
**Rubber and rubber products —
Determination of combustion energy
and carbon dioxide emission from
biobased and non-biobased materials**

*Élastomères et produits à base d'élastomères — Méthode de
détermination de l'énergie de combustion et de l'émission de CO₂ des
matériaux biosourcés et non biosourcés*

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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).

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. (standards.iteh.ai)

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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Introduction

To reduce the use of exhaustible fossil resources such as petroleum, coal, or natural gas, as well as the amount of carbon dioxide emission from those during rubber production process or waste disposal, it is very important to shift the raw materials from fossil-based resources to “biomass” resources. Biomass includes starch, cellulose, hemicellulose or lignin which living plants photosynthesize converting the carbon dioxide in the atmosphere. It is preferred to utilize inedible biomass such as agricultural waste or food-industries’ wastes rather than using edible biomass. Using biobased resources instead of fossil-based ones will benefit to make sustainable social systems and to preserve the global environment.

Products that are produced fully or partially from biomass resources are “biobased” products. Many rubber products today include natural rubber as component, so there are many biobased products in the rubber market already. That is a great advantage for the rubber industry to contribute to sustainable social systems.

Recycling chemical products is an important act to preserve limited resources, and basically there are two ways to recycle end-of-life rubber products, i.e. “material recycling” and “thermal recycling”. It is useful to develop concrete indices to evaluate the effect of thermal recycling.

This document specifies how to determine the biobased combustion energy and the amount of biobased carbon dioxide emission hoping to promote rubber-product waste as an alternative fuel.

This document introduces the idea of biobased combustion energy as an index to examine the degree of contribution of thermal recycling of rubber wastes. At the same time, the amount of biobased carbon dioxide emission from the thermal recycling process will act as a direct comparison to the fossil-based carbon dioxide emission.

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Rubber and rubber products — Determination of combustion energy and carbon dioxide emission from biobased and non-biobased materials

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 the measuring methods of the combustion energy (i.e. gross calorific value) and the carbon dioxide emission amount from biobased and non-biobased materials in rubber or rubber products.

This document applies to rubber and rubber products (including polyurethane) 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 1382, *Rubber — Vocabulary*

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

ISO 1928, *Solid mineral fuels — Determination of gross calorific value by the bomb calorimetric method and calculation of net calorific value*

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

ISO 19984-2, *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 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 <https://www.iso.org/obp>

3.1

biobased component

biobased part of a biobased constituent which is wholly or partly from biomass resource(s)

[SOURCE: ISO 19984-1:2017, 3.2]

3.2

biobased carbon content

biobased component(s) (3.1) in a product expressed by carbon% to total carbon

[SOURCE: ISO 19984-1:2017, 3.4]

3.3

biomass

material of biological origin excluding material embedded in geological formations and/or fossilized

[SOURCE: ISO 19984-1:2017, 3.6]

3.4

biobased combustion energy

energy obtained from the combustion of the biobased carbon contained in rubber or a rubber product

Note 1 to entry: The combustion energy is measured as gross calorific value or net calorific value.

Note 2 to entry: The biobased combustion energy is expressed in J/g, or calorific value (joules, J) per sample mass (g).

3.5

biobased carbon dioxide emission

amount of carbon dioxide emitted from the biobased carbon contained in rubber or a rubber product

Note 1 to entry: The biobased carbon dioxide emission is expressed in g/g, or emitted carbon dioxide amount (g) per sample mass (g).

3.6

gross calorific value

absolute value of the specific energy of combustion, for unit mass of rubber or rubber-product sample burned in oxygen in a calorimetric bomb under specified conditions

Note 1 to entry: The products of combustion are assumed to consist of gaseous oxygen, nitrogen, carbon dioxide and sulfur dioxide, of liquid water (in equilibrium with its vapour) saturated with carbon dioxide under the conditions of the bomb reaction, and of solid ash, all at the reference temperature.

Note 2 to entry: The gross calorific value is expressed in J/g.

3.7

net calorific value

absolute value of the specific energy of combustion, for unit mass of rubber or rubber-product sample burned in oxygen in a calorimetric bomb under such conditions that all the water of the reaction products remains as water vapour, the other products being as for the *gross calorific value* (3.6), all at the reference temperature

Note 1 to entry: The net calorific value is expressed in J/g.

4 Principle

A sample from rubber or a rubber product is completely combusted in a pressure-proof sealed vessel (bomb) with high pressure oxygen gas settled in an insulated area. The combustion energy of the sample is calculated from the increased heat in the insulated area. The carbon dioxide emission amount is determined by measuring both the combustion gas volume collected from the used bomb and its carbon dioxide concentration.

The biobased combustion energy and the biobased carbon dioxide emission can be calculated in proportion by multiplying the obtained values by the biobased carbon content.

NOTE There are two kinds of combustion energies, i.e. gross calorific value and net calorific value (see [Annex A](#) for information). This document specifies the determination of gross calorific value of rubber and rubber products.

5 Sampling

For raw rubber, carry out sampling in accordance with ISO 1795. For vulcanized rubber, carry out sampling in accordance with ISO 4661-2.

6 Determination of the combustion energy of biobased and non-biobased materials

6.1 General

This method specifies how to determine the combustion energy of rubber or a rubber product using a bomb calorimeter. A high-pressure proof sealed bomb is used as a measuring vessel. A test sample is placed in the bomb filled with high-pressure oxygen with an ignition wire contacting the sample. The bomb is then placed in a water vessel the temperature of which is accurately controlled and measured. The test sample is combusted by igniting the wire and the calorific value is determined by the temperature increase, the volume of water in the water vessel (or the heat capacity of calorimeter) and the heat capacity of the bomb.

