INTERNATIONAL STANDARD

ISO 16620-4

First edition
2016-12-15

Plastics — Biobased content —

Part 4: Determination of biobased mass content

Plastiques — Teneur biosourcée —

Partie 4: Détermination de la teneur en masse biosourcée

Reference number
ISO 16620-4:2016(E)

COPYRIGHT PROTECTED DOCUMENT

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

Contents

Page

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).

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 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.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

A list of all parts in the ISO 16620 series can be found on the ISO website.

Introduction

Increased use of biomass resources for manufacturing plastics products is effective in reducing global warming and the depletion of fossil resources.

Current plastics products are composed of biobased synthetic polymers, fossil-based synthetic polymers, natural polymers and additives that can include biobased materials.

“Biobased plastics” refer to plastics that contain materials wholly or partly of biogenic origin.

In ISO 16620 series, the “biobased content” of biobased plastics refers to the amount of the biobased carbon content, the amount of the biobased synthetic polymer content or the amount of the biobased mass content only.

This document is harmonized with EN 16785-1:2015[12].

Plastics — Biobased content —

Part 4: Determination of biobased mass content

1 Scope

This document specifies a method of determining the biobased mass content in plastics products, based on the radiocarbon analysis and elemental analysis.

This document is applicable to plastic products and plastic materials, polymer resins, monomers or additives, which are made from biobased or fossil-based constituents.

This method is applicable, provided that the plastic product contains carbon element and that a statement giving its elemental composition and its biobased mass content is available.

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 16620-2:2015, *Plastics — Biobased content — Part 2: Determination of biobased carbon content*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16620-1 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/

3.2 Symbols

^{14}C	carbon isotope with an atomic mass of 14
C	symbol for element carbon
H	symbol for element hydrogen
N	symbol for element nitrogen
O	symbol for element oxygen
m_B	biobased mass content, expressed as a percentage of the total mass of sample
x_B	biobased carbon content, expressed as a percentage of the total mass of the sample

x^{TC} total carbon content, expressed as a percentage of the total mass of the sample

x^{TH} total hydrogen content, expressed as a percentage of the total mass of the sample

x^{TN} total nitrogen content, expressed as a percentage of the total mass of the sample

x^{TO} total oxygen content, expressed as a percentage of the total mass of the sample

W mass of a sample, expressed in grams

3.3 Abbreviated terms

CL confidence level

TC total carbon

TH total hydrogen

TN total nitrogen

TO total oxygen

4 Principle

4.1 Product groups

For the purpose of this document, two groups of products are distinguished as follows:

a) Group I products are obtained by chemical or biological reaction(s);

b) Group II products are obtained by mixing Group I products without chemical or biological reaction.

Natural constituents (4.4) can be used to produce Group I products or as constituent(s) of Group II products.

4.2 Group I products

This method, supported by rules described in Clause 6, consists in

a) the determination of the biobased carbon content and elemental composition of the product by using the radiocarbon analysis and elemental analysis respectively (6.3), and

b) the comparison between

   1) the data of the statement (6.1) comprising the composition and the origin (biobased and/or fossil resources) of the product, and

   2) the data resulting from the radiocarbon analysis and elemental analysis of the product (6.3).

NOTE The “statement” in the sense of this document is to be distinguished from the “declaration” of the biobased mass content resulting of this method, which is in the scope of ISO 16620-5[1)].

4.3 Group II products

This method consists in

a) the determination of the biobased carbon content of the product by using the radiocarbon analysis (7.3), and

1) To be published.

b) the comparison between

1) the data of the statement (7.1) comprising the composition and the origin (biobased and/or fossil resources) of the product, and

2) the data resulting from the radiocarbon analysis of the product (7.3).

NOTE The "statement" in the sense of this document is to be distinguished from the "declaration" of the biobased mass content resulting of this method, which is in the scope of ISO 16620-5[1)].

4.4 Natural constituents

This method is not needed for the determination of the biobased mass content in natural constituents (e.g. natural polymers) wholly derived from biomass.

The biobased mass content of a natural constituent is equal to 100 %.

