# INTERNATIONAL STANDARD

# ISO 19984-2

First edition
2017-08

# 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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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

 

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on 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 the following URL: 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.

# 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 plants sources and a variety of tree species. Biomass nowadays also includes plant or animal matter used for the production of fibres or chemicals. It may 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 also 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 — Persons using this document should be familiar with normal laboratory practice. 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 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made 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 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 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 terminological databases for use in standardization at the following addresses:

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

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

**3.1**
**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: The reference value of 100 % biobased carbon is given in Table 2.

**3.2**
**$^{14}C$ 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.

## 4 Principle

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

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 Formula (1):

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

where

$\chi_B^{TC}$ 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.

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.



## 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 for rubber products and raw materials

### 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 to determine the $^{14}C$ content with their own equipment or to outsource the determination process to other laboratories who 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).

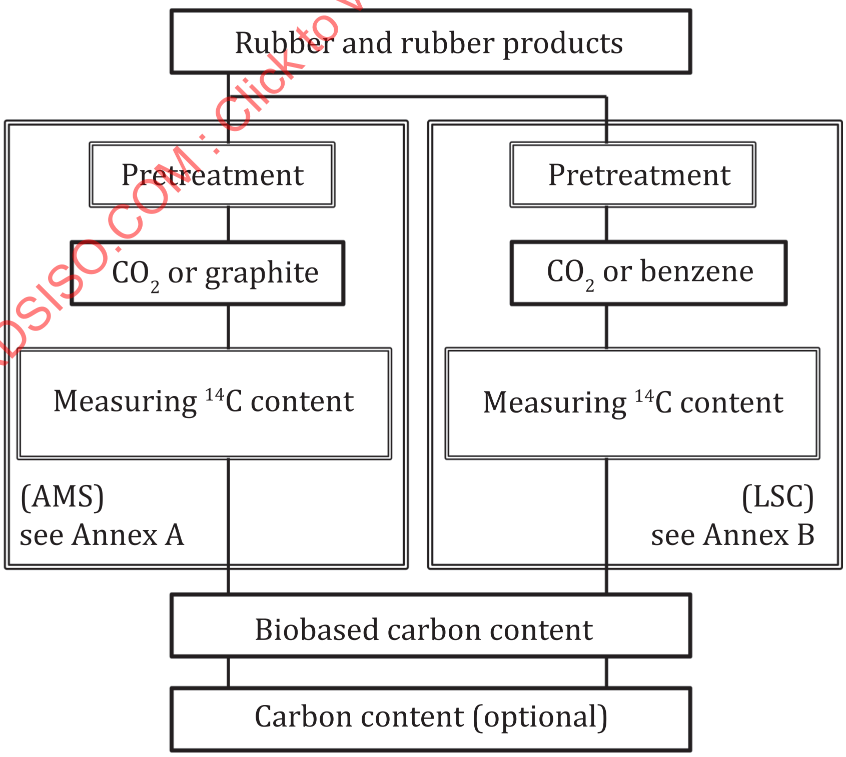



**Figure 1 — Outline for the determination of biobased carbon content**

## 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 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, $\chi_B^{TC}$, 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 (started around 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 sample grown before 1955 has a well-defined "modern" $^{14}C$ activity, and the fossil contribution could be determined in a straightforward way. After that, $^{14}C$ generated during the bomb-testing era increased the atmospheric $^{14}C$ level up to 200 % (pMC) and 27,12 dpm ($^{14}C$ activity) in 1962. The values declined gradually, however, to 101 % (pMC) and 13,70 dpm ($^{14}C$ activity), respectively by 2017, because the large emission of fossil C during the last decades have contributed to the decrease of the atmospheric $^{14}C/^{12}C$ ratio. The REF value of 100 % biobased carbon is indicated in Table 2 in accordance with ASTM D6866.



**Table 2 — REF value of 100 % biobased carbon in determined year**

| Year | REF (pMC, %) |
|---|---|
| 2015 | 102,0 |
| 2016 | 101,5 |
| 2017 | 101,0 |
| 2018 | 100,5 |
| 2019 | 100,0 |
| 2020 | to be determined |

For the calculation of the biobased carbon content, a $^{14}C$ content of 101 pMC [*REF* used in Formula (2) and Formula (3)] is considered as a 100 % biobased carbon content for biomass in 2017.

NOTE A hundred (100) % pMC obtained by AMS measurement (Method A) corresponds to 13,56 dpm obtained by LSC measurement (Method B). A hundred and one (101) % pMC for biomass carbon harvested in 2017 corresponds to 13,70 dpm.

