BS EN ISO 18123:2015
Incorporating corrigendum January 2017

BSI Standards Publication

Solid biofuels — Determination of the content of volatile matter

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National foreword

This British Standard is the UK implementation of EN ISO 18123:2015. Itsupersedes BS EN 15148:2009 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2017

ISBN 978 0 580 97272 0

ICS 75.160.40

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2015.

Amendments/corrigenda issued since publication

Date	Text affected
31 January 2017	Correction to supersession details in national foreword

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 18123

November 2015

ICS 75.160.10

English Version

Solid biofuels - Determination of the content of volatile matter (ISO 18123:2015)

Biocombustibles solides - Méthode de détermination de la teneur en matières volatiles (ISO 18123:2015)

Biogene Festbrennstoffe - Bestimmung des Gehaltes an flüchtigen Bestandteilen (ISO 18123:2015)

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Ref. No. EN ISO 18123:2015 E

European foreword

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This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

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Foreword

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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.

INTERNATIONAL STANDARD ISO 18123:2015(E)

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 14778[1)], *Solid Biofuels — Sampling*

EN 14780[2)], *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 °C ± 10 °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 °C ± 10 °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 °C ± 10 °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 ±10 °C in order to compensate for inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

Dimensions in millimetres

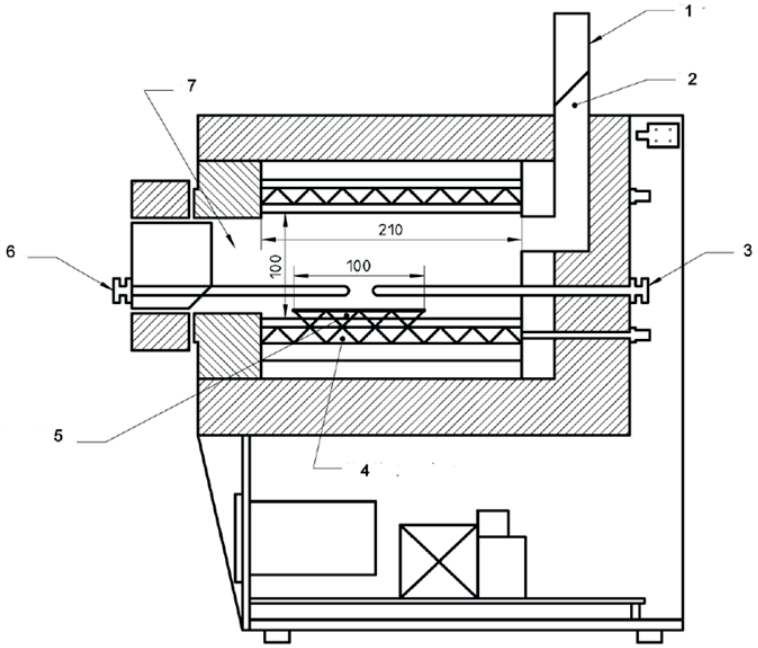


Figure 1 — Example of suitable furnace

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

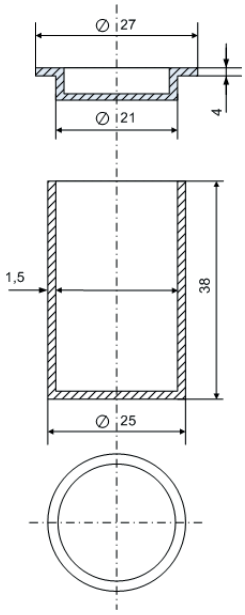


Figure 2 — Silica crucible and lid

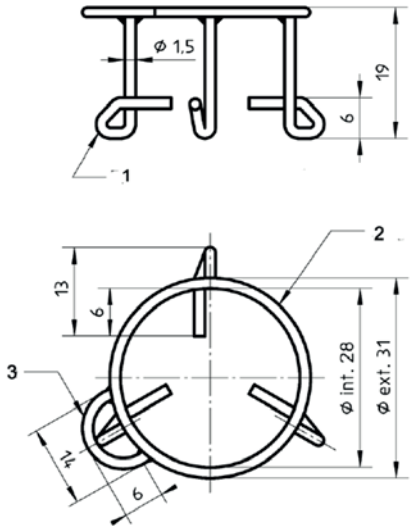
5.4 Crucible stand

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

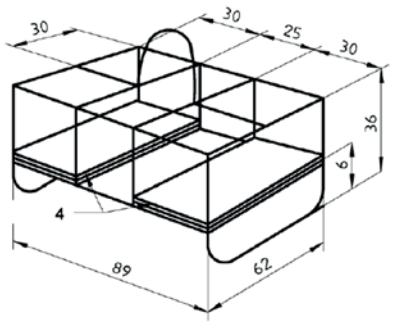
a) for single determinations, a ring of heat-resistant steel wire as shown in Figure 3 a), with ceramic discs, 25 mm diameter and 2 mm thick, resting on the inner projection of its legs, or

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 °C ± 10 °C for 7 min ± 5 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 g ± 0,1 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 °C ± 10 °C. Close the door