The biobased carbon content of a natural constituent, expressed as a percentage of the total carbon content, is equal to 100 %.

NOTE This differentiates the calculation of the biobased mass content according to this document from the calculation of the biobased synthetic polymer content according to ISO 16620-3, where the biobased synthetic polymer content of a natural constituent is 0 % (see ISO 16620-1:2015, Clause 4).

4.5 Monomers and additives

In case of monomers and additives for which the composition and raw material(s)/chemical(s) from which they are made are known, and whose chemical identification is unequivocal, the method may consist only in a determination of the biobased carbon content. The biobased mass content may be validated by applying the validation criteria (6.4), considering only the biobased carbon content.

5 Rules for allocation of elements

NOTE According to the current state of the art, it is not possible by isotopic measurements to establish a distinction between elements originating from biomass and elements originating from non-biomass for elements such as oxygen, hydrogen or nitrogen.

For a product/constituent of a product obtained by chemical or biological reaction(s) (Group I), the following rules shall apply:

a) if the reactants are exclusively derived from biomass, the biobased mass content of the product/constituent of the product is 100 %;

b) if none of the reactants is derived from biomass, the biobased mass content of the product/constituent of the product is 0 %;

c) if the reactants are derived from both biomass and non-biomass, the following conventions apply:

1) if oxygen (O) and/or hydrogen (H) and/or nitrogen (N) element(s) is(are) bound to a carbon structure derived from biomass, its(their) fraction is(are) considered to be part(s) of the biobased mass content;

2) element(s) other than C, H, O and N elements are not considered in this document.

EXAMPLE Esters derived from the condensation of an acid with a primary alcohol keep the O element coming from the alcohol.

6 Group I products

6.1 Statement

The statement to be provided with the product under consideration shall include

a) information related to the relevant chemical or biological reaction(s) and the raw materials/chemicals from which the product is made,

b) a complete elemental composition of the product (x_1^{TC}, x_1^{TH}, x_1^{TO} and x_1^{TN}), and

c) the biobased carbon content (x_{B1}) and biobased mass content (m_{B1}), of the product, obtained by calculation, following the rules defined in Clause 5.

NOTE 1 The biobased carbon content can be expressed on the basis of total organic carbon (TOC) or total carbon (TC) or total mass.

NOTE 2 In this document, "biobased carbon content" refers to "biobased carbon content by mass, expressed as a percentage of the total mass".

For products which contain water, the biobased mass content (m_{B1}) is expressed by mass of dry matter.

EXAMPLE Poly(ethylene terephthalate) obtained by polycondensation of terephthalic acid from fossil resources with biobased ethylene glycol (see Table 1).

Table 1 — Example of calculation for poly(ethylene terephthalate)

Fraction	C %	H %	O %	Total %
Fossil fraction (from terephthalic acid)	50,0	2,1	16,6	68,7
Biobased fraction (from ethylene glycol)	12,5	2,1	16,6	31,2
Total	62,5	4,2	33,3	100,0
x_1^{TC} = 62,5 % x_1^{TH} = 4,2 % x_1^{TO} = 33,3 %		x_{B1} = 12,5 % m_{B1} = 31,2 %		

6.2 Sampling

The samples shall be representative of the product under consideration.

If available, product sampling procedures for the determination of the biobased carbon content and elemental composition shall be used and the details shall be documented.

6.3 Determination of the biobased carbon content and elemental composition

6.3.1 Procedure

Determine the biobased carbon content of the sample according to ISO 16620-2.

Express the biobased carbon content (x_{B2}) as a percentage of the total mass of the sample.

Determine the contents of total carbon (x_2^{TC}), hydrogen (x_2^{TH}), oxygen (x_2^{TO}) and/or nitrogen (x_2^{TN}) of the sample according to suitable standard analytical methods. If other element(s) is(are) present, its(their) content(s) may be also determined.

For determining the total carbon content and organic carbon content, test methods as described in ISO 609, ISO 8245, ISO 10694, ISO 15350, ISO 17247, ASTM D5291-02, ASTM E1019-11 or EN 13137 may be used, as applicable.

The oxygen content shall be obtained by analysis and not by calculation [i.e. by subtraction of the C, H and N contents from the total content (100 %)].