### 6.3.3 Calculation of $\chi_B^{TC}$, the biobased carbon content by Method A (AMS)

Calculate the biobased carbon content as a fraction of total carbon, $\chi_B^{TC}$, expressed as a percentage, using Formula (2) (see Annex A):

$$\chi_B^{TC} = \frac{pMC_S}{REF} \times 100 \tag{2}$$

where

$pMC_S$ is the measured value, expressed in pMC, of the sample;

*REF* is the reference value, expressed in pMC (see 6.3.2).

### 6.3.4 Calculation of $\chi_B^{TC}$, the biobased carbon content by Method B (LSC)

Calculate the biobased carbon content as a fraction of total carbon, $\chi_B^{TC}$, expressed as a percentage, using Formula (3) (see Annex B):

$$\chi_B^{TC} = \frac{^{14}C_{activity}}{13{,}56 \times \frac{REF}{100} \times m} \times 100 \tag{3}$$

where

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

*REF* is the reference value, expressed in pMC, of the sample (see 6.3.2);

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

### 6.3.5 Examples

For examples of biobased carbon content determination, see Annexes C and D.

## 7 Precision

See Annex E.

## 8 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 19984-2;

b) all information necessary for complete identification of the rubber material or product tested, including the origin of the biomass from which the material or product is constituted;

c) test method used for the determination of the $^{14}C$ content (Method A or B);

d) information on whether $\delta^{13}C$ correction was applied or not (see A.5);

e) method for the oxidation of the carbon (see A.4.1 and B.4.1);

f) $^{14}C$ value expressed in pMC (Method A), or $^{14}C$ activity of the sample expressed in dpm (Method B);

g) REF value used;

h) test results: biobased carbon content by total carbon content, $x_B^{TC}$, expressed as carbon %, of the sample; if the calculating value exceeds 100, the value shall be reported as 100 %;

i) any additional information, including details of any deviations from the test methods and any operations not specified in this document which could have had influences on the results;

j) date of the test.

# Annex A
(normative)

# Method A — Determination by accelerator mass spectrometry (AMS)

## A.1 Principle

This annex provides the procedures to determine $^{14}C$ amount in rubber or rubber products (including polyurethane) by accelerator mass spectrometry (AMS).

To determine $^{14}C$ amount by AMS, the rubber or rubber products shall be converted into carbon graphite. Some AMS apparatus allow to determine $^{14}C$ by the form of $CO_2$. Follow the manufacturer's instruction to select the procedure to be applied.

The determination shall always be accompanied by the reference material determinations at the same time to confirm the accuracy.

## A.2 Reagents and materials

**A.2.1 Standard reference material**, e.g. SRM 4990c[2)].

**A.2.2 Copper oxide (CuO)**.

**A.2.3 Sulfur absorbent** (e.g. Sulfix or silver ribbon).

**A.2.4 Iron or cobalt catalyst**.

**A.2.5 Hydrogen**.

**A.2.6 Dry ice**.

**A.2.7 Organic solvent**, acetone or ethanol.

**A.2.8 Liquid nitrogen**.

**A.2.9 Argon and air**, for elemental analyser.

## A.3 Apparatus

**A.3.1 Balance**, accurate to the nearest 0,1 mg.

**A.3.2 Quartz tube** (I.D.; ca. 5 mm, length; ca. 200 mm).

**A.3.3 Tube furnace**, capable to perform to 1 000 °C adapted to the quartz tube size.

2) 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.

**A.3.4 Elemental analyser**, with sulfur absorbing column.

**A.3.5 Gas manifold apparatus**.

## A.4 Procedure

### A.4.1 Oxidation of samples to obtain $CO_2$

#### A.4.1.1 Procedures

Obtain $CO_2$ by either of the methods in A.4.1.2 or A.4.1.3.

#### A.4.1.2 Quartz tube method with copper oxide (CuO)

This method collects $CO_2$ by mixing homogenized test piece (of 1 mg to 2 mg) with CuO in a sealed, evacuated quartz tube. The sufficient amount of CuO should be added to achieve complete oxidation of all carbons in the test piece. Practice has shown that over 400 mg of CuO shall be added for each 2 mg test piece.

NOTE Under insufficient quantity of CuO, carbon black in rubber compounds, which is usually fossil-based (non-biobased), tends to remain unoxidized, so the relative biobased carbon content is wrongly increased.

Sulfur compounds inhibit conversion of graphite from $CO_2$. When the test piece includes sulfur compounds, put a sulfur absorbent such as sulfix or silver ribbon for this procedure.

