
**Solid biofuels — Determination of the
content of volatile matter**

*Biocombustibles solides — Méthode de détermination de la teneur en
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).

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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 238, *Solid biofuels*.

Introduction

The volatile matter content 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 the content of volatile matter

1 Scope

This International Standard aims to define the requirements and method used to determine the volatile matter content 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 to all persons and organisations involved in producing, purchasing, selling, and utilizing solid biofuels.

The volatile matter content is determined as the loss in mass, less that due to moisture, when solid biofuel is subject to partial pyrolysis under standardized conditions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

EN 14778¹⁾, *Solid Biofuels — Sampling*

EN 14780²⁾, *Solid biofuels — Sample preparation*

3 Terms and definitions

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

3.1

nominal top size

aperture of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559]

3.2

laboratory sample

combined sample or a sub-sample of a combined sample for use in a laboratory

[SOURCE: ISO 16559]

3.3

test portion

sub-sample either of a laboratory sample or a test sample

[SOURCE: ISO 16559]

1) To be replaced by ISO 18135.

2) To be replaced by ISO 14780.

3.4

general analysis sample

sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses

[SOURCE: ISO 16559]

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

5.1 Furnace

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

NOTE 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 $900\text{ °C} \pm 10\text{ °C}$, the temperature is regained within 4 min after 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 $\pm 10\text{ °C}$ in order to compensate for inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

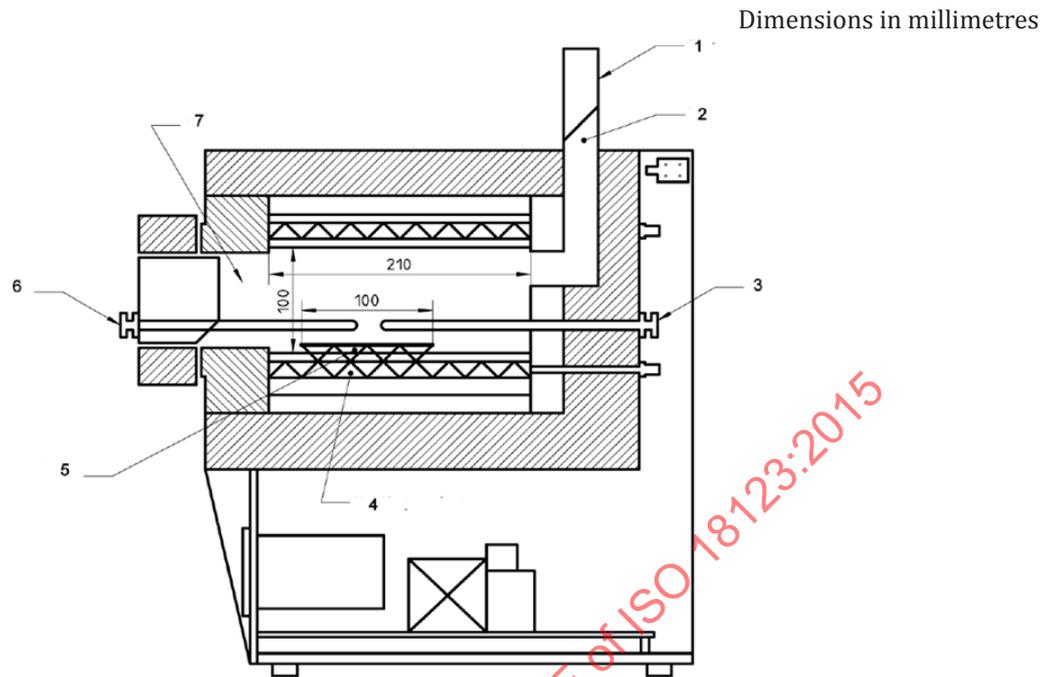


Figure 1 — Example of suitable furnace

Key

1	flue	5	zone of uniform temperature
2	valve	6	check thermocouple
3	thermocouple	7	chamber (width 700 mm)
4	heating system		

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 [Clause 5](#)) shall be positioned as close as possible to the area of the permanently installed thermocouple.