Express the contents of total carbon (x_2^{TC}), hydrogen (x_2^{TH}), oxygen (x_2^{TO}) and/or nitrogen (x_2^{TN}) as percentages of the total mass of the sample.

For the validation (6.4), use the test results, expressed by mass of dry matter.

6.3.2 Variability of test results

The results obtained by the analytical methods can differ from the stated values for the following reasons:

a) the composition of the product can present variability due to its natural origin;

   EXAMPLE Natural fatty acids used in the production of fatty acid esters.

b) the production process can be, to a certain extent, a cause of variability of the composition of the final product;

c) the analytical methods are also a source of uncertainty, as follows:

   1) ±3 % of the measured value for the biobased carbon content;

   2) ±0,4 % of the measured value for the total carbon, total oxygen or total nitrogen content;

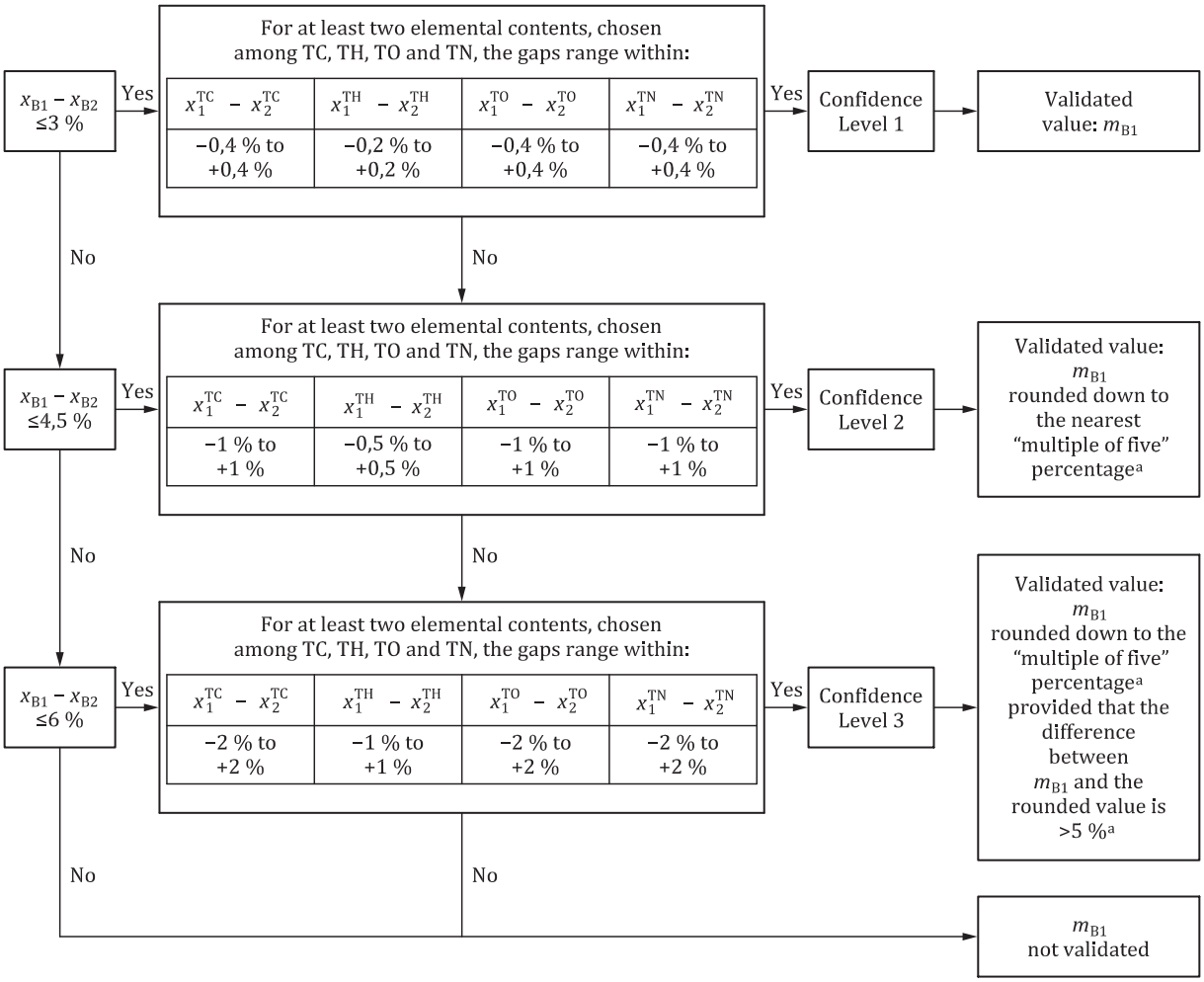
   3) ±0,2 % of the measured value for the total hydrogen content.