The tube is heated to 850 °C to 1 000 °C for 3 h to 5 h. The formed $CO_2$ is collected by breaking the tube using a tube-cracker connected to a gas manifold apparatus.

Purify the collected $CO_2$ in the gas manifold apparatus using liquid nitrogen and dry ice in ethanol or acetone.

#### A.4.1.3 Elemental analyser method

This method collects $CO_2$ using an elemental analyser. Depending on the apparatus, the required amount of the test piece will range from 1 mg to 10 mg.

NOTE Under insufficient oxidation, carbon black in rubber compounds, which is usually fossil-based (non-biobased), tends to remain unoxidized, so the relative biobased carbon content is wrongly increased.

When the test piece includes sulfur compounds, use a sulfur absorbent column connected to the elemental analyser to remove sulfur gases such as $SO_2$ from oxidation gas.

The formed $CO_2$ flows into the gas manifold apparatus connecting to the elemental analyser.

Purify the collected $CO_2$ in the gas manifold apparatus using liquid nitrogen and dry ice in ethanol or acetone.

### A.4.2 Conversion to graphite

If the AMS can measure the obtained $CO_2$ (see A.4.1) directly, this procedure is unnecessary.

Transfer the $CO_2$ to the gas manifold apparatus.



Introduce the gaseous sample in the system as it is released from a quartz tube or after it is trapped in liquid nitrogen followed by the subsequent heating. Then convert the purified $CO_2$ to graphite using an iron or cobalt catalyst with hydrogen gas as follows:

$$CO_2 + 2H_2 \rightarrow C + 2H_2O$$

Repeat the procedure from A.4.1 to A.4.2 with standard reference material.

### A.4.3 Determination of $^{14}C$ concentration

Load the prepared graphite of rubber/rubber products and of standard reference material into the AMS. The equipment counts the number of each carbon isotope ($^{12}C$, $^{13}C$ and $^{14}C$) and indicates the relative ratios of $^{14}C/^{12}C$ as well as $^{13}C/^{12}C$.

## A.5 Calculation of the results

Obtain percent modern carbon ($pMC_S$) values from the $^{14}C/^{12}C$ relative ratio (see A.4.3) using Formula (A.1):

$$pMC_S = \frac{A_S}{A_R} \times pMC_R \quad \text{(A.1)}$$

where

$pMC_S$ is the percent modern carbon value of sample;

$pMC_R$ is the percent modern carbon value of standard reference material;

$A_S$ is the $^{14}C/^{12}C$ relative ratio of radiocarbon of sample;

$A_R$ is the $^{14}C/^{12}C$ relative ratio of radiocarbon of standard reference material.

The obtained percent modern carbon ($pMC_S$) values may be corrected using stable isotope ratio ($^{13}C/^{12}C$) of the test sample and standard reference material. In some areas such as carbon dating, it is commonly employed to correct the results with another isotopic ratio ($^{13}C/^{12}C$). However, the survey in 2013 to 2015 has shown that this correction has little influence on biobased carbon content of rubber samples. As far as possible, it is preferable to have results without any corrections to avoid confusions.

# Annex B
(normative)

# Method B — Determination by liquid scintillation counter (LSC)

## B.1 Principle

This annex provides the procedures to determine $^{14}C$ activity in rubber or rubber products (including polyurethanes) by liquid scintillation counter (LSC).

To determine $^{14}C$ activity by LSC, $CO_2$ obtained from the rubber or rubber products shall be either absorbed to carbamate solution or be converted to benzene.

Each determination shall be accompanied by reference determination to confirm accuracy.

## B.2 Reagents and materials

**B.2.1 Standard reference materials**, e.g. SRM 4990c[3)].

**B.2.2 Scintillation liquid**.

**B.2.3 Absorbent solution**.

## B.3 Apparatus

**B.3.1 Balance**, accurate to the nearest 0,1 mg.

**B.3.2 Bomb calorimeter**, based on ISO 1928.

**B.3.3 Tube furnace**, as specified in ISO 19242.

**B.3.4 Sample oxidizing apparatus**.

**B.3.5 Liquid scintillation counter (LSC)**, with a low level counter.

**B.3.6 Benzene synthesis apparatus**.

**B.3.7 Gas bag**.



3) 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.