NOTE 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 between 10 g and 14 g and dimensions approximate 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

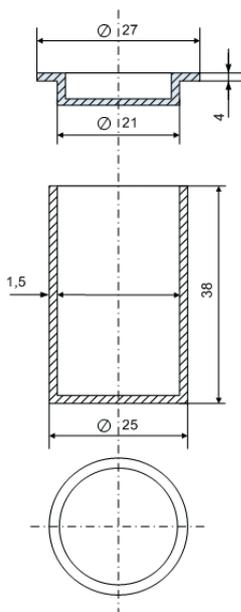


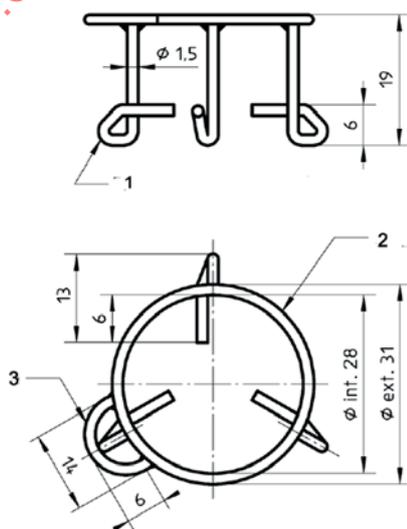
Figure 2 — Silica crucible and lid

5.4 Crucible stand

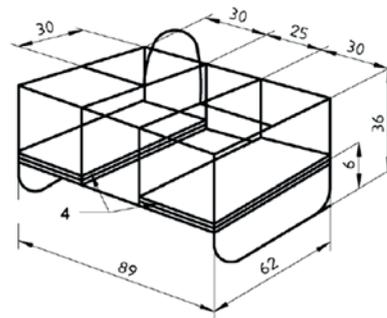
The crucible stand to hold the crucible(s) in place in the furnace shall be such that the appropriate rate of heating can be achieved. For example, it may consist of

- a) for single determinations, a ring of heat-resistant steel wire as shown in Figure 3 a), with ceramic discs, 25 mm diameter and 2 mm thick, resting on the inner projection of its legs, or
- 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.

Dimensions in millimetres



a) Suitable for single determination



b) Suitable for multiple determinations

Key

- 1 three legs paced 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

The test sample shall be obtained in accordance with EN 14778. A general analysis sample shall be prepared in accordance with EN 14780, for moisture determination and for determination of volatile content. The material shall have a nominal top size of 1 mm or less.

6.1 Sample size

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

6.2 Sample conditioning

The determination of volatile content 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 content determinations (the test portion shall be kept in a closed container in a desiccator with desiccant).

NOTE For some solid biofuels, it could 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 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 $900\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ 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.

7.2 Charging of crucibles

Weigh empty crucible(s) with lid(s) in cool condition.

Fill crucible(s) with $1\text{ g} \pm 0,1\text{ g}$ to the nearest 0,1 mg of material from the general analysis sample. Replace the lid(s) and tap the crucible(s) on a clean hard surface until the material forms a layer of even thickness at the bottom of the crucible.

7.3 Volatilization of test portion

Place the charged crucible(s) again in a cool stand and place in the furnace already maintained at $900\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$. Close the door and leave for $7\text{ min} \pm 5\text{ s}$.

Remove the crucible(s) from the furnace and place on a thermo-resistant surface and allow to cool for approximately 5 min to 10 min and then place the crucible(s) in a desiccator and let cool to room temperature. When cool, weigh the crucible(s) to the nearest 0,1 mg.

NOTE 1 The initial heating and cooling of crucible(s) followed by storage in desiccator and final heating and cooling followed by storage in a desiccator minimizes the risk of distortion of results due to water adsorption on surfaces. Also, the rapid cooling and storage in desiccator minimizes the absorption of moisture by the solid biofuel material.

NOTE 2 If multiple determinations are being made, any vacant places in the stand are to be filled with empty crucibles.

NOTE 3 For some types of solid biofuels, it could be necessary to carry out the determination on a dry sample in order to avoid loss of material due to violent reactions during the heating process. In such case, the charged crucible(s) are dried at $105\text{ }^{\circ}\text{C}$ and cooled in accordance with ISO 18134-3, before being placed in the furnace.

8 Calculation

The volatile matter V_d in the test portion, expressed as a percentage by mass on the dry basis, is given by Formula (1):

$$V_d = \left[\frac{100(m_2 - m_3)}{m_2 - m_1} - M_{\text{ad}} \right] \times \left(\frac{100}{100 - M_{\text{ad}}} \right) \quad (1)$$

where

m_1 is the mass in g of the empty crucible and lid;