6.4 Validation criteria of the biobased mass content

Determine the gaps between the values given in the statement (6.1) and values resulting from testing (6.3.1) for the biobased carbon content, total carbon content, total hydrogen content, total oxygen content and/or total nitrogen content, as relevant.

For the validation of the biobased mass content, apply the decision scheme according to Figure 1.

For monomers and additives, see also 4.5.



a Percentage related to the total mass of the sample.

Figure 1 — Decision scheme for Group I products

EXAMPLE 1 For confidence level 2, 52 % is rounded down to 50 % and 57 % is rounded down to 55 %.

EXAMPLE 2 For confidence level 3, 48 % is rounded down to 40 % and 42 % is rounded down to 35 %.

If nitrogen and/or oxygen element(s) is(are) not present in the molecule, it(they) is(are) not taken into account.

If the stated biobased carbon content is lower than the biobased carbon content obtained by testing, then the gap is to be considered as fulfilling the criteria for confidence level 1.

EXAMPLE 3 Stated biobased carbon content: 53,2 %, biobased carbon content obtained by analysis: 56,5 %, gap between the two values: 3,3 %. As the stated value is lower than the value obtained by analysis, the confidence level to be applied is 1 for this value.

EXAMPLE 4 In case of poly(ethylene terephthalate) (see 6.1, EXAMPLE), N is not present in the molecule; therefore N cannot be taken into account. The validation is done by comparing the values of the biobased carbon content, as well as the TC content and the TH content. The TO content may also be considered instead of the TC content or TH content.

6.5 Examples of application of the decision scheme

Examples are given in Tables 2, 3 and 4.

Table 2 — Example 1

	Biobased carbon content %	Total carbon %	Total hydrogen %	Total oxygen %	Biobased mass content %
Stated values	25,0	50,0	5,6	44,4	56
Measured values	25,9	49,8	5,7	44,6	
Gap between the stated and measured values	$x_{B1} - x_{B2}$	$x_1^{TC} - x_2^{TC}$	$x_1^{TH} - x_2^{TH}$	$x_1^{TO} - x_2^{TO}$	
	−0,9	0,2	−0,1	−0,2	
Criteria for CL 1	≤3	±0,4	±0,2	±0,4	
Criteria satisfied	Yes	Yes	Yes	Yes	
Validated value for the biobased mass content: 56 %.					

Table 3 — Example 2

	Biobased carbon content %	Total carbon %	Total hydrogen %	Total oxygen %	Biobased mass content %
Stated values	40,8	53,0	9,0	24,2	52
Measured values	40,1	52,1	8,8	22,5	
Gap between the stated and measured values	$x_{B1} - x_{B2}$	$x_1^{TC} - x_2^{TC}$	$x_1^{TH} - x_2^{TH}$	$x_1^{TO} - x_2^{TO}$	
	+0,7	+0,9	+0,2	+1,7	
Criteria for CL 2	≤4,5	±1,0	±0,5	±1,0	
Criteria satisfied	Yes	Yes	Yes	No	
Validated value for the biobased mass content: 50 %.					