## B.4 Procedure

### B.4.1 Combustion of the sample and the standard reference material to obtain $CO_2$

#### B.4.1.1 Procedures

Obtain sufficient amount of $CO_2$ from the rubber sample and the standard reference material in accordance with B.4.1.2 or B.4.1.3. Care has to be taken as the required amount of $CO_2$ varies depending on

— the further cocktail preparation procedures (i.e. the absorbent-solution method in B.4.2.1 or the conversion-into-benzene method in B.4.2.2),

— the sort and the amount of $CO_2$ absorbent used,

— the size of scintillation vial, or

— the sensitivity of the LSC apparatus used.

If sufficient $CO_2$ is not obtained, repeat these methods. For these methods, automated sample oxidizing apparatus such as sample oxidizer can be used.

#### B.4.1.2 Calorimetric bomb method

For the combustion of the test sample and the standard reference material in a calorimetric bomb, any suitable test method such as ISO 1928 may be used.

After the complete combustion in the oxygen bomb, the combustion gases are collected in a gas bag.

#### B.4.1.3 Tube furnace method

For the combustion of the test sample and the standard reference material in a tube furnace, any suitable test method such as ISO 19242 may be used.

The combustion gases from the tube furnace are collected in a gas bag.

NOTE Under insufficient oxidation, carbon black in rubber compounds, which is usually fossil-based (non-biobased), tends to remain unoxidized, so the relative biobased carbon content is wrongly increased.

### B.4.2 Preparation of cocktail for LSC test

#### B.4.2.1 Absorption to absorbent solution

The obtained $CO_2$ (see B.4.1) in the gas bag is absorbed in $CO_2$ absorbent solution under the suitable conditions depending on the used absorbent and the used LSC apparatus.

Transfer the absorbent to the measuring vial and add an equal volume of the scintillation liquid and homogenize the mixture for used LSC apparatus.

#### B.4.2.2 Benzene synthesis from $CO_2$

Refer to ASTM D6866-16, 12.7 to 12.12. Automated apparatus for benzene synthesis can be used. Benzene is synthesized from the obtained $CO_2$ (see B.4.1).

The obtained benzene is added to an organic scintillator with suitable ratio for used scintillator and used LSC apparatus.

#### B.4.2.3 $^{14}C$ activity measurement by LSC

Place the vial of the cocktail (see B.4.2) in the LSC to measure $^{14}C$ activity, i.e. β counts of $^{14}C$ decay. Typical counting times are 4 h to 24 h. Perform measurements both for rubber-origin cocktail and reference cocktail. Subtract the background count rate from the sample count rate to obtain the net count rate. The $^{14}C$ activity (dpm) is obtained by normalizing the net count rate to the count rate of the standard reference material (oxalic acid, e.g. SRM 4990c).

# Annex C
(informative)

# Examples of the determination of biobased carbon content for rubber and rubber products

Table C.1 indicates measuring results of the biobased carbon contents for rubber compound and polyurethane determined by AMS. Theoretical values of biobased carbon contents are calculated based on ISO 19984-1. Table C.2 indicates measuring results of the biobased carbon contents for rubber products such as parts of tyre and a rubber glove.

**Table C.1 — Examples of calculating and measuring biobased carbon contents for rubber compounds and polyurethane**

| NO | Outline of compound recipe | | | | Biobased carbon content, % | |
|---|---|---|---|---|---|---|
| | Rubber | Characteristic ingredient | Carbon black (CB), phr | Silica, phr | Calculating value, % | Measuring results, % |
| 1 | NR/BR = 60/40 | None | 35 | 0 | 39,2 | 39,2 |
| 2 | NR = 100 | None | 45 | 0 | 61,0 | 60,4 |
| 3 | NR/S-SBR = 70/30 | None | 40 | 15 | 41,8 | 42,0 |
| 4 | NR/S-SBR = 20/80 | None | 10 | 45 | 13,1 | 13,7 |
| 5 | NR/BIIR = 30/70 | None | 45 | 0 | 19,7 | 21,2 |
| 6 | ENR = 100 | None | 5 | 60 | 78,2 | 79,8 |
| 7 | NR = 100 | Castor oil | 45 | 0 | 67,4 | 65,7 |
| 8 | S-SBR = 100 | Phenoic resin | 10 | 70 | 0,7 | 2,0 |
| 9 | NR/S-SBR = 80/20 | Resin partly from biomass | 50 | 0 | 45,9 | 44,5 |
| 10 | NR = 100 | CB from rapeseed oil | 30 | 0 | 70,8 | 71,1 |
| 11 | polyurethane | Biobased polyol | — | — | 69,6 | 67,8 |
| NOTE NR: Natural rubber, BR: Butadiene rubber, S-SBR: Solution polymerized styrene-butadiene rubber, BIIR: Bromobutyl rubber, ENR: Epoxidized NR. | | | | | | |

