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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 CO_2 des matériaux biosourcés et non biosourcés

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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

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.

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.
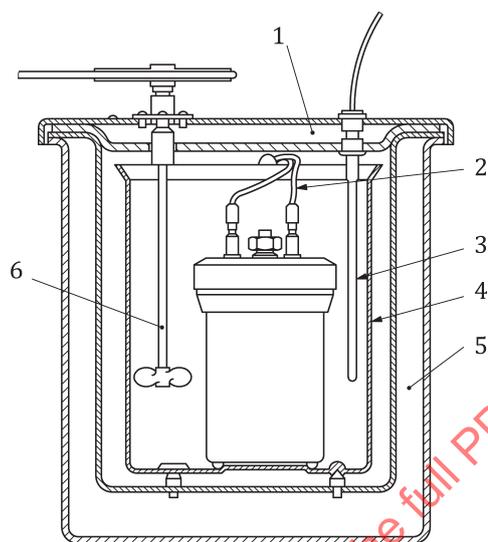
Key

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.

Key
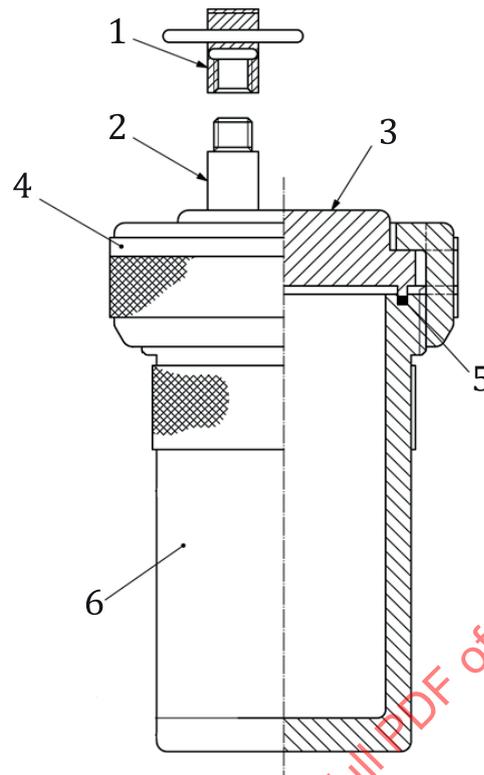
1 valve cover
2 valve housing
3 cap

4 closure ring
5 sealing ring
6 vessel body

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.

6.4   Procedure

6.4.1   Preparation of test sample

Weigh out 0,3 g to 0,8 g of the sample to the nearest 0,1 mg. Block, sheet, powder or liquid sample can be used. The amount of sample shall correspond to the measurable energy range of the calorimeter used.

6.4.2   Calibration of calorimeter

Weigh approximately 1 g of benzoic acid pellet certified for combustion energy measurement and place it on a combustion dish in the bomb. Settle an ignition wire so that it touches this pellet and connects to the ignition electrode of the ignition circuit. Close the vessel body and tighten the closure ring firmly.

Connect the pipe from the oxygen pressure regulator to the valve of the combustion bomb. The pipe shall be certified to perform under 3,5 MPa pressure and have a branched purge line with an on-off valve.

Open the bomb valve and control the regulator to gradually fill oxygen in the bomb. When the pressure reaches 2,5 MPa to 3,0 MPa, close both the bomb valve and the regulator valve, and remove the pipe from the bomb.

Immerse the sealed, assembled bomb in the water vessel. Watch for babbles in water and make sure there is no oxygen leak.

When the water temperature has become stable, combust the benzoic acid pellet by applying voltage to the ignition wire.

Adjust the obtained combustion energy as 26 460 J/g of benzoic acid. Repeat the same procedure for another pellet until two consecutive measured values fall within 26 460 J/g ± 80 J/g.

6.4.3   Measurement of combustion energy

Place the accurately weighed test sample (6.4.1) on the combustion dish in the bomb. Settle the ignition wire so that it touches the sample and connects to the ignition electrode of the ignition circuit. Close the vessel body and tighten the closure ring firmly.

Connect the pipe from the oxygen pressure regulator to the valve of the combustion bomb. The pipe shall be certified to perform under 3,5 MPa pressure and have a branched purge line with an on-off valve.