Table 4 — Example 3

	Biobased carbon content %	Total carbon %	Total hydrogen %	Total oxygen %	Biobased mass content %
Stated values	47,2	50,2	7,2	42,6	67
Measured values	42,2 ± 6	47,5 ± 2	7,9	40,8	
Gap between the stated and measured values	$x_{B1} - x_{B2}$	$x_1^{TC} - x_2^{TC}$	$x_1^{TH} - x_2^{TH}$	$x_1^{TO} - x_2^{TO}$	-
	+5	+2,7	−0,7	+1,8	
Criteria for CL 3	≤6	±2	±1	±2	
Criteria satisfied	Yes	No	Yes	Yes	
Validated value for the biobased mass content: 60 %.					

6.6 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 16620-4;

b) all information necessary for complete identification of the product under consideration;

c) the product group, i.e. Group I;

d) the statement, as defined in 6.1 (m_{B1}, x_{B1}, x_1^{TC}, x_1^{TH}, x_1^{TO}, x_1^{TN}), expressed as a percentage of total mass of the sample;

e) method used for the determination of the biobased carbon content, e.g. ISO 16620-2:2015, Method C;

f) the value resulting from testing of the biobased carbon content (x_{B2}), expressed as a percentage of the total mass of the sample;

g) analytical methods used for the determination of the elemental composition;

h) the measured values for each present element C, H, O and/or N (x_2^{TC}, x_2^{TH}, x_2^{TO}, x_2^{TN}), expressed as a percentage, of the total mass of the sample;

i) the validated value for the biobased mass content (m_B), expressed as a percentage of total mass of the dried sample matter;

j) 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 test results;

k) the identification of the testing laboratory performing the test;

l) the date of the test.

Annex A gives an example of a format for reporting the results for Group I products.

7 Group II products

7.1 Statement

This method is applicable, provided that each of the constituents of the product, except the natural constituent(s) (4.4), if any, have been first analysed according to Clause 6, and the statement giving the biobased mass content of each constituent has been validated.

The statement to be provided with the product under consideration shall include:

a) information related to the production process and the raw materials/chemicals from which the product is made,

b) the validated biobased carbon content ($x_{B,i}$) based on a test report according to ISO 16620-2 and the biobased mass content ($m_{B,i}$) of each of the biobased constituents of the product based on a test report according to 6.6, except for natural constituent(s);

c) the biobased carbon content (x_{B1}) and the biobased mass content (m_{B1}) of the product, obtained by calculation according to Annex B.

For products which contain water, the biobased mass content (m_{B1}) is expressed by mass of dry matter.

7.2 Sampling

The samples shall be representative of the product under consideration.

If available, product sampling procedures for the determination of the biobased carbon content shall be used and the details shall be documented.

7.3 Determination of the biobased carbon content

7.3.1 Procedure

Determine the biobased carbon content of the sample according to ISO 16620-2.

Express the biobased carbon content (x_{B2}) as a percentage of the total mass of the sample.

For the validation (7.4), use the test results expressed by mass of dry matter.

7.3.2 Variability of test results

The results obtained by the radiocarbon method can differ from the stated values for the following reasons:

a) the composition of the product can present variability due to its natural origin;

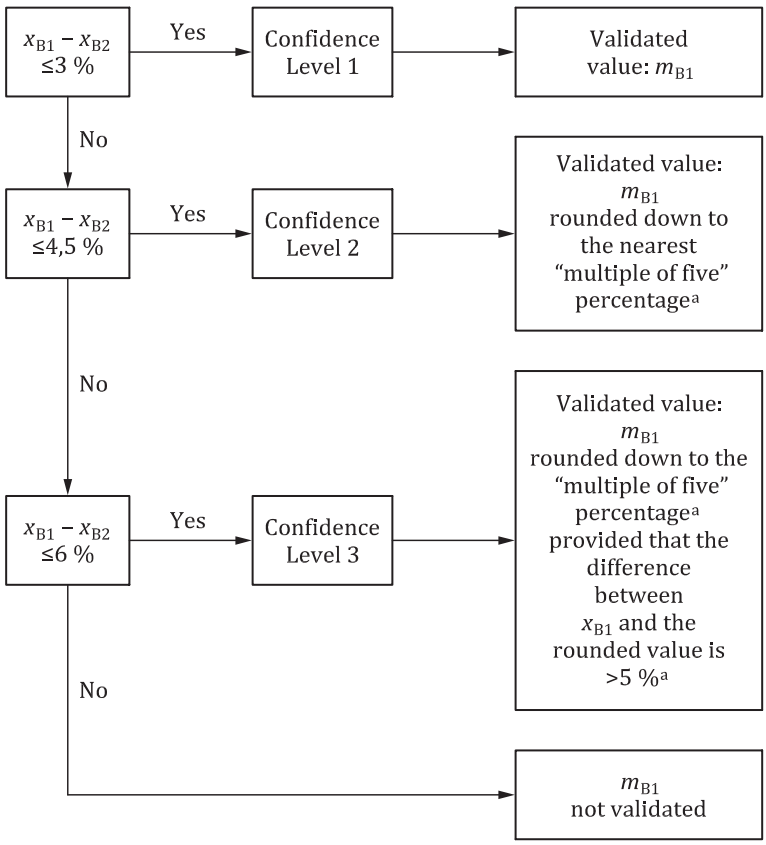
b) the production process can be, to a certain extent, a cause of variability of the composition of the final product;

