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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

Reference number
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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects*.

This second edition cancels and replaces the first edition (ISO 16620-2:2015), which has been technically revised.

The main changes compared to the previous edition are as follows:

— REF values for calculation of biobased carbon content from percent modern carbon vs. years are listed in Table 2.

A list of all parts in the ISO 16620 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 the ISO 16620 series, 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 document might involve hazardous materials, operations, and equipment. This document 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 document to establish appropriate safety and health practices and determine any restrictions prior to use.

1 Scope

This document 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 document is applicable to plastic products and plastic materials (e.g. plasticisers or modifiers), 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 are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at http://www.electropedia.org/

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, NIST SRM 4990b or NIST SRM 4990c or Sucrose (NIST SRM 8542)[1)]

Note 1 to entry: The reference value of 100 % biobased carbon is given in Table 2.

1) NIST SRM 4990b or NIST SRM 4990c or Sucrose (NIST SRM 8542) 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 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} % 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

^{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

NOTE 1 “Biobased carbon content by mass, x_B” used in this document corresponds to “biobased carbon content on mass” defined in 3.3.9 of ASTM D6866-18.

NOTE 2 “Biobased carbon content by total carbon content, x_B^{TC}” corresponds to “biogenic carbon content” defined in 3.3.8 of ASTM D6866-18.

NOTE 3 “Biobased carbon content by total organic carbon content, x_B^{TOC}” corresponds to “biobased carbon content” defined in 3.3.7 of ASTM D6866-18.

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 document are based:

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

— Method B: Beta-ionization (BI);

— Method C: Accelerator mass spectrometry (AMS).

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	2 % to 5 %	Low
Method B (BI)	— Low background laboratory — Gas purification device	8 h to 24 h	0,2 % to 5 %	Low
Method C (AMS)	— 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. 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 A.

NOTE 2 At this moment compact new AMS equipment has become available. In a number of cases, no graphite conversion is required anymore. CO_2 gas can be measured directly by these AMS.

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 document. 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 two 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;

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

This measurement can also be carried out according to Method B [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 C.

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 validation of the 100 % biomass content, the oxalic acid standard reference material NIST SRM 4990b or 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 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 total organic carbon content shall be determined according to suitable methods.

Test methods as described in ISO 609, ISO 8245, ISO 10694, ISO 15350, ISO 17247, ASTM D5291-16, ASTM E1019 or EN 13137, 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 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 6866-18 the 100 % bio-based C value of 100,5 pMC (for year 2018) 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.

The 100 % bio-based C value equates to decline of 0,5 pMC per year. Therefore, on January 1st of each year, the values given in Table 2 are used through 2019, reflecting the 0,5 pMC decrease per year.

Table 2 — 100 % biobased carbon values versus year

Year	100 % biobased carbon value REF (pMC, %)
2015	102,0
2016	101,5
2017	101,0
2018	100,5