based carbon content. These include as a percentage of: the mass; the total carbon content, or the total organic carbon content, of the sample. These are detailed in the relevant standards of CEN/TC 411.

[SOURCE: prEN 16575:2013 [5]]

3.2
biomass content
see bio-based content

[SOURCE: prEN 16575:2013 [5]]

3.3
bio-based content
fraction of a product that is derived from biomass

Note 1 to entry: Normally expressed as a percentage of the total mass of the product.

Note 2 to entry: For the methodology to determine the bio-based content, see TC 411 WI 00411002 bio-based content.

[SOURCE: prEN 16575:2013 [5]]

3.4
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

3.5
organic carbon
carbon from organic material

3.6
isotope abundance
fraction of atoms of a particular isotope of an element

3.7
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, SRM 4990c[2)]

Note 1 to entry: In 2009, the value of 100 % bio-based carbon was set at 105 pMC.

3.8
laboratory sample
sub-quantity of a sample suitable for laboratory tests

3.9
sample
quantity of material, representative of a larger quantity for which the property is to be determined

3.10
sample preparation
all the actions taken to obtain representative analysis samples or test portions from the original sample

3.11
beta- particle
electron emitted during radioactive decay

3.12
total carbon
TC
quantity of carbon present in a product in the form of organic, inorganic and elemental carbon

[SOURCE: prEN 16575:2013 [5]]

2) SRM 4990c is the trade name of a product 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.

3.13
total organic carbon
TOC
quantity of organic carbon present in a product

Note 1 to entry: Total organic carbon is often determined as the carbon that is converted into carbon dioxide by combustion and which is not liberated as carbon dioxide by acid treatment.

[SOURCE: prEN 16575:2013 [5]]

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	mass of a sample expressed in grams
MOP	3-methoxy 1-propyl amine
PE	polyethylene
PLA	poly(lactic acid)
pMC	percentage of modern carbon
pMC(s)	measured value, expressed in pMC, according to AMS method, of the sample
REF	reference value, expressed in pMC, of 100 % bio-based carbon depending on the origin of organic carbon
TC	total carbon
TOC	total organic carbon
x_B	bio-based carbon content by mass, expressed as a percentage of the mass of the sample
x^{TC}	total carbon content, expressed as a percentage of the mass of the sample

x_B^{TC} bio-based carbon content by total carbon content, expressed as a percentage of the total carbon content

x^{TOC} total organic carbon content, expressed as a percentage of the mass of the sample

x_B^{TOC} bio-based carbon content by total organic carbon content, expressed as a percentage of the total organic 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 technical specification are based:

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

— Method B: Beta-ionization (BI), or

— Method C: Accelerator mass spectrometry (AMS).

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

Table 1 — Advantages and disadvantages of the methods

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

For the ^{14}C LSC measurement a Low Level Counter should 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.

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 Technical Specification. 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 three 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;

— Method B: Beta-ionization (BI): indirect determination of the isotope abundance of ^{14}C, through its emission of beta-particle (Geiger-Müller type detector), specified in Annex D, or

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

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 blank's during the sampling stage.