c) the radiocarbon method is also a source of uncertainty: ±3 % of the measured value.

7.4 Validation criteria of the biobased mass content

Determine the gap between the value given in the statement (7.1) and value resulting from testing (7.3.1) for the biobased carbon content.

For the validation of the biobased mass content, apply the decision scheme according to Figure 2.



[a] Percentage related to the total mass of the sample.

Figure 2 — Decision scheme for Group II products

EXAMPLE 1 For confidence level 2, 52 % is rounded down to 50 % and 57 % is rounded down to 55 %.

EXAMPLE 2 For confidence level 3, 48 % is rounded down to 40 % and 42 % is rounded down to 35 %.

7.5 Example of application of the decision scheme

EXAMPLE Stated value for the biobased mass content: 53 %

Stated value for the biobased carbon content: 48 %

Measured value for the biobased carbon content: 44 %

Gap between stated value and measured value: 48 % − 44 % = 4 % (in confidence level 2)

Validated value for the biobased mass content: 50 %

7.6 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 16620-4;

b) all information necessary for complete identification of the product under consideration;

c) the product group, i.e. Group II;

d) the statement, as defined in 7.1;

e) the calculated value of the biobased carbon content (x_{B1}), expressed as a percentage of the total mass of the sample;

f) the calculated value of the biobased mass content (m_B), expressed as a percentage of the total mass of the sample;

g) method used for the determination of the biobased carbon content (e.g. ISO 16620-2:2015, Method C);

h) the value resulting from testing of the biobased carbon content (x_{B2}), expressed as a percentage of the total mass of the sample;

i) the validated value of the biobased mass content, expressed as a percentage of the total mass of the dried sample matter;

j) 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 test results;

k) the identification of the testing laboratory performing the test;

l) the date of the test.

Annex C gives an example of a format for reporting the results for Group II products.

Annex A
(informative)

Example of format for reporting results for Group I products

Table A.1 — Example of format for reporting results

Sample:	**%**	**CL**	**%**
Stated biobased mass content, m_B (provided by the producer or his representative)			
Stated value for biobased carbon content, x_{B1} (provided by the producer or his representative)			
Measured value for biobased carbon content, x_{B2}			
Gap between the stated and measured value, $x_{B1} - x_{B2}$			
Confidence level for biobased carbon (based on the gap: $x_{B1} - x_{B2}$)			
Stated value for total carbon content, x_1^{TC} (provided by the producer or his representative)			
Measured value for total carbon content, x_2^{TC}			
Gap between the stated and the measured total carbon content, $x_1^{TC} - x_2^{TC}$			
Confidence level for total carbon (based on the gap: $x_1^{TC} - x_2^{TC}$)			
Stated value for the total hydrogen content, x_1^{TH} (provided by the producer or his representative)			
Measured value for the total hydrogen content, x_2^{TH}			
Gap between the stated and measured total hydrogen content, $x_1^{TH} - x_2^{TH}$			
Confidence level for total hydrogen (based on the gap: $x_1^{TH} - x_2^{TH}$)			
Stated value for the total oxygen content, x_1^{TO} (provided by the producer or his representative)			
Measured value for the total oxygen content, x_2^{TO}			
Gap between the stated and measured total oxygen content, $x_1^{TO} - x_2^{TO}$			
Confidence level for total oxygen (based on the gap: $x_1^{TO} - x_2^{TO}$)			
Stated value for the total nitrogen content, x_1^{TN} (provided by the producer or his representative)			
Measured value for the total nitrogen content, x_2^{TN}			
Gap between the stated and measured total nitrogen content, $x_1^{TN} - x_2^{TN}$			
Confidence level for total nitrogen (based on the gap: $x_1^{TN} - x_2^{TN}$)			
Assigned final confidence level (use defined confidence levels for ^{14}C and two best confidence levels out of the remaining four elements: C, H, N, O)			
Validated value of the biobased mass content accordingly to assigned final confidence level [m_B or m_B rounded down depending on the confidence level (6.4)]			

