
Solid biofuels — Determination of volatile matter

Biocombustibles solides – Détermination des matières volatiles

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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).

This second edition cancels and replaces the first edition (ISO 18123:2015), which has been technically revised.

The main changes are as follows:

- ISO 21945 added to [Clause 2](#);
- specification that the entire procedure shall be done in duplicate;
- EN standards replaced by ISO documents;
- minor editorial corrections.

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

Volatile matter is determined as the loss in mass, less that due to moisture, when a solid biofuel is heated out of contact with air under standardised conditions. The test is empirical and, in order to ensure reproducible results, it is essential that the rate of heating, the final temperature and the overall duration of the test are carefully controlled. It is also essential to exclude air from the solid biofuel during heating to prevent oxidation. The fit of the crucible lid is therefore critical. The moisture content of the general analysis sample is determined at the same time as the volatile matter so that the appropriate correction can be made.

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Solid biofuels — Determination of volatile matter

1 Scope

This document specifies the requirements and method used to determine the volatile matter of solid biofuels. It is intended for persons and organisations that manufacture, plan, sell, erect or use machinery, equipment, tools and entire plants related to solid biofuels, and for all persons and organisations involved in producing, purchasing, selling and utilizing solid biofuels.

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 14780, *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Vocabulary*

ISO 18134-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

ISO 18135, *Solid Biofuels — Sampling*

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 apply.

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

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

4 Principle

A portion of the general analysis sample is heated mostly out of contact with air at $900\text{ °C} \pm 10\text{ °C}$ for 7 min. The furnace is normally not equipped with a vacuum; however, there is a partial vacuum created during heating to a degree depending on air influx during charging of the test portion into the chamber and the air trapped in the crucible before the lid is put on. The percentage of volatile matter is calculated from the loss in mass of the test portion after deducting the loss in mass due to moisture.

Automatic equipment may be used when the method is validated with biomass reference samples of an adequate biomass type. The automatic equipment shall fulfil all the requirements given in [Clauses 5 to 8](#) regarding sample size, atmosphere, temperatures and weighing accuracy.

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

5.1 Furnace

The furnace shall be heated by electricity, in which a zone of uniform temperature of 900 °C ± 10 °C can be maintained (see example in [Figure 1](#)).

It is important for furnaces to have flues so that the furnace door seals well. The flue should not reach far out of the oven and should be fitted with a butterfly valve to restrict airflow through the furnace.

Its heating capacity shall be such that, with an initial temperature of 900 °C ± 10 °C, the temperature is regained within 4 min of insertion of a cold stand and its crucibles. The temperature is measured with a thermocouple, as described in [5.2](#).

Normally the furnace will be designed specifically either for multiple determinations using a number of crucibles in one stand or for receiving one crucible and its stand.

The crucible stand shall be placed in the middle of the furnace. The temperature of 900 °C shall be attained as closely as possible with a specified tolerance of ±10 °C in order to compensate for inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

