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# Solid recovered fuels — Methods for the determination of biomass content

Combustibles solides de récupération – Méthode de détermination de

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#### ISO/FDIS 21644:2020(E)

## 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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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

For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 300, Solid recovered fuels.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Introduction

The biomass content of solid recovered fuels is relevant for the evaluation of the impact of energy production on greenhouse gas emission. Instrumental methods, wet chemical and manual procedures are available for the calculation of the renewable energy fraction. Instrumental methods are based on the determination of  $^{14}$ C content while manual procedures are based on separation of different fractions by visual inspection. The wet chemical procedure differentiate biomass from non-biomass materials as function of the acid dissolution behaviour.

The fraction of biomass is expressed:

- by mass;
- by energy content (gross or net calorific value);
- by carbon content.

This document is primarily intended for laboratories, producers, suppliers and purchasers of solid recovered fuels, but is also useful for the authorities and inspection organizations.

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# Solid recovered fuels — Methods for the determination of biomass content

#### 1 Scope

This document specifies three methods for the determination of the biomass content in solid recovered fuels: the <sup>14</sup>C content method, the selective dissolution and the manual sorting methods.

#### 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 21637:—<sup>1</sup>), Solid recovered fuels — Terminology, definitions and descriptions

ISO 21645:—<sup>2)</sup>, Solid recovered fuels — Methods for sampling

ISO 21654:—<sup>3)</sup>, Solid recovered fuels — Determination of calorific value

ISO 21656: $-^{4)}$ , Solid recovered fuels — Determination of ash content

ISO 21663:—<sup>5)</sup>, Solid recovered fuels — Methods for the determination of total carbon (C), hydrogen (H), nitrogen (N) and sulphur (S) by the instrumental method

EN 15443, Solid recovered fuels. Methods for the preparation of the laboratory sample

CEN/TS 15414-1, Solid recovered fuels — Determination of moisture content using the oven dry method — Part 1: Determination of total moisture by a reference method

#### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637:— and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <u>https://www.iso.org/obp</u>

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

#### 3.1

#### ash content on dry basis

mass of inorganic residue remaining after ignition of a fuel under specified conditions, expressed as mass fraction in percent of the dry matter in the fuel, also includes removed ash contributors

Note 1 to entry: This is typically expressed as a percentage of the mass of dry matter in the fuel source.

<sup>1)</sup> Under preparation. Stage at the time of publication ISO/FDIS 21637.

<sup>2)</sup> Under preparation. Stage at the time of publication ISO/DIS 21645.

<sup>3)</sup> Under preparation. Stage at the time of publication ISO/DIS 21654.

<sup>4)</sup> Under preparation. Stage at the time of publication ISO/DIS 21656.

<sup>5)</sup> Under preparation. Stage at the time of publication ISO/DIS 21663.

Note 2 to entry: Depending on the combustion efficiency the ash may contain combustibles.

Note 3 to entry: If a complete combustion is realized, ash contains only inorganic, non-combustible components.

#### [SOURCE: ISO 21637:-, 3.3]

#### 3.2

#### biogenic

produced in natural processes by living organisms but not fossilized or derived from fossil resources

#### 3.3

#### biomass

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

[SOURCE: ISO 16559:2014, 4.32, modified — Notes 1 and 2 to entry have been removed.]

#### 3.4

#### calorific value

quantity of heat produced by the complete combustion, at a constant pressure equal to 1 013,25 mbar, of a unit volume or mass of gas, the constituents of the combustible mixture being taken at reference conditions and the products of combustion being brought back to the same conditions

[SOURCE: EN 437: 2018, modified — Second paragraph (the list) has been removed.]

#### 3.5

#### gross calorific value

calorific value where the water produced by combustion is assumed to be condensed Bstandards

[SOURCE: ISO 21637:-, 3.34]

#### 3.6

#### isotope abundance

fraction of atoms of a particular isotope of an element Jardsitell.

#### 3.7

#### laboratory sample

1-10923 part of the sample (3.13) sent to or received by the laboratory

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection, but it is the initial sample from the point of view of the laboratory.

Note 3 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

#### 3.8

#### moisture

water removable under specific conditions

[SOURCE: ISO 21637:-, 3.46]

#### 3.9

#### net calorific value at constant volume

calorific value where the water produced by combustion is assumed to be in the vapour state

[SOURCE: ISO 21637:-, 3.47]

#### 3.10

#### nominal minimum particle size

aperture size of the sieve used for determining the particle size distribution of solid recovered fuels through which no more than 5 % by mass of the material passes

#### 3.11

#### nominal top size

smallest aperture size of the sieve used for determining the particle size distribution of solid recovered fuels through which at least 95 % by mass of the total material passes through the sieve

[SOURCE: ISO 21637:-, 3.48]

#### 3.12 percentage modern Carbon

pmC

carbon mass fraction from biogenic origin

Note 1 to entry: The internationally accepted radiocarbon dating reference value is 95 percent of the activity, in AD 1950, of this NBS oxalic acid SRM4990B.

