
**Solid biofuels — Determination
of water sorption and its effect
on durability of thermally treated
biomass fuels —**

**Part 1:
Pellets**

*Biocombustibles solides — Détermination de la sorption d'eau et de
son influence sur la durabilité des combustibles de biomasse traités
thermiquement —*

Partie 1: Granulés

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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).

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 238, *Solid biofuels*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 335, *Solid biofuels*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

Thermally treated biomass fuels, particularly in compressed form, are increasingly considered as a replacement of fossil coal or for co-firing in large energy plants for production of heat and/or power. Compressed biomass fuels which are not thermally treated easily absorb moisture; this compromises the durability and generates fines. Thermally treated biomass fuels vary in their affinity to absorb moisture (absorption and/or adsorption – here collectively called sorption) depending on the extent and/or type of thermal treatment, feedstock used to make the product, compression, potential additives used, etc. For this purpose, it is important to understand the degree to which thermally treated compressed solid biofuels are resistant to moisture uptake and the degree to which they maintain durability when exposed to moisture, primarily in the form of rain during outdoor storage.

Thermally treated biomass fuel such as pellets or briquettes may be classified based on these characteristics as suitable or unsuitable to be handled and stored under conditions with limited or no weather protection. This document was developed specifically for the classification of pellets and is not intended to be applicable to other forms of densified fuel (e.g. briquettes). It is intended that other parts will be developed as necessary to apply these principles to other forms of thermally treated biomass fuels.

It should be noted that in large-scale storage of thermally treated biomass fuels the degree of wetting will likely vary within the storage. Therefore, this document is not intended to be used to draw conclusions on the average degree of wetting for any particular storage, but rather provides an indication of the degree to which durability and/or moisture content can be affected under worst case conditions. This method can be used for comparative purposes towards other pelletized thermally treated biomass fuels.

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Solid biofuels — Determination of water sorption and its effect on durability of thermally treated biomass fuels —

Part 1: Pellets

1 Scope

This document specifies a method for the determination of water sorption in a laboratory setting and provides a measure for how the durability is impacted as a result of immersion in water. Post-immersion durability reduction is calculated as the difference between the durability of the as-received sample and the durability of the wetted product.

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 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 14780, *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 17831-1, *Solid biofuels — Determination of mechanical durability of pellets and briquettes — Part 1: Pellets*

ISO 18134-1, *Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method*

ISO 18134-2, *Solid biofuels — Determination of moisture content — Oven dry method — Part 2: Total moisture — Simplified method*

ISO 18135, *Solid Biofuels — Sampling*

ISO 18846, *Solid biofuels — Determination of fines content in quantities of pellets*

ISO 21945, *Solid biofuels — Simplified sampling method for small scale applications*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

**3.1
post-immersion**

p_i
basis of measurement whereby the material tested has been immersed in water prior to the test being conducted

Note 1 to entry: The post-immersion is done in accordance with ISO 23343 (all parts).

Note 2 to entry: Examples for the use of the basis p_i are moisture content post-immersion, M_{p_i} , and durability post-immersion, DU_{p_i} .

**3.2
post-immersion durability reduction**

DUR_{p_i}
measure of the drop in the durability of thermally treated biomass fuel pellets after immersion in water using this test procedure

Note 1 to entry: A post-immersion durability reduction value of 0 means there was no change to the durability of the thermally treated pelletized fuel as a result of wetting, whereas for example, a post-immersion durability reduction value of 3 indicate that the durability dropped by three percentage points as a result of wetting.

**3.3
water sorption**

S_w
gain of water/moisture by solid biofuels through absorption and/or adsorption when exposed to water or varying levels of humidity

4 Principle

For this test method, the as-received sample is initially tested for moisture content and durability. Fines are then removed from two separate sub-samples by screening, and then each sub-sample is immersed in water for 24 h. After immersion the excess water is drained, and the wetted material is again tested for moisture content and an air-dried portion is tested for durability. Water sorption and post-immersion durability reduction are then calculated.

5 Apparatus

5.1 Sieve.

The sieve shall have round holes with a diameter of 3,15 mm and aperture geometry in accordance with ISO 3310-2.

A sieve with a diameter of 400 mm is recommended. The frame of the sieve shall have a height that enables the sieve to contain a sample and allow a free movement of the sample during the sieving process. Other sizes of sieves can be used for practical reasons, but it is important to make sure the sieve is not overloaded which can result in insufficient agitation of the test sample which can impact the flow of fines through the apertures of the sieve.

