



FINAL DRAFT International Standard

ISO/FDIS 19984-2

Rubber and rubber products — Determination of biobased content —

Part 2: Biobased carbon content

*Élastomères et produits à base d'élastomères — Détermination
de la teneur en composés biosourcés —*

Partie 2: Teneur en carbone biosourcé

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 document 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 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

A list of all parts in the ISO 19984 series can be found on the ISO website.

This second edition cancels and replaces the first edition (ISO 19984-2:2017), which has been technically revised.

The main changes are as follows:

- REF values after 2020 have been added in [Table 2](#);
- several bibliographic entries have been added.

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

The use of biomass materials in rubber compounds helps to decrease the rubber industry's dependence on fossil resources. It is also expected to lead to a reduction of carbon dioxide emission, reducing global warming and promoting a sustainable global environment.

In the ISO 19984 series, biomass is the term used for the biological material from living or recently living organisms such as wood and agricultural waste materials.

Industrial scale biomass is now readily being grown from numerous types of plant sources and a variety of tree species. Biomass also includes plant or animal matter used for the production of fibres or chemicals. It can also include biodegradable wastes. Biomass excludes organic materials which have been transformed by geological processes into substances, such as petroleum or coal. Although fossil fuels have their origin in ancient biomass, they are not considered biomass by the generally accepted definition because they contain carbon that has been "out" of the modern carbon cycle.

The composition of biomass is mainly carbon, hydrogen and oxygen. Nitrogen and small quantities of other elements can also be found.

The ISO 19984 series specifies methods for the determination of the biobased content of rubber and rubber products. The results will give manufacturers and users a quantitative indication of their contribution to the preservation of the environment.

ISO 19984-1 specifies how to categorize constituents of rubber and rubber products and how to calculate the biobased content using the compound formulation and the chemical structure of each constituent.

ISO 19984-2 specifies how to determine the biobased carbon content by radio chemical analyses, i.e. determination of ^{14}C . It can be obtained from the fraction of carbon atoms derived from biomass against the whole amount of carbon atoms in the rubber or rubber products. The methods specified in ISO 19984-2 allow consumers to determine the biobased carbon content even when the formulation of the rubber is unavailable.

ISO 19984-3 specifies how to separate rubber compounds into constituents, how to obtain each constituent's composition ratio and how to determine the biobased carbon content of each constituent by chemical analyses. Thus, the biobased mass content for each constituent can be derived and the biobased mass content for the whole rubber can be obtained by summing up all the constituent values.

Rubber and rubber products — Determination of biobased content —

Part 2: Biobased carbon content

WARNING 1 — This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document can involve the use or generation of substances, or the generation of waste, that can constitute a local environmental hazard. It is the responsibility of the user to refer to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies measuring methods for the determination of biobased carbon contents in rubber and rubber products, including polyurethanes. The methods focus on carbon atoms in rubber or rubber products, and determine whether the carbon-containing component is biobased or not, judging from the concentration of ^{14}C , radiocarbon isotope.

This document applies to rubber and rubber products such as raw materials, materials and final products.

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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 1382, *Rubber — Vocabulary*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 1928, *Coal and coke — Determination of gross calorific value*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 19242, *Rubber — Determination of total sulfur content by ion chromatography*

ISO 19984-1, *Rubber and rubber products — Determination of biobased content — Part 1: General principles and calculation methods using the formulation of the rubber compound*

3 Terms, definitions, and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382 and ISO 19984-1 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://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, SRM 4990c¹⁾

Note 1 to entry: The reference value of 100 % biobased carbon is given in [Table 2](#).

3.1.2

^{14}C activity

relative concentration of radiocarbon ^{14}C expressed as a counting of β -irradiation from the decayed radiocarbon atoms per minute

Note 1 to entry: The unit of ^{14}C activity is “dpm” (decay per minute).

Note 2 to entry: The ^{14}C activity is determined relatively using standard reference material (SRM 4990c) whose ^{14}C activity is set at 13,56 dpm.

3.2 Abbreviated terms

AMS	accelerator mass spectroscopy
dpm	disintegrations per minute
LSC	liquid scintillation-counter or liquid scintillation-counting
pMC	percent modern carbon
REF	reference value
SRM	standard reference materials

4 Principle

This document specifies those methods to determine the biobased carbon contents derived from biomass resources.

1) SRM 4990c is an example of a suitable 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 may be used if they can be shown to lead to the same results.

When the formulation of the rubber product is available, the biobased carbon content can be calculated (see ISO 19984-1). The biobased carbon content is defined as the amount of biobased carbon to the total carbon in rubber or rubber products as shown in [Formula \(1\)](#):

$$\chi_B^{TC} = \frac{C_B}{C_B + C_F + C_{NB}} \times 100 \quad (1)$$

where

χ_B^{TC} is the biobased carbon content (%);

C_B , C_F and C_{NB} are the mass of biobased, fossil-based and non-biobased carbon, respectively.

When there is no available information for the rubber or the rubber product, the biobased carbon content can be determined by the ^{14}C concentration. Due to its radioactive decay, ^{14}C hardly exists in fossil products older than 20 000 years to 30 000 years. Therefore, the ^{14}C present in products is estimated to have come from recent atmospheric carbon dioxide, and consequently, it can be considered as a tracer of recently produced bio-products.