**Table C.2 — Examples of biobased carbon contents for rubber products**

| Sample name | Biobased carbon content, % |
|---|---|
| Rubber glove | 100,0 |
| Tread of tyre | 5,9 |
| Sidewall of tyre | 18,8 |
| Undertread of tyre | 47,0 |

# Annex D
(informative)

# Determination of the biobased carbon content of a tyre



## D.1 General

To obtain the result below, cut samples of a tyre were prepared and each size of the cross-sections of different components/layers was calculated first. The circumference and the rubber content relevant to each component were multiplied then. Finally, from the biobased carbon content determined in accordance with this document and the ratio of each section by mass, the whole tyre's biobased carbon content was calculated.

## D.2 Preparation of cut samples of tyre

**D.2.1** Upon cutting out samples, pay attention to avoid joint parts of the tyre.

**D.2.2** Finish the cut samples with buffing such that the structure of the surface is shown clearly.

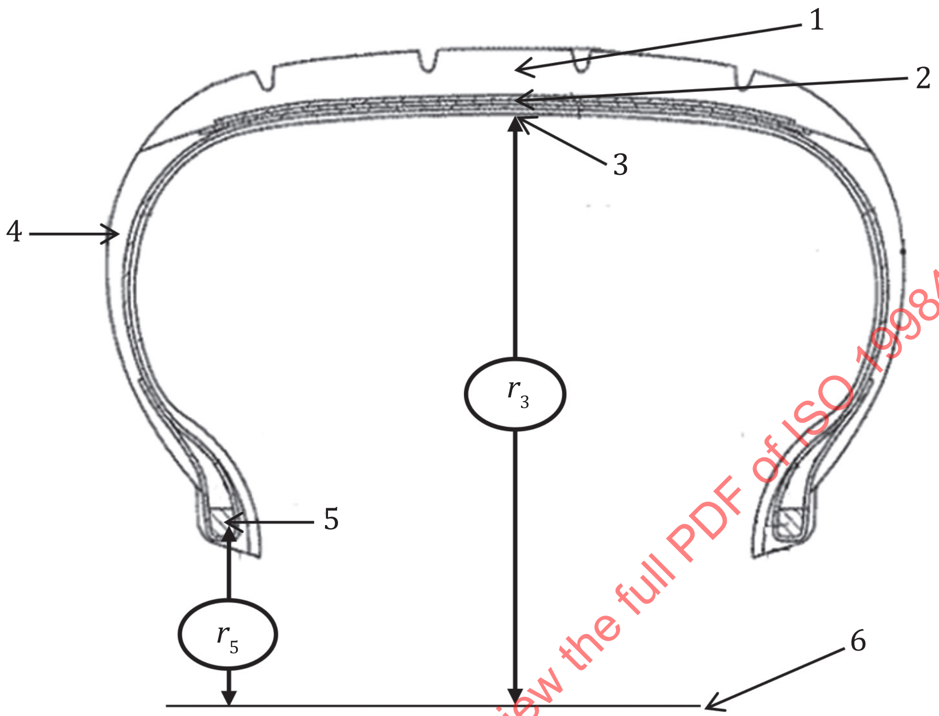
## D.3 Determination of the size of cross-section

Take digital pictures of the cut surfaces, and calculate the area size ($a_i$) of each section using a planimetry software such as ImageJ™[4)] or ImageMeasure ™[4)].

4) ImageJ and ImageMeasure are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

## D.4 Determination of composition for each component of tyre

**D.4.1** Determine the radius ($r_i$), i.e. the straight length between the centre of the tyre and the gravity point of each component's cut surface. Examples of the gravity point of typical tyre components such as tread, sidewall, inner-liner, rubberized belts and bead-filler are shown in Figure D.1.




**Key**

1 gravity point of tread
2 gravity point of rubberized belts
3 gravity point of inner-liner
4 gravity point of sidewall
5 gravity point of bead-filler
6 centre line of the tyre
$r_i$ length between the centre line and the gravity point of each component in the tyre

**Figure D.1 — Gravity points of typical tyre components**



**D.4.2** Calculate the mass of each component ($w_i$) according to Formula (D.1):

$$w_i = a_i \times 2r_i \times \pi \times d_i \quad (D.1)$$

where

$w_i$ is the mass of the component (g);

$a_i$ is the size of cross-section of the component ($cm^2$);

$r_i$ is the length between the centre line of the tyre and the gravity point of the component (cm);

$d_i$ is the degree of density of the component, determined in accordance with ISO 2781.