5.2 Collecting pan.

For the collection of material passing through the sieve.

5.3 Water bath.

The water bath can be any container large enough to hold a single immersion container (in which case multiple water baths will be required) or to hold multiple immersion containers within it and a high enough water level to fully submerge the sub-sample(s).

5.4 Immersion container(s) and immersion container cover(s).

For the purposes of this test method, two 1,0 mm wire mesh sieves of 400 mm diameter are recommended to be used as immersion containers. Other sieve diameters can be used or a specially made wire mesh basket(s). If an alternate immersion container(s) is used the screen size shall be 1,0 mm wire mesh and the alternate container shall provide the same level of loading as is achieved when using the 400 mm diameter sieve (e.g. a 300 mm diameter sieve has a sieve area that is 44 % smaller than the sieve area of a 400 mm diameter sieve therefore the sample amount placed on a 300 mm diameter sieve should also be 44 % less). The volume of the immersion container(s) shall be large enough to ensure not to compress the material after expansion during the test.

Additionally, material that floats because of air bubbles or due to having a density $<1 \text{ g/cm}^3$ are to be held under the water level by using a second sieve as a cover over the immersion container. If an alternate immersion container is used, then it shall also include a suitable cover that holds the material under the water level. While a sieve of 1,0 mm aperture is preferred for use as the immersion container cover, sieves not exceeding 3,15 mm round hole may be used if no visible material is escaping from the top sieve during the test. Sieve apertures below 1,0 mm shall not be used as they can trap air under the sieve.

5.5 Flat drying trays.

Flat large bottom tray(s) for spreading out the wetted material to air-dry and to subsequently equilibrate the material in the lab atmosphere. The tray(s) shall have a surface loading not to exceed 1 g of material per cm^2 of surface area.

5.6 Drying oven.

The drying oven can be a convection or forced air oven, allowing to adjust the temperature to a constant level of not higher than 40 °C and the air velocity in such a way that the sub-sample particles are not dislodged from the tray.

5.7 Balance.

The balance shall be capable of reading to the nearest 0,1 g.

6 Sampling and test sample preparation

A laboratory sample with a minimum mass of 5,0 kg shall be obtained in accordance with ISO 18135 or ISO 21945. If additional sample preparation is necessary to establish the test sample, then it shall be done in accordance with ISO 14780. The test sample shall be divided into four equal sub-samples also in accordance with ISO 14780. If a large amount of fines is observed within the test sample, then the size of the test sample shall be increased to assure that at least 1,0 kg is obtained when the sub-samples are sieved to remove fines.

7 Procedure

7.1 Testing the as-received moisture content and durability

One sub-sample shall be used for the determination of total moisture in accordance with ISO 18134-1 or ISO 18134-2. A second sub-sample shall be used for the determination of durability in accordance with ISO 17831-1.

7.2 Sieving procedure

Remove the fines from the two remaining sub-samples in accordance with ISO 18846 utilizing the sieve and collection pan specified in 5.1 and 5.2 respectively. The separated fines shall not be used as part of this test method and can be discarded.

7.3 Wetting of the sub-samples

Fill the water bath(s) specified in 5.3 with purified water (e.g. deionized and/or distilled) to a level that will fully immerse the sub-sample(s) when the immersion container(s) is placed within it. The water shall be 23 ± 5 °C. If two baths are used the temperature in both baths shall not differ by more than 2 °C of each other.

Transfer a portion of $1,000 \text{ g} \pm 10 \text{ g}$ of each of the two sieved sub-samples (7.2) into separate immersion containers as specified in 5.4 and place each immersion container into a water bath (the water level shall completely cover the sub-sample during the entire immersion period).

The sub-sample shall remain in the water bath for $24 \text{ h} \pm 0,25 \text{ h}$. During this time, it is important not to move or otherwise disturb the water bath(s) or the immersion container(s) within the water bath(s) so as not to artificially generate fines. Air bubbles can form on the test material during immersion. If so, the air bubbles shall not be disturbed. Material that floats as a result of air bubbles or due to having a density $<1 \text{ g/cm}^3$ shall be held under the water level by using the immersion container cover specified in 5.4. In such cases the immersion container cover is placed on top of the immersion container and the water level is raised to a level above the screen of the immersion container cover.