The biobased carbon content values determined in accordance with this document can also be compared to the theoretical values calculated from the formulation, so that the reliability of the information about the rubber in the rubber product is confirmed.

In order to determine the biobased mass content for raw rubber, organic ingredient, rubber products or separated constituents, refer to ISO 19984-3.

5 Sampling

5.1 In the case of latex, carry out sampling in accordance with ISO 123 and dry the sample in accordance with ISO 124.

5.2 In the case of raw material, carry out sampling in accordance with ISO 15528.

5.3 In the case of raw rubber, carry out sampling in accordance with ISO 1795.

5.4 In the case of vulcanized rubber, carry out sampling in accordance with ISO 4661-2.

NOTE The procedure of washing the surface of samples by acid and alkaline solution, which is a familiar preparation process for carbon dating, is not necessary.

6 Measuring method of biobased carbon content by determination of ^{14}C content

6.1 General

Sample preparation and two methods for the determination of the ^{14}C content are described in this document. With these modular approaches, it is possible for normally equipped laboratories to prepare samples and choose either method to determine the ^{14}C content with their own equipment or to outsource the determination process to other laboratories that are specialized in this technique.

For the collection of the ^{14}C from the sample, commonly accepted methods for the conversion of the carbon present in the sample to carbon dioxide are adopted as indicated in [Figure 1](#) (see also [A.4.1](#) and [B.4.1](#)).

For the measurement of the ^{14}C content, two methods are adopted that have been generally accepted as age determination methods as indicated in [Figure 1](#) (see also [Annex A](#) and [Annex B](#)).

NOTE: See [Annex A](#) for information regarding the accelerator mass spectrometry (AMS) method and [Annex B](#) for information regarding the LSC method.

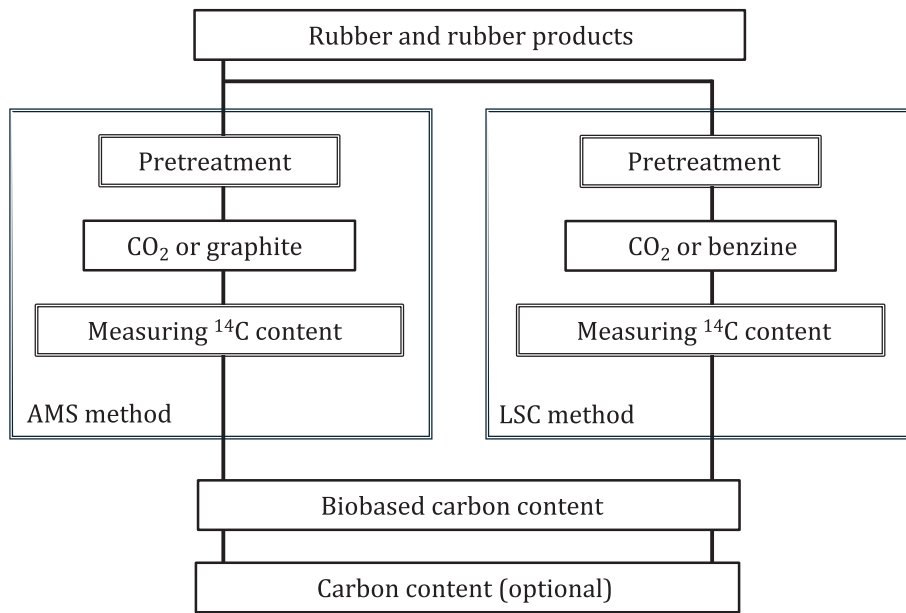


Figure 1 — Outline for the determination of biobased carbon content

6.2 Sample preparation and two methods to determine ^{14}C concentration

Carry out an oxidation of the sample as specified in A.4.1 and B.4.1. A complete oxidization of all the carbons present in the sample shall be performed in order to obtain exact results. The measurement shall be made according to one of the following two methods:

- Method A — Accelerator mass spectrometry (AMS): Direct determination of the isotope abundance of ^{14}C , specified in Annex A.
- Method B — Liquid scintillation counter (LSC): Indirect determination of the isotope abundance of ^{14}C through its emission of beta-particles (interaction with scintillation molecules), specified in Annex B.

The comparison between these test methods is given in Table 1.

Table 1 — Characteristics of the measurement methods of ^{14}C

Method	Determination	Sample amount	Measurement time	Relative standard deviation
Method A (AMS)	Relative ratios between isotope ^{12}C , ^{13}C and ^{14}C	1 mg to 10 mg	10 min to 30 min	0,2 % to 2,0 %
Method B (LSC)	β counts of ^{14}C decay	0,5 mg to 2,0 g	4 h to 24 h	0,2 % to 10 %

6.3 Calculation of the biobased carbon content

6.3.1 General

The biobased carbon content as a fraction to the total carbon content, $\chi_{\text{B}}^{\text{TC}}$, using the ^{14}C content value, is determined by calculation from one of the test methods specified in 6.3.3 or 6.3.4, and applying the correction factor in Table 2.

6.3.2 Correction factor

Before the above-ground hydrogen bomb testing (beginning approximately in 1955 and terminated in 1962), the atmospheric ^{14}C level was constant with a few percent range of change for the past millennium. Hence, a