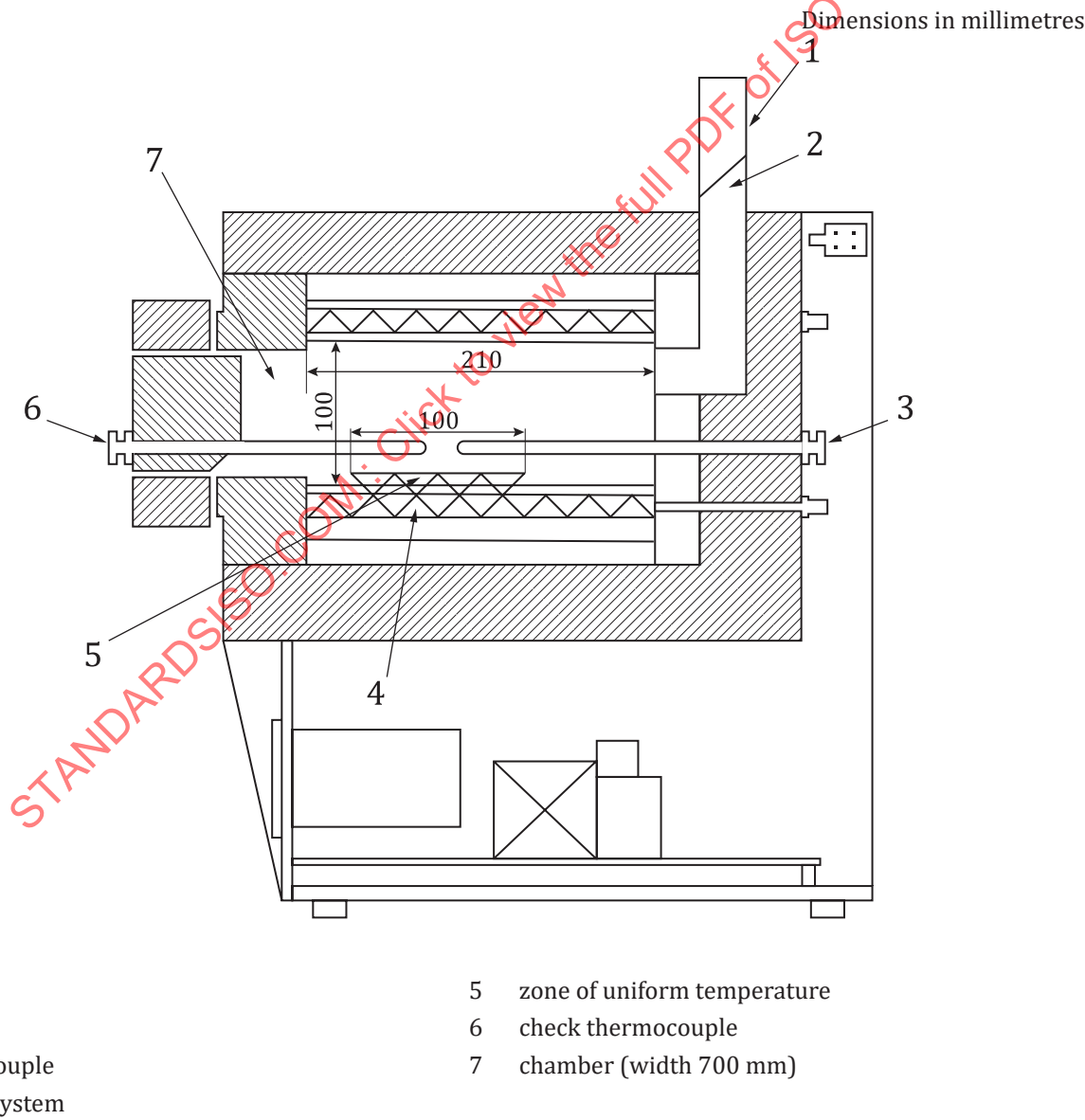


Figure 1 — Example of suitable furnace

5.2 Thermocouples and temperature calibration

A sheathed thermocouple shall be permanently installed in the furnace (see right hand side of [Figure 1](#)) with its thermo junction as close as possible to the centre of the heating chamber.

An unsheathed thermocouple (see left hand side of [Figure 1](#)) long enough to reach the centre of the heating chamber is used for calibration.

The furnace temperature readings shall be checked at regular time intervals with an unsheathed calibrated thermocouple. The unsheathed thermocouple (see [5.1](#)) shall be positioned as close as possible to the area of the permanently installed thermocouple.

The temperature–electromotive force (EMF) relationship of a thermo junction maintained at elevated temperatures gradually changes with time, which means that the time for the determination should not be longer than necessary.

5.3 Crucible

The crucible shall be cylindrical, with a well-fitting lid, both made of fused silica. The crucible with lid shall have a mass of between 10 g and 14 g and dimensions of approximately those shown in [Figure 2](#). The fit of the lid on the crucible is critical to the determination and a lid shall be selected to match the crucible so that the horizontal clearance between them is no greater than 0,5 mm. After selection, the crucible and the lid shall be ground together to give smooth surfaces and then be given a common distinguishing mark.

