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Plastics — Biobased content —

Part 2: Determination of biobased carbon content

Plastiques — Teneur biosourcée —

Partie 2: Détermination de la teneur en carbone biosourcé

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 16620 consists of the following parts, under the general title *Plastics — Biobased content*:

- *Part 1: General principles*
- *Part 2: Determination of biobased carbon content*
- *Part 3: Determination of biobased synthetic polymer content*

The following parts are under preparation:

- *Part 4: Determination of the biobased mass content*
- *Part 5: Declaration of biobased carbon content, biobased synthetic polymer content and biobased mass content*

Introduction

Increased use of biomass resources for manufacturing plastic products is effective in reducing global warming and the depletion of fossil resources.

Current plastic products are composed of biobased synthetic polymers, fossil-based synthetic polymers, natural polymers, and additives that can include biobased materials.

Biobased plastics refer to plastics that contain materials, wholly or partly of biogenic origin.

In this series of International Standards, the biobased content of biobased plastics refers to the amount of the biobased carbon content, the amount of the biobased synthetic polymer content, or the amount of the biobased mass content only.

Plastics — Biobased content —

Part 2: Determination of biobased carbon content

WARNING — The use of this part of ISO 16620 might involve hazardous materials, operations, and equipment. This part of ISO 16620 does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 16620 specifies a calculation method for the determination of the biobased carbon content in monomers, polymers, and plastic materials and products, based on the ^{14}C content measurement.

This part of ISO 16620 is applicable to plastic products and plastic materials, polymer resins, monomers, or additives, which are made from biobased or fossil-based constituents.

Knowing the biobased content of plastic products is useful when evaluating their environmental impact.

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.

ISO 16620-1, *Plastics — Biobased content — Part 1: General principles*

3 Terms, definitions, symbols, and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16620-1 and the following apply.

3.1.1
percent 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[1)]

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

[SOURCE: ISO 13833:2013, 3.5]

1) 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 International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products can be used if they can be shown to lead to the same results.

3.1.2
radiocarbon
radioactive isotope of the element carbon, ^{14}C, having 8 neutrons, 6 protons, and 6 electrons

Note 1 to entry: Of the total carbon on Earth, 1×10^{-10} % (mass fraction) is ^{14}C. It decays exponentially with a half-life of 5 730 years and, as such, it is not measurable in fossil materials derived from petroleum, coal, natural gas, or any other source older than about 50 000 years.

[SOURCE: ISO 13833:2013, 3.7]

3.2 Symbols

For the purposes of this document, the symbols given in ISO 16620-1 and the following apply.

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

m mass of a sample expressed in grams

pMC(s) measured value, expressed in pMC, according to AMS method, of the sample

REF reference value, expressed in pMC, of 100 % biobased carbon depending on the origin of organic carbon

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 biobased carbon content by mass, expressed as a percentage of the mass of the sample

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

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

3.3 Abbreviated terms

AMS accelerator mass spectroscopy

BI beta-ionization

Bq Bequerel (disintegrations per second)

cpm counts per minute

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

pMC percentage of modern carbon

TC total carbon

TOC total organic carbon

4 Principle

The ^{14}C present in chemicals originates 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. Thus, the ^{14}C content might 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 polymers which allows the calculation of the biobased 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 part of ISO 16620 are based:

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

— Method B: Beta-ionization (BI);

— 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.

5 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 can 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.

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 International 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 biobased carbon in the biobased 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 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:

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

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

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

6.3 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 the Annex A.

6.4 Measurement techniques

The ^{14}C content of the sample shall be determined using one of the methods as described in Annex B, Annex C, or Annex D.

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.

For the determination of the 0 % biomass content, the combustion of a coal standard (e.g. BCR 181) can be used.

For the 100 % biomass content, the N.I.S.T. oxalic acid standard reference material (SRM 4990c) can be used. Mixing this reference material 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 wood standard reference material calibrated against the oxalic acid is sufficient.