When the amount of carbon dioxide emission is to be measured afterwards (see Clause 7), purge the inside air with oxygen. To do that, connect an oxygen bottle, introduce ca. 0,2 MPa oxygen and evacuate the oxygen and the inside air together either by removing the pipe or by using the branched purge line. Repeat the process at least twice.

Open the bomb valve and control the regulator to gradually fill oxygen in the bomb. When the pressure reaches 2,5 MPa to 3,0 MPa, close both the bomb valve and the regulator valve, and remove the pipe (if a purge line is equipped, release oxygen before removing the pipe).

Immerse the sealed, assembled bomb in the water vessel. Watch for babbles in water and make sure there is no oxygen leak.

When the water temperature has become stable, combust the test sample by applying voltage to the ignition wire. Read the measured value for combustion energy.

Repeat the measurement procedure for the same test sample and compare the results. When the difference falls into ±160 J/g, finish the measurement and take the mean value as the total combustion energy.

6.4.4   Measurement of biobased carbon content

To determine the biobased combustion energy (E_B) of the test sample, information on its biobased carbon content (x_B) is indispensable. If the sample's chemical formulation and the resource of each

component (i.e. the biobased carbon content of each component) is available, the biobased carbon content of the sample can be calculated in accordance with ISO 19984-1. When those are unavailable, the biobased carbon content shall be measured in accordance with ISO 19984-2.

6.5 Calculation method of biobased and non-biobased combustion energy

The biobased combustion energy (E_B) can be calculated by applying the total combustion energy (E) measured in 6.4.3 to Formula (1). Non-biobased combustion energy (E_{NB}) can be derived by subtracting E_B from E. This calculation is based on the fact that the combustion energy is in proportion to the total carbon amount in a product, which is the sum of biobased and fossil-based (non-biobased) carbon amounts.

$$E_B = E \times x_B/100 \tag{1}$$

where

E_B is the biobased combustion energy (J/g);

E is the combustion energy (gross calorific value) (J/g);

x_B is the biobased carbon content (%).

Examples of results for measuring and calculating of combustion energy are shown in Annex B.

The biobased carbon content, x_B, i.e. the ratio of the biobased carbon to the total carbon in rubber or a rubber product, can be calculated in accordance with ISO 19984-1 when the chemical formulation and the information of each component are known. When no such information is available, it can be determined in accordance with ISO 19984-2.

If any materials are used at the time of combustion such as ignition wires, combustion improvers or sample holders, the combustion energy of those should be measured in advance. Then the value shall be subtracted from the whole value to obtain the true amount of the combustion energy of the test sample.

7 Determination of the amount of carbon dioxide emission from biobased and non-biobased materials

7.1 General

This clause specifies the method to determine the amount of carbon dioxide emission from rubber or rubber products as well as the combustion energy measurement (see Clause 6). The carbon dioxide and the oxygen are collected in a gas sampling bag from the bomb, and the carbon dioxide emission amount is determined from its concentration and the volume ratio compared to the total gas collected.

The gas analysing system is calibrated by the reference gas with certified concentration of carbon dioxide in nitrogen gas. The validity of this measurement is confirmed by the amount of carbon dioxide emission from benzoic acid used as a calorimetric standard.

7.2 Apparatus

7.2.1 Gas sampling bag, sufficient enough for combustion gas volume in the bomb under atmospheric pressure (preferably of 10 l to 20 l size), with a cock. If the sample gas is to be inserted by a micro syringe to the gas chromatograph (7.2.2), it is recommended to use a bag with a rubber-septum-mounted sampling port.

7.2.2 Gas chromatograph (GC), with the following specifications:

— detector, i.e. thermal conductive detector (TCD) or helium ionization detector (HID);

— column, such as molecular sieve, active carbon, or porous polymer (ethylvinylbenzene-divinylbenzene) column, to separate carbon dioxide and other gases;

— injector, a septum rubber port for the gas tight syringe or a gas sample injector with a gas sampling valve and a sample loop, of the volume of the gas to be determined (0,5 ml to 1,0 ml).

7.2.3   Gas volume meter, capable to measure gas volume (of carbon dioxide and other gases) over 30 l with resolution of 10 ml.

7.2.4   Vacuum sampling bottles, of 100 ml and 1,0 l volume, to prepare the gas for calibration curve with determined concentration of standard carbon dioxide gas in inert dilution gas such as oxygen or nitrogen gas.

7.2.5   Gas tight syringes, of 5 ml and 20 ml volume, to dilute the standard carbon dioxide inserting to vacuum sampling bottle or inject the gas to GC.