Upon completion of the immersion period lift the immersion containers (and cover if present) out of the water bath(s) and allow the water to drain. To allow for a complete drain, carefully tilt the immersion container propping one side up at an angle. In doing so, care shall be used to minimize the amount of material movement during this process and no material shall be allowed to fall out of the immersion container. Allow it to sit for $30 \text{ min} \pm 1 \text{ min}$ at room temperature.

Fine material will likely be lost through the screen of the immersion container during the immersion period. This material can be discarded with the water. All material retained on the screen of the immersion container shall be used for subsequent testing.

7.4 Determination of moisture content and durability of the post-immersion sub-samples

After the sub-samples are fully drained use one sub-sample for the determination of moisture content post-immersion, M_{pi} , in accordance with ISO 18134-1 or ISO 18134-2, with the exception that the entire sub-sample is to be used in a single determination. The other sub-sample shall be poured into flat trays as specified in 5.5. Particles remaining in the immersion container or caught in the screen of the immersion container shall be transferred to the flat tray as part of the sample. Transfer as much of the material as possible using a spatula or other laboratory device without further washing the immersion container. Particles remaining in the water bath and water that is adhering to the immersion container are not to be transferred.

Place the flat tray with the sub-sample into the drying oven specified in 5.6. The drying temperature shall be set to a maximum of 40 °C and adjust the air velocity in such a way that sub-sample particles are not dislodged from the tray. Dry the sub-sample in the oven for several hours (overnight is preferred). The amount of drying time will depend on the amount of moisture absorbed by the material. The drying oven is used to remove the majority of the moisture gained during the test procedure so that the equilibration time required in the next step will not be excessive.

After drying in the oven, equilibrate the sub-sample within the flat tray in laboratory air for a minimum of two hours, but as long as necessary for the test material to reach equilibrium with the laboratory atmosphere. To determine if equilibrium has been reached weigh the sub-sample with the flat tray using the balance specified in 5.7. Record the weight and then let the sub-sample and flat tray sit for an additional hour in the lab atmosphere and then weigh again. Equilibrium is reached when the weight gained or lost during the additional hour of equilibration is less than 0,2 % of the total weight of the sub-sample and flat tray.

Once equilibrium is reached, test the material for durability in accordance with ISO 17831-1 with the exception that the material is not to be pre-screened and that all material is to be used by splitting the sample in half and both halves tested as-is for durability (tested weight may not be within the specified tolerance of $500 \text{ g} \pm 10 \text{ g}$). Record the result as durability post-immersion, DU_{pi} .

8 Calculations

All calculations pertaining to testing moisture content and durability are to be conducted in accordance with the calculation sections of their respective ISO test methods.

The water sorption, S_w , expressed as a mass fraction in % shall be calculated using [Formula \(1\)](#):

$$S_w = M_{pi} - M_{ar} \quad (1)$$

where

S_w is the amount of water sorption, expressed as a mass fraction in %;

M_{ar} is the moisture content of the as-received sample, expressed as a mass fraction in %;

M_{pi} is the moisture content of the sub-sample post-immersion, expressed as a mass fraction in %.

The post-immersion durability reduction, DUR_{pi} , expressed as a mass fraction in % shall be calculated using [Formula \(2\)](#):

$$DUR_{pi} = DU_{ar} - DU_{pi} \quad (2)$$

Where

DUR_{pi} is the post-immersion durability reduction expressed as a mass fraction, in %;

DU_{ar} is the durability of the as-received sample expressed as a mass fraction, in %;

DU_{pi} is the durability of the sub-sample post-immersion expressed as a mass fraction, in %;

The post-immersion durability reduction shall be reported to within a mass fraction of 0,1 %.

9 Performance characteristics

The achievable performance of the method is given in [Annex A](#), showing the results obtained by an international laboratory comparison study was carried out on two industrial samples of 6 mm diameter pellets made from thermally treated wood.

10 Test report

The test report shall include at least the following information:

- a) identification of laboratory performing the test and the date of the test;
- b) identification of product (or sample) tested;
- c) a reference to this document, i.e ISO 23343-1:2021;
- d) durability as-received, DU_{ar} , and moisture content as-received, M_{ar} , expressed as a mass fraction in %;
- e) durability post-immersion, DU_{pi} , and moisture content post-immersion, M_{pi} , expressed as mass fraction in %;
- f) post-immersion durability reduction, DUR_{pi} , expressed as a mass fraction, in %;
- g) water sorption, S_w , expressed as a mass fraction, in %;
- h) any unusual features noted during the determination which may affect the result;

- i) any operation not included in this document or regarded as optional.

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