7 Determination of the total carbon content and total organic carbon content

The total carbon content and organic carbon content shall be determined according to suitable methods.

Test methods as described in ISO 10694, ISO 8245, EN 13137, ISO 17247, ISO 15350, ISO 609, ASTM D5291-02, or ASTM E1019 can be used, as applicable.

8 Calculation of the biobased carbon content

8.1 General

The calculation of the biobased carbon content includes the following steps:

a) the determination of the total carbon content of the sample, x^{TC}, determined by one of the test methods specified in Clause 7, expressed as a percentage of the total mass or the determination of the total organic carbon content of the sample, x^{TOC}, determined by one of the test methods specified in Clause 7, expressed as a percentage of the total mass;

b) the calculation of the biobased carbon content by mass, x_B, using the ^{14}C content value, determined by calculation from one of the test methods specified in Clause 6, and applying the correction factors detailed in 8.2;

c) the calculation of the biobased carbon content as a fraction of the total carbon content, x_B^{TC} (see 8.3.2) or a fraction of the total organic carbon content, x_B^{TOC} (see 8.3.3).

8.2 Correction factors

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 105 pMC in 2010. 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 D6866–12, the 100 % biobased C value of 105 pMC (for year 2010) is used. This value shall be the base of calculations. Other values are only acceptable if they are based on experimental evidence. From the 105 pMC value, the correction factor of 0,95 (1/1,05) is derived. It is considered that such correction factor is now stable during a period of a few years.

For the calculation of the biobased carbon content, a ^{14}C content of 100/0,95 pMC or 13,56/0,95 dpm per gram C is considered as a 100 % biobased carbon content for biomass that is grown in year 2010.

NOTE This correction value of 0,95 is in accordance with the value that is given in ASTM D6866–12.

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

Table 2 — Typical values for biomass fractions

Material[a]	x^{TC} %	***REF*** pMC
Wood (coniferous and deciduous)	48	114
Bark	52	111
Paper	47	114
Fresh biomass (from year 2010)	48	105
Silk	49	107
Wool	51	107

[a] These values are given on "dry basis".

8.3 Calculation method

8.3.1 Calculation of the biobased carbon content by mass, x_B

8.3.1.1 ^{14}C content determined by Method A (LSC) or Method B (BI)

Calculate the biobased carbon content by mass, x_B, expressed as a percentage, using Formula (1):

$$x_B = \frac{^{14}C_{activity}}{13{,}56 \times \frac{REF}{100} \times m} \times 100 \quad (1)$$

where

$^{14}C_{activity}$ is the ^{14}C activity, expressed in dpm, of the sample obtained by calculation when using Method A or Method B (see Annex B or Annex C);

REF is the reference value, expressed in pMC, of 100 % biobased carbon of the biomass from which the sample is constituted;

m is the mass, expressed in grams, of the sample.

8.3.1.2 ^{14}C content determined by Method C (AMS)

Calculate the biobased carbon content by mass, x_B, expressed as a percentage, using Formula (2):

$$x_B = x^{TC} \frac{\frac{pMC(s)}{100}}{\frac{REF}{100}} = x^{TC} \frac{pMC(s)}{REF} \quad (2)$$

where

x^{TC} is the total carbon content obtained by elemental analysis, expressed as a percentage, of the total mass, of the sample;

pMC(s) is the measured value, expressed in pMC, of the sample;

REF is the reference value, expressed in pMC, of 100 % biobased carbon of the biomass from which the sample is constituted.

8.3.2 Calculation of the biobased carbon content, x_B^{TC}, as a fraction of TC

Calculate the biobased carbon content as a fraction of the total carbon content, x_B^{TC}, expressed as a percentage, using Formula (3):

$$x_B^{TC} = \frac{x_B}{x^{TC}} \times 100 \quad (3)$$

where

x_B is the biobased carbon content by mass, expressed as a percentage;

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