7.2.6   Vacuum pump, of 10 l/min to 20 l/min exhaust velocity, to evacuate the remaining gas in the tube, the cock and the gas sampling bag connected to the bomb.

7.2.7   Air pump, of 1 l/min to 2 l/min speed to meet the gas volume meter specification, to transfer the gas from the gas sampling bag to the gas volume meter.

7.2.8   Thermometer, capable to measure the room temperature to the nearest 1 °C.

7.2.9   Atmospheric pressure meter, capable to measure the atmospheric pressure to the nearest 0,1 kPa.

7.3   Reagents

7.3.1   Standard gas (carbon dioxide), to calibrate the gas chromatograph, with an assay of at least 99,9 % volume fraction, traceable to national standards.

Observe the manufacturer's expiry date or recommended shelf-life.

7.3.2   Dilution gas, such as nitrogen, argon, helium or oxygen gas, of purity greater than 99,9 % and free of carbon dioxide, to prepare the gas for calibration curve of carbon dioxide.

7.3.3   Carrier gas for gas chromatograph, such as pure helium gas, with an assay of at least 99,99 % volume fraction.

7.3.4   Water, for determination of the internal volume of the bomb.

Not necessary if the volume specification is provided by the manufacturer.

7.4   Procedure

7.4.1   Verification of the measurement

Use benzoic acid as the reference material. The amount of carbon dioxide emission from benzoic acid is measured after the calibration of calorimeter. The measured value shall be 2,525 g/g ± 0,1 g/g. If not, procedures and apparatus are checked and adjusted to obtain this valid value.

7.4.2 Transfer of the combustion gas to a gas sampling bag from bomb

Transfer the carbon dioxide, other gases and remaining oxygen from the bomb to a gas sampling bag in the following procedure.

a) Take out the sealed bomb from the water vessel after the combustion energy measurement (see Clause 6).

b) Connect a gas sampling bag with a cock to the bomb as indicated in Figure 3.

c) To evacuate the remaining gas in tubes or a gas sampling bag, connect a vacuum pump to a three-neck valve with position 4 and deaerate inside. Then set the three-neck valve to position 5.

d) Open the cock of the gas sampling bag and gradually release the valve (2 in Figure 3) to transfer the combustion gas inside the bomb into the gas sampling bag.

e) After transfer, close the cock (7 in Figure 3) and the valve and remove the gas sampling bag from the bomb.
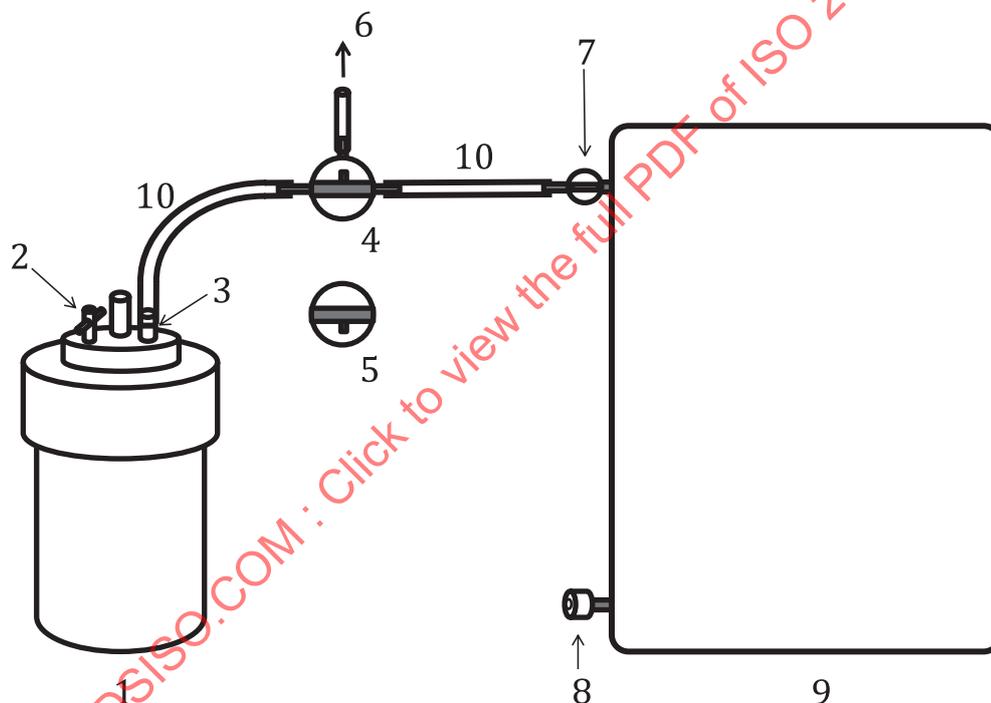
Key