Note 2 to entry: In 2015, the value of 100 % biogenic carbon was set at 102 pmC.

Note 3 to entry: The biogenic origin is expressed in percentage.

#### 3.13

sample

quantity of material, from a larger amount for which the quality is to be determined

[SOURCE: ISO 21637:—, 3.63, modified  $\bigcirc$  Notes 1–3 to entry have been removed.] pelstar

3.14 sample preparation actions taken to obtain representative laboratory samples (3.7) or test portions from the original sample (<u>3.13</u>) as received

[SOURCE: ISO 21637:-, 3.66]

#### Symbols and abbreviations 4

For the purposes of this document, the following symbols and abbreviations apply.

С	symbol for element carbon
D	diameter (mm)
<sup>14</sup> C	carbon isotope with an atomic mass of 14 u
LSC	Liquid Scintillation Counter or Liquid Scintillation Counting
Msort	manual sorting method
RSD	relative standard deviation
SDM	selective dissolution method
SRF	solid recovered fuel
ТС	total carbon content
u	atomic mass unit

- mass fraction expressed as a percentage by mass W
- content expressed as a percentage of the energy content  $W_{cal}$
- content expressed as a percentage of the total carbon content  $W_{\rm TC}$

The different references used in this document are indicated by the following indices:

- (ad) for air dried (dried at room temperature 20-25 °C for 24 hours)
- (ar) for as received
- (d) for dry
- (daf) for dry and ash free, where appropriate.

 $w_{cal,NB(d)}$  means the fraction of energy content in the non-biomass fraction by calorific value, on **EXAMPLE** dry basis.

#### **Principle** 5

The determination of the biomass content is based on selective dissolution, manual sorting or <sup>14</sup>C measurement of biomass in solid recovered fuel. The choice for the method to be used is described Standards, tella in <u>Clause 6</u>. The biomass content gives an estimation of the content of the biogenic fraction in solid er. recovered fuel. Haren and and a south of the so

standard.

#### Determination of biomass content 6

#### 6.1 Sampling

Sampling, transport, storage of the solid recovered fuel and sample preparation in the field shall be conducted according to ISO 21645:— and EN 15443?

#### Sample preparation 6.2

Preparation of the test sample for the <sup>14</sup>C or SDM shall be conducted according to EN 15443. For the Msort, no sample preparation is performed.

Since SRF is considered as a heterogeneous material, the minimum sample amount to be used for each test shall be:

- $^{14}$ C method: a quantity between 0,4 and 2 g of the material with a nominal top size of 1 mm or less, depending on the device used for combustion (bomb, combustion tube furnace or elemental analyser) or the quantity indicated by the constructor in the case of the use of a laboratory scale combustion apparatus;
- selective dissolution method (SDM): at least 5 g of the material with a nominal top size of 1 mm or less;
- manual sorting method (Msort): at least as big as the minimum sample size according to ISO 21645:-(as received), as calculated in EN 15443.

#### 6.3 Applicable methods

For the determination of biomass content three methods are available:

1) the instrumental <sup>14</sup>C method shall be according to <u>Annex A</u>. This method is based on the determination of the ratio of <sup>14</sup>C to the total carbon content; the <sup>14</sup>C is proportional to the biomass content of the SRF. This method is suitable for samples of all types of fuel and shall be according to

<u>Annex A</u>. A value of 10 % biogenic carbon can be considered as the lower range of application of <sup>14</sup>C method by liquid scintillation counter (LSC);

- 2) the selective dissolution method (SDM) shall be according to <u>Annex B</u>. The determination of the biomass content by the SDM is based on the property of biomass that it can be dissolved in a sulphuric acid / hydrogen peroxide mixture. This method has limitations that makes it less suitable if the content of natural and/or synthetic rubber in the SRF is more than 10 %, or if the sum of the content of hard coal, coke, brown coal, lignite, degradable plastics of fossil origin, non-degradable plastic of biogenic origin, oil or fat present as a constituent of biomass, wool, viscose, nylon, polyurethane or other polymers containing molecular amino groups and silicon rubber exceeds 5 %. Additional information about these limitations is found in <u>Annex D</u>. The selective dissolution method (SDM) is applicable for the biomass percentage content between 10 % and 90 %;
- 3) the manual sorting method (Msort) shall be according to <u>Annex C</u>. The determination of the biomass content by the manual sorting method is based on the visual examination of fractions and their separation on the basis of their nature and origin. The method is suitable for samples with a particle size >10 mm.

For the limitations of the three methods see <u>Annex D</u>.

