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Rubber and rubber products — Determination of biobased content —

Part 3: Biobased mass content

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

Partie 3: Teneur en masse biosourcée

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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 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 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.

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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.

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Rubber and rubber products — Determination of biobased content —

Part 3:
Biobased mass 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 the biobased mass contents in rubber and rubber products.

The biobased mass content of rubber materials or final products can be determined by separating the sample into the rubber, the inorganic ingredient (including carbon black) and the solvent extract, each of whose ^{14}C content is determined, and then converting the results to the biobased mass content using the procedure described in this document.

This document applies to NR, IR, BR, SBR, IIR, BIIR, CIIR and their modified rubbers, as well as to their mixtures.

This document uses only the accelerator mass spectrometry (AMS) method for the purpose of determination of biobased content.

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 1382, *Rubber — Vocabulary*

ISO 1407:2011, *Rubber — Determination of solvent extract*

ISO 4650, *Rubber — Identification — Infrared spectrometric methods*

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

ISO 9924-3, *Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry — Part 3: Hydrocarbon rubbers, halogenated rubbers and polysiloxane rubbers after extraction*

ISO 17257, *Rubber — Identification of polymers — Pyrolytic gas-chromatographic method using mass-spectrometric detection*

ISO 19984-1:2017, *Rubber and rubber products — Determination of biobased content — Part 1: General principles and calculation methods using formulation of the rubber compound*

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ISO 19984-2:2017, *Rubber and rubber products — Determination of biobased content — Part 2: Biobased carbon content*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382, ISO 19984-1 and ISO 19984-2 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

4 Principle

As described in ISO 19984-2, measuring ^{14}C in a rubber sample only allows to determine its biobased carbon content. When the sample consists of a single component such as a raw rubber material or a certain chemical, the biobased carbon content equals to the biobased mass content theoretically. However, when the sample is a mixture of components each of which has different amount of carbon included, the two biobased content values will be different because of the varied carbon amount as well as the varied formulation ratio. Therefore, in order to determine the biobased mass content of the whole, the information of each component's biobased carbon content and the formulation ratio is required. For a vulcanized rubber, each content such as rubber, inorganic ingredients (including carbon black) and solvent extract, is to be analysed for calculations.

5 Separation of a vulcanized rubber sample and determination of the biobased mass content

5.1 General

To separate a vulcanized rubber sample, employ a series of thermal decomposition methods. Any organic ingredients that are free from cross-linking formations are extracted by the solvent. Inorganic filler ingredients, including carbon black, are to be separated by thermogravimetry. If it is known that there are carbonate included in the filler, apply hydrochloric acid treatment to remove them. As a result, the vulcanized rubber is to be separated into rubber, inorganic fillers (including carbon black and excluding carbonate) and solvent extract. When the rubber sample is unvulcanized, vulcanize it before the separation procedure.

The flow chart of the separation scheme of a vulcanized rubber is shown in Figure 1.

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