Dimensions in millimetres

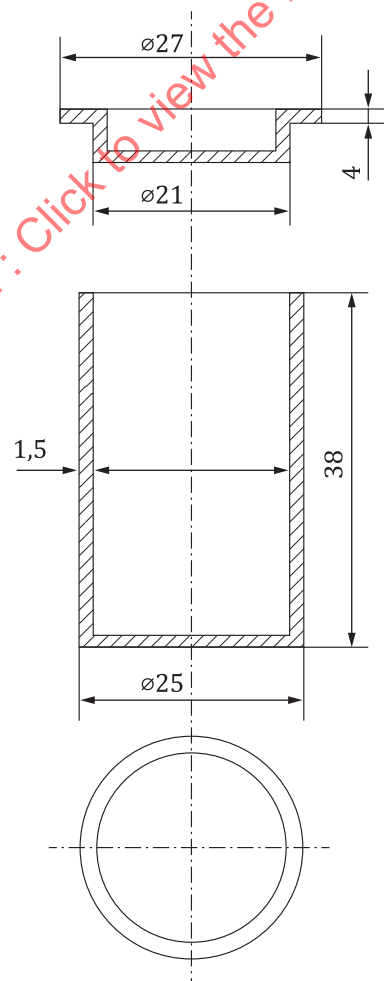


Figure 2 — Silica crucible and lid

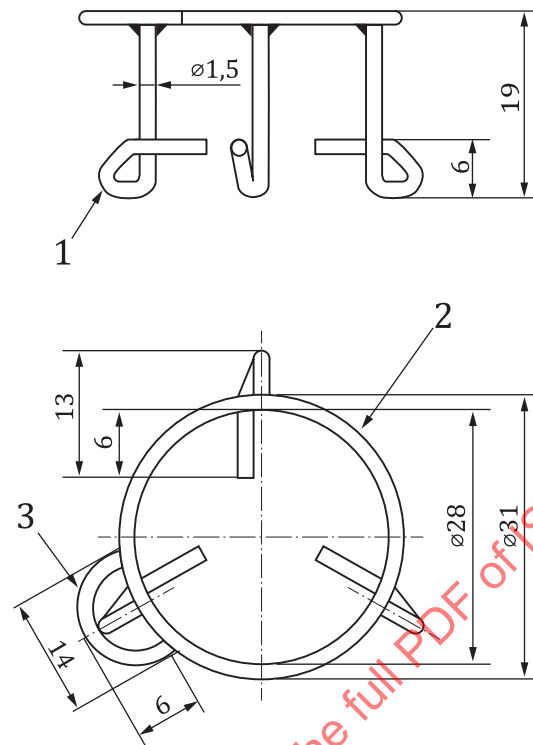
5.4 Crucible stand

The crucible stand to hold the crucible(s) in place in the furnace shall be such that the appropriate rate of heating can be achieved. For example, it can consist of one of the following:

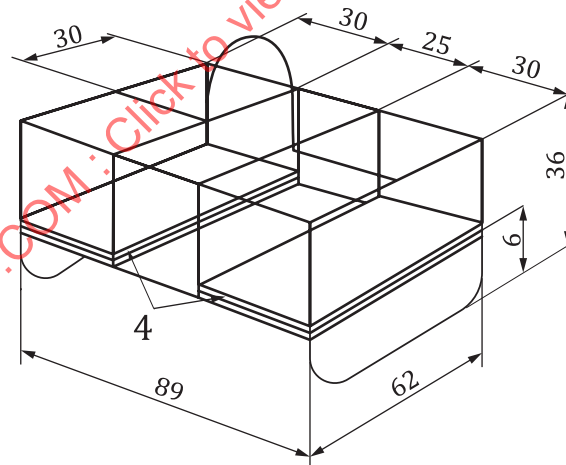
- a) for single determinations, a ring of heat-resistant steel wire as shown in [Figure 3 a\)](#), with a ceramic disc, 25 mm diameter and 2 mm thick, resting on the inner projection of its legs;
- b) for multiple determinations, a tray of heat-resistant steel wire as shown in [Figure 3 b\)](#), of appropriate size, with ceramic plates 2 mm thick supporting the crucibles.

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Dimensions in millimetres



a) Suitable for single determination (top: side view; bottom: top view)



b) Suitable for multiple determinations

Key

- 1 three legs placed 120° apart
- 2 ring
- 3 handle
- 4 ceramic plates

Figure 3 — Examples of crucible stands

5.5 Balance

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

5.6 Desiccator and desiccant

A desiccator with appropriate desiccant to prevent absorption of moisture from the atmosphere by the test portion.

WARNING — Ash from solid biofuel is very hygroscopic and there is a risk that moisture bound in the desiccant can be absorbed in the sample. Therefore, the desiccant shall be controlled frequently and dried if necessary. In addition, lids shall be used to cover dishes while in the desiccator to prevent the absorption of moisture.

6 Sample preparation

6.1 General

A test sample shall be obtained in accordance with ISO 18135 or ISO 21945. A general analysis sample shall be prepared in accordance with ISO 14780, for moisture determination and for determination of volatile matter. The material shall have a nominal top size of 1 mm or less.

6.2 Sample size

The general analysis sample shall include material sufficient for the determination of the volatile matter and the moisture content.

6.3 Sample conditioning

The determination of volatile matter shall be done either:

- a) directly on a test portion of a general analysis sample, including a concurrent determination of the moisture content in accordance with ISO 18134-3; or
- b) from a test portion of the general analysis sample which has been dried using the same drying procedure as in the determination of the moisture content of the test portion and kept absolutely dry before the weighing for the volatile matter determinations (the test portion shall be kept in a closed container in a desiccator with desiccant).

NOTE For some solid biofuels, it can be necessary to prepare a general analysis sample to a nominal top size of less than 1 mm (e.g. 0,25 mm) in order to keep the stated precision.

7 Procedure

7.1 General

The entire procedure shall be done in duplicate.

7.2 Conditioning of crucibles

Fill either a stand with one empty crucible and lid [see [Figure 3 a\)](#)] or a stand with the requisite number of empty crucibles and lids [see [Figure 3 b\)](#)] and insert in the furnace. Maintain at $900\text{ °C} \pm 10\text{ °C}$ for $7\text{ min} \pm 5\text{ s}$. Remove the stand with crucible(s) from the furnace and allow to cool to room temperature on a plate of thermo-resistant material and store the crucibles in a desiccator.

7.3 Charging of crucibles

Weigh the empty crucible(s) with lid(s) in cool conditions.