#### 7 Expression of results

Depending on the use of the results, three different dimensions are used to express the biomass content:

- a) biomass in percent by mass  $w_{\rm B}$ ;
- b) biomass in percent by calorific value  $W_{B,cal}$ ;
- c) biomass in percent by carbon content  $W_{B,TE}$

The expression of results by <sup>14</sup>C method shall be according to <u>Annex A</u>.

The expression of results by SDS method shall be according to <u>Annex B</u>.

The expression of results by Msort method shall be according to <u>Annex C</u>.

#### 8 Performance characteristics

External data for the calculation of the expanded uncertainty of measurements are presented in <u>Annex E</u> where results of round robin and validation studies are summarized. These values should be used in combination with individual laboratory performance characteristics and a desired coverage factor to get the overall uncertainty.

Practical examples of use of the data from <u>Annex E</u>:

EXAMPLE 1

A laboratory wants to determine the expanded uncertainty of measurement of SDM method (% by mass).

The intra-laboratory reproducibility for the laboratory calculated from internal validations studies and control charts was determined to be 2,5 % (RSD).

The round robin results from the QUOVADIS<sup>[3]</sup> study (<u>Table E.2</u>) give a RSD value of 3,43 % (at 67,79 % level).

u c,rel =  $\sqrt{(2,5^2+3,43^2)}$  = 4,24 %

U rel = 2 × u c,rel = 8,48 %

where u c,rel is the combined uncertainty of measurement and U rel is the expanded uncertainty of measurement using a coverage factor of 2 ( $\sim$ 95 % confidence interval).

#### **EXAMPLE 2**

A laboratory measures the biomass content by <sup>14</sup>C method – LSC B (% by TC).

The intra-laboratory reproducibility for the laboratory calculated from internal validations studies and control charts was determined to be 2,4 % (RSD).

The round robin results from the OUOVADIS<sup>[3]</sup> study (Table E.6) give a RSD value of 2.5 % (at 55.5 % level).

u c,rel =  $\sqrt{(2,4^2+2,5^2)} = 3,5\%$ 

U rel = 2 × u c,rel = 7,0 %

where u c,rel is the combined uncertainty of measurement and U rel is the expanded uncertainty of measurement using a coverage factor of 2 (~ 95 % confidence interval).

#### 9 **Test report**

The test report shall contain at least the following information:

- identification of the laboratory performing the test; a)
- b) date of the test;
- identification of product (sample) tested; c)
- sample preparation (e.g. method of size reduction, drying, subdivision); d) ndard standari
- storage conditions; e)
- date of receipt of laboratory sample and dates of the test (beginning and end); f)
- a reference to this document (ISO 21644:—) and the method used; g)
- in case of <sup>14</sup>C-method, the results of the test including the basis on which they are expressed and h) application of the isotope correction; 💉
- the biomass content expressed as a percentage by mass, calorific value and/or carbon content, i) rounded to the nearest 0,1 %;
- any operation not included in this document, or regarded as optional; j)
- any unusual features noted during the test procedure. k)

#### Annex A (normative)

## Determination of the biomass content based on the <sup>14</sup>C method

#### A.1 General

The two proposed methods for <sup>14</sup>C measurement, Proportional Scintillation Method (PSM) or Accelerated Mass Spectrometry (AMS), require specialised personnel and instrumentation. However, the preparation step for instrumental analysis can be completed as normal routine laboratory activity. For the collection from the sample of the <sup>14</sup>C fraction, generally accepted methods for the conversion of the carbon present in the sample to  $CO_2$  are described.

#### A.2 Principle

The methods for the determination of the biomass content specified in this annex are based on the determination of the <sup>14</sup>C content. The amount of biomass carbon in solid recovered fuel is proportional to this <sup>14</sup>C content.

The carbon present in the sample is converted to  $\rm CO_2$  by combustion. The combustion is carried out in a way to comply with the requirements of the subsequent measurement of the <sup>14</sup>C content. This measurement is carried out according to one of the two following methods, Proportional Scintillation Method (PSM) or Accelerated Mass Spectrometry (AMS). These methods are considered equivalent, giving the same results within the scope of this document. The results are expressed as the percentage biomass carbon of the total carbon content. The fraction of biomass content by mass and the fraction of biomass by energy content are calculated from the carbon content of biomass, using the carbon content of biomass and the energy content of the biomass fraction that is present in the sample.

#### A.3 Limitations

For the limitation of this method see <u>Annex D</u>.

#### A.4 Symbols

For the purposes of this annex, the following symbols apply.

С	symbol for element carbon
140	aarban jaatana with an atomia ma

- <sup>14</sup>C carbon isotope with an atomic mass of 14
- AMS Accelerator Mass Spectrometry
- β beta particle, electron emitted during radioactive decay
- Bq Bequerel, disintegrations per second
- d on dry base
- DPM disintegrations per minute
- CPM counts per minute