Annex B
(normative)

Calculation of the biobased carbon content and biobased mass content for Group II products

B.1 Calculation of the biobased carbon content

Calculate the biobased carbon content, as a percentage of the total mass of the sample, using Formula (B.1):

$$x_B = \frac{\sum_{i=1}^{n} W_i \cdot x_{B,i}}{W} \quad \text{(B.1)}$$

where

x_B is the biobased carbon content, expressed as a percentage of the total mass of the sample;

$x_{B,i}$ is the biobased carbon content of the constituent (i), expressed as a percentage of the mass of the constituent (i);

W_i is the mass of the constituent (i), expressed in grams;

W is the total mass of the sample, expressed in grams;

n is the number of constituents of the sample.

B.2 Calculation of the biobased mass content

Calculate the biobased mass content using Formula (B.2):

$$m_B = \frac{\sum_{i=1}^{n} W_i \cdot m_{B,i}}{W} \quad \text{(B.2)}$$

where

m_B is the biobased mass content of the product expressed as a percentage of the total mass of sample;

$m_{B,i}$ is the biobased mass content of the constituent (i), expressed as a percentage of the mass of the constituent (i);

W_i is the mass of the constituent (i), expressed in grams;

W is the total mass of the sample, expressed in grams;

n is the number of constituents of the sample.

Annex C
(informative)

Example of format for reporting results for Group II products

Table C.1 — Example of format for reporting results

Sample:	**%**	**CL**	**%**
Stated biobased mass content, m_B (provided by the producer or his representative)			
Stated value for biobased carbon content, x_{B1} (provided by the producer or his representative)			
Measured value for the biobased carbon content, x_{B2}			
Gap between the stated and measured value, $x_{B1} - x_{B2}$			
Confidence level for biobased carbon (based on the gap: $x_{B1} - x_{B2}$)			
Validated value of the biobased mass content accordingly to assigned final confidence level [m_B or m_B rounded down depending on the confidence level (7.4)]			

Bibliography

[1] ISO 609, *Solid mineral fuels — Determination of carbon and hydrogen — High temperature combustion method*

[2] ISO 8245, *Water quality — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

[3] ISO 10694, *Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis)*

[4] ISO 15350, *Steel and iron — Determination of total carbon and sulfur content — Infrared absorption method after combustion in an induction furnace (routine method)*

[5] ISO 16620-1:2015, *Plastics — Biobased content — Part 1: General principles*

[6] ISO 16620-3, *Plastics — Biobased content — Part 3: Determination of biobased synthetic polymer content*

[7] ISO 16620-5[2)], *Plastics — Biobased content — Part 5: Declaration of the biobased carbon content, biobased synthetic polymer content and biobased mass content*

[8] ISO 17247, *Coal — Ultimate analysis*

[9] ASTM D5291-10, *Standard test methods for instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants*

[10] ASTM E1019-11, *Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques*

[11] EN 13137, *Characterization of waste — Determination of total organic carbon (TOC) in waste, sludges and sediments*

[12] EN 16785-1:2015, *Bio-based products — Bio-based content — Part 1: Determination of the bio-based content using the radiocarbon analysis and elemental analysis*

2) To be published.

ICS 83.080.01

Price based on 14 pages