1	bomb after measuring for combustion energy	6	connection to vacuum pump
2	valve housing	7	cock
3	oxygen gas insertion port	8	gas sampling port with septum rubber
4	three neck valve (vacuum gas position)	9	gas sampling bag
5	three neck valve (gas transfer position)	10	gas tube

Figure 3 — Typical gas transfer connection from bomb to gas sampling bag

7.4.3 Measurement of carbon dioxide concentration by GC

7.4.3.1 GC measurement conditions

Employ the conditions optimized for the used column and detector in instruction manuals.

For example, the conditions for Porapak-Q[TM1)] (length 2 m, inside diameter 3 mm) and TCD detector are as follows:

— injection temperature: 170 °C;

— detector temperature: 130 °C;

— column temperature: 130 °C (isothermal);

— flow rate of carrier gas: 20 ml/min.

7.4.3.2 Calibration curve for carbon dioxide

7.4.3.2.1 Preparation of standard gases for calibration curve

Prepare standard gases for calibration curve, of more than 4 different concentrations of carbon dioxide in dilution gas such as nitrogen, argon, helium or oxygen gas. The concentrations should range around the expected carbon dioxide concentration of the combustion gas to be determined.

7.4.3.2.2 Preparation of calibration curve

Make a calibration curve between the carbon dioxide concentration and the peak area in GC chart as follows.

a) Inject 300 µl to 500 µl of the standard gas (7.4.3.2.1) to the gas chromatograph using a gas-tight syringe. When a helium ionization detector (HID) is equipped, control the injection volume in accordance with the detector's sensitivity.

b) Determine the area of carbon dioxide peak from the GC chart.

c) Plot a calibration curve between carbon dioxide concentration (x-axis) and peak area of its peak in GC chart (y-axis) by the linear least-square with approximate expression of a quadratic function. Repeat the plot if the correlation coefficient R^2 is smaller than 0,99.

##### 7.4.3.2.3 Determination of the carbon dioxide concentration of the combustion gas

Collect the combustion gas from the gas sampling port (8 in Figure 3) of a gas sampling bag using a gas-tight syringe. Inject the same volume of this gas as the standard gas used in 7.4.3.2.2, and determine the carbon dioxide concentration of the gas from the obtained peak area and the calibration curve.

7.4.4 Determination of the total volume of the combustion gas

Determine the total gas volume of the combustion gas as follows.

a) Measure the volume of the combustion gas in the gas sampling bag using a gas volume meter suitable for carbon dioxide and oxygen (7.2.3) connected as in Figure 4.

b) Add the measured volume to the inside volume of the bomb known as provided by the manufacturer, to determine the total volume of the combustion gas (V). If the volume of the bomb is unknown, it can be calculated by weighing the bomb filled with water and subtracting the weight of the empty bomb from it. Measure the room temperature near the weighed bomb to the nearest 1 °C and convert the mass value of the inside water into the volume.

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1) Porapak-Q is an example 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.
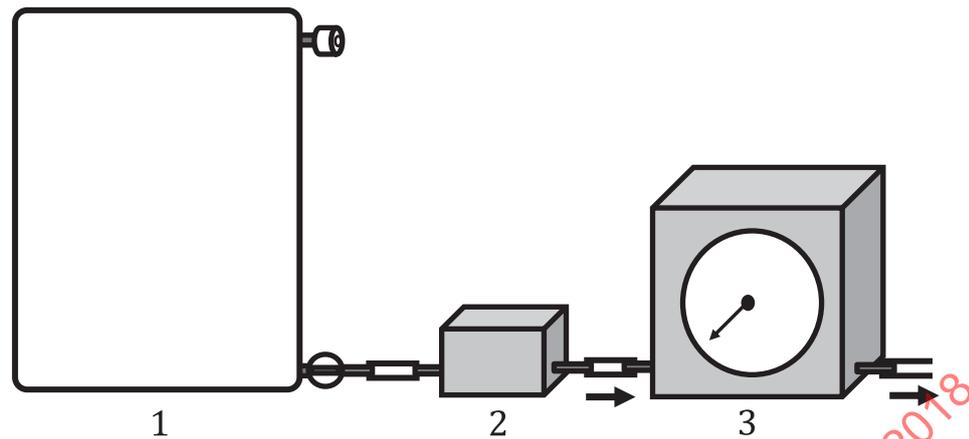
Key