The combustion system is calibrated by combusting the calorimetric standard, i.e. certified benzoic acid.

If the carbon dioxide emission amount is to be measured in the later process, the combustion gas in the bomb shall be collected and used for the determination.

6.2 Reagents and materials

6.2.1 Oxygen, at a pressure high enough to fill the bomb to 3 MPa, pure, with an assay of at least 99,5 % volume fraction, and free from combustible matter.

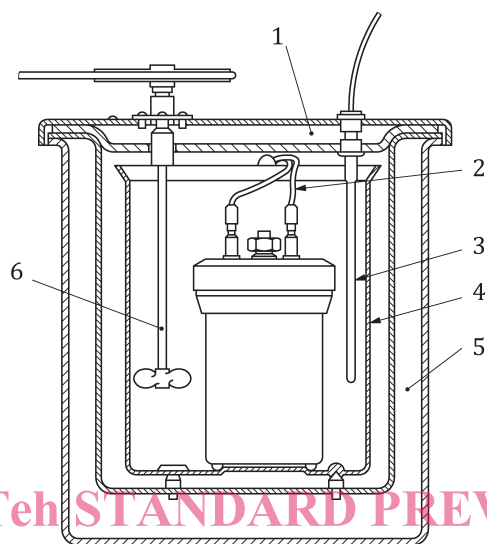
NOTE Oxygen made by the electrolytic process can contain up to 4 % volume fraction of hydrogen.

6.2.2 Benzoic acid, of calorimetric-standard quality, certified by (or traceable to) a recognized standardizing authority.

6.3 Apparatus

6.3.1 Bomb calorimeter

The calorimeter (see [Figure 1](#)) consists of the assembled combustion bomb, the calorimeter can (with or without a lid), the calorimeter stirrer, water, temperature sensor and leads with connectors inside the calorimeter can required for ignition of the sample or as part of temperature measurement or control circuits. The calorimeter shall conform to ISO 1928 or provide the equivalent test results. During measurements, the calorimeter is enclosed in a thermostat. The manner in which the thermostat temperature is controlled defines the working principle of the instrument and, hence, the strategy for evaluating the corrected temperature rise.



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Key

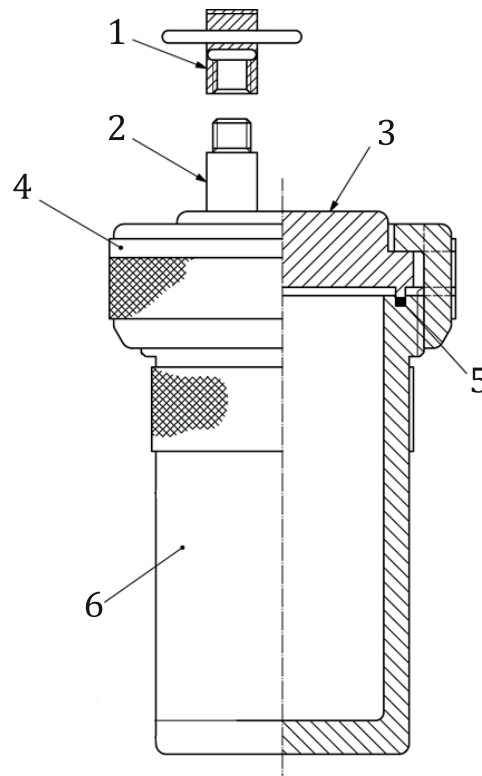
- | | | | |
|---|----------------|---|-----------------|
| 1 | thermostat lid | 4 | calorimeter can |
| 2 | ignition leads | 5 | thermostat |
| 3 | thermometer | 6 | stirrer |

Figure 1 — Classical-type combustion-bomb calorimeter with thermostat

6.3.2 Combustion bomb, capable of withstanding safely the pressures developed during combustion; see [Figures 1](#) and [2](#).

The material of construction shall resist corrosion by the acids produced in the combustion of rubber or a rubber product. A suitable internal volume of the bomb is from 250 ml to 350 ml and it is preferred that the bomb is equipped with a relief valve or a bursting disc.

WARNING — Bomb parts shall be inspected regularly for wear and corrosion; particular attention shall be paid to the condition of the threads of the main closure. Manufacturers' instructions regarding the safe handling and use of the bomb shall be observed. When more than one bomb of the same design is used, it is imperative to use each bomb as complete unit. Colour coding is recommended. Swapping of parts can lead to a serious accident.



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Key

- | | | | |
|---|---------------|---|--------------|
| 1 | valve cover | 4 | closure ring |
| 2 | valve housing | 5 | sealing ring |
| 3 | cap | 6 | vessel body |

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Figure 2 — Typical calorimeter bomb

6.3.3 Fuse

6.3.3.1 Ignition wire, of nickel-chromium 0,10 mm to 0,20 mm in diameter, platinum 0,05 mm to 0,10 mm in diameter, or another suitable conducting wire with well characterized thermal behaviour during combustion.

6.3.3.2 Cotton fuse, of white cellulose cotton, or equivalent.

6.3.4 Pressure regulator, to control the filling of the bomb with oxygen.

6.3.5 Pressure gauge (e.g. 0 MPa to 6 MPa), to indicate the pressure in the bomb with a resolution of 0,05 MPa.

6.3.6 Relief valve or bursting disc, operating at 3,5 MPa, and installed in the filling line, to prevent overfilling the bomb.

CAUTION — Equipment for high-pressure oxygen shall be kept free from oil and grease. Do not test or calibrate the pressure gauge with hydrocarbon fluid.

6.3.7 Balance, capable of weighing the sample, ignition wire, etc., with a resolution of at least 0,1 mg.