SLOVENSKI STANDARD
SIST-TS CEN/TS 16137:2011

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Polimerni materiali - Določevanje ogljika biološkega izvora

Plastics - Determination of bio-based carbon content

Kunststoffe - Bestimmung des biobasierten Kohlenstoffgehalts

Plastiques - Détermination de la teneur en carbone biosourcé

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83.080.01	Polimerni materiali na splošno	Plastics in general

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English Version

Plastics - Determination of bio-based carbon content

Plastiques - Détermination de la teneur en carbone biosourcé

Kunststoffe - Bestimmung des biobasierten Kohlenstoffgehalts

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Foreword

This document (CEN/TS 16137:2011) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

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Introduction

This Technical Specification specifies the calculation method for the determination of bio-based carbon content in monomers, polymers, plastics materials and products using the ^{14}C method.

This calculation method using the ^{14}C method is based on the well established 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 polymer materials and products. It can be also useful for authorities and inspection organizations.

NOTE 1 This Technical Specification is based on EN 15440 [4] prepared by CEN/TC 343, Solid recovered fuels.

NOTE 2 The analytical test methods specified in this Technical Specification are compatible with those described in ASTM D6866 - 10 [9].

1 Scope

This Technical Specification specifies a calculation method for the determination of the bio-based carbon content in monomers, polymers and plastic materials and 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: Proportional scintillation-counter method (PSM);

— Method B: Beta-ionisation (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 polymers 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TR 15932:2010, *Plastics — Recommendation for terminology and characterisation of biopolymers and bioplastics*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 15932:2010 and the following apply.

3.1
bio-based carbon content
amount of carbon in a sample that is of recent origin, as evidenced by its ^{14}C isotope content

3.2
biomass content
mass fraction of bio-based material in a sample

3.3
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

3.4
organic carbon
carbon from organic material

3.5
isotope abundance
fraction of atoms of a particular isotope of an element

3.6
percentage modern carbon
pmC
percent modern carbon relative to the N.I.S.T. [1)] oxalic acid radiocarbon standard reference material SRM 4990B

NOTE In 1950, the internationally accepted radiocarbon dating reference value is 95 % of this activity of this NBS oxalic acid SRM 4990b. In 2010, the value of 100 % bio-based carbon is set at 105 pmC.

3.7
laboratory sample
sub-quantity of a sample suitable for laboratory tests

3.8
sample
quantity of material, representative of a larger quantity for which the property is to be determined

3.9
sample preparation
actions taken to obtain representative analyses samples or test portions from the original sample

3.10
β particle
electron emitted during radioactive decay

3.11
total carbon
TC
quantity of carbon present in a sample in the form of organic, inorganic and elemental carbon

[EN 13137:2001]

3.12
total organic carbon
TOC
quantity carbon that is converted into carbon dioxide by combustion and which is not liberated as carbon dioxide by acid treatment

[EN 13137:2001]

4 Symbols and abbreviations

4.1 Symbols

C symbol for element carbon

^{14}C carbon isotope with an atomic mass of 14

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

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

x_B bio-based carbon content by mass, expressed as a percentage of the mass of the sample

1) National Institute of Standards and Technology - Gaithersburg, Maryland, USA.

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

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

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

m mass of a sample expressed in grams

4.2 Abbreviations

AMS accelerator mass spectroscopy

BI beta-ionisation

Bq Bequerel (desintegrations per second)

cpm counts per minute

CV coefficient of variation

dpm disintegrations per minute

GM Geiger-Müller

LLD lower limit of detection

LSC liquid scintillation-counter or liquid scintillation-counting

MOP 3-methoxy 1-propyl amine

PE polyethylene

PLA poly(lactic acid)

pmC percentage of modern carbon

PSM proportional scintillation-counter method

TC total carbon

TOC total organic carbon

5 Principle

The ^{14}C present in chemicals 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 chemicals 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 polymers which allows the calculation of the bio-based carbon fraction.

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

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

— Method B: Beta-ionisation (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 (PSM)	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

6 Sampling

If there is a standard sampling procedure for the material or product to be evaluated that is widely accepted by the different parties, such a procedure may be used and the details of sampling recorded.

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.

7 Determination of the ^{14}C content

7.1 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.

7.2 Principle

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

Complete combustion (see Annex A) 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