1   gas sampling bag
2   air pump
3   gas volume meter

Figure 4 — Connection of the gas sampling bag, air pump and gas volume meter

7.4.5   Measurement of the room temperature and the atmospheric pressure

Measure the temperature near the gas volume meter (T_M) to the nearest 1 °C and the atmospheric pressure (P) to the nearest 0,1 kPa.

7.4.6   Calculation of the amount of carbon dioxide emission

The amount of carbon dioxide emission from the test sample (W) is calculated using Formula (2).

$$W = \frac{C}{100} \times \frac{V}{22,7} \times \frac{273}{T_M + 273} \times \frac{P - P_t}{100} \times \frac{44,01}{m} \tag{2}$$

where

W is the amount of carbon dioxide emission (g/g);

C is the carbon dioxide concentration (%);

V is the total gas volume (l);

T_M is the measured temperature (°C);

P is the atmospheric pressure (kPa);

P_t is the saturated water vapour pressure at measuring temperature (kPa);

m is the mass of the test sample (g).

To determine the amount of carbon dioxide emission (W) from the test sample under standard conditions, the W is calculated from the measured C and V values with corrections by measuring T_M, P, and P_t as indicated in Formula (2). For P_t, use the value at measured temperature specified in Annex C.

If any materials are used at the time of combustion such as ignition wires, combustion improvers or sample holders, the amount of carbon dioxide emitted from those should be measured in advance. Then the value shall be subtracted from the whole value to obtain the true amount of the carbon dioxide emitted from the test sample.

7.5 Calculation of biobased and non-biobased carbon dioxide emission

The amount of biobased carbon dioxide emission (W_B) is calculated by applying the measured carbon dioxide emission (W) to Formula (3). Non-biobased carbon dioxide emission (W_{NB}) can be obtained by subtracting W_B from W (the information can be found in Annex D).

$$W_B = W \times x_B/100 \tag{3}$$

where

    x_B is the biobased carbon content (%);

    W_B is the biobased carbon dioxide emission (g/g);

    W is the carbon dioxide emission (g/g).

The biobased carbon content (x_B), i.e. the ratio of the biobased carbon to the total carbon in rubber or a rubber product, can be calculated in accordance with ISO 19984-1 when the chemical formulation and the information of each component are known. When no such information is available, it can be determined in accordance with ISO 19984-2.

8 Precision

See Annex E.

9 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 20463;

b) all the 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) the temperature, the atmospheric pressure, and the saturated water vapour pressure at the gas volume measurement;

d) test results: biobased combustion energy (gross calorific value), non-biobased combustion energy, expressed as J/g, of the sample;

e) test results: biobased combustion carbon dioxide emission, non-biobased combustion carbon dioxide emission, expressed as g/g, of the sample;

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

g) date of test.

Annex A
(informative)

Calculation of net calorific value for combustion energy

A.1 General

The combustion energy can be expressed as the gross calorific value (the higher heating value) and the net calorific value (the lower heating value).

When the moisture content is low like in most of rubber and rubber products, the gross calorific value and the net calorific value are nearly equal as indicated in Table A.1. The gross calorific value is basically sufficient to report, but when necessary, the net calorific value can be calculated after measuring the hydrogen content and the moisture content of the rubber or the rubber product.

A.2 Procedure

Measure the hydrogen content of the test sample by elemental analysis in accordance with ISO 17247 and ISO 29541. Measure the moisture content by the Karl Fischer method in accordance with ISO 12492.

The relation between the gross calorific value (E) and the net calorific value (E_I) is expressed as Formula (A.1):

$$E_I = E - 2\ 500 \times \left(9 \times x_H + x_w\right)/100 \tag{A.1}$$

where

E_I is the net calorific value (J/g);

E is the gross calorific value (J/g);

x_H is the hydrogen content of test sample (%);

x_w is the moisture content of test sample (%);

2 500   is the latent heat of vaporization for water (J/g).

Table A.1 — Gross calorific value and net calorific value of samples related to rubber products

Sample	Calorific value J/g		Hydrogen content[c] mass%	Moisture content[d] mass%	N/G[e]
	Gross calorific value[a]	Net calorific value[b]			
Rubber compound 1[f]	40 100	38 100	8,8	0,62	0,95
Rubber compound 9[g]	39 600	37 700	8,3	0,68	0,95
ASTM oil No.2	45 000	42 000	13,4	0,05	0,93
Castor oil	37 400	34 700	11,9	0,04	0,93
Natural rubber	43 500	40 700	12,1	0,40	0,94
S-SBR[h]	44 200	41 700	11,1	0,01	0,94
Carbon black (N330)	33 700	33 600	less than 0,1	0,40	1,00
Coal[i]	30 000	28 700	5,4	4,40	0,96

[a] Gross calorific value of sample measured by calorimeter (see Clause 6).

[b] Net calorific value calculated from gross calorific value, hydrogen content, and moisture content of sample based on Formula (A.1).

[c] Hydrogen content measured by elemental analysis of sample.

[d] Moisture content measured by moisture meter for sample.

[e] N/G is net calorific value divided by gross calorific value.

[f] This sample includes NR/BR = 60/40, and 35 phr of carbon black same as sample Number 1 indicated in Tables B.1 and D.1.

[g] This sample includes NR = 100, and 30 phr of carbon black (rapeseed oil) same as sample Number 9 indicated in Tables B.1 and D.1.

[h] Solution polymerized styrene-butadiene rubber.

[i] This coal was mined in Japan.

Annex B
(informative)

Examples of determination of combustion energy of biobased and non-biobased materials

This annex provides the results of biobased and non-biobased combustion energy determinations operated with adiabatic bomb calorimeter on natural rubber and rubber products. The procedure was carried out in accordance with this document.

The results are shown in Table B.1.

Table B.1 — Measured biobased carbon contents, combustion energy (total, biobased and non-biobased) for rubber compounds and polyurethane

Number	Outline of compound recipe				Biobased carbon content[a] %	Combustion energy (gross calorific value) J/g		
	Rubber	Charac-teristic ingredient	Carbon black (CB) phr	Silica phr		Total[b]	Biobased[c]	Non-biobased[d]
1	NR/BR = 60/40	None	35	0	39,2	40 100	15 700	24 400
2	NR = 100	None	45	0	60,4	39 400	23 800	15 600
3	NR/S-SBR = 70/30	None	40	15	42,0	37 000	15 500	21 500
4	NR/S-SBR = 20/80	None	10	45	13,7	33 300	4 600	28 700
5	NR/BIIR = 30/70	None	45	0	21,2	40 700	8 600	31 100
6	ENR = 100	None	5	60	80,9	27 900	22 600	5 300
7	S-SBR = 100	Phenolic resin	10	70	2,0	30 700	600	30 100
8	NR/S-SBR = 80/20	Resin partly from biomass	50	0	44,5	40 000	17 800	22 200
9	NR = 100	CB from rapeseed oil	30	0	71,1	39 600	28 200	11 400
10	NR/S-SBR = 15/85	All silica	0	40	10,6	32 500	3 400	29 100
11	Polyurethane	Biobased polyol	—	—	67,8	32 900	22 300	10 600

NR: Natural rubber, BR: Butadiene rubber, S-SBR: Solution polymerized styrene-butadiene rubber,

BIIR: Bromobutyl rubber, ENR: Epoxidized NR.

[a] The biobased carbon content was measured in accordance with ISO 19984-2.

[b] The total combustion energy was measured in accordance with Clause 6 of this document.

[c] The biobased combustion energy was calculated using Formula (1) of this document.

[d] The non-biobased combustion energy was calculated by subtracting the biobased combustion energy from the total combustion energy.

Annex C
(normative)

Saturated water vapour pressure

Table C.1 gives the saturated water vapour pressures at different temperatures[6].

Table C.1 — Saturated water vapour pressure

Temperature °C	Saturated water vapour pressure (P_t) kPa	Temperature °C	Saturated water vapour pressure (P_t) kPa
15	1,71	25	3,17
16	1,82	26	3,36
17	1,94	27	3,57
18	2,06	28	3,78
19	2,20	29	4,01
20	2,34	30	4,25
21	2,49	31	4,50
22	2,64	32	4,76
23	2,81	33	5,03
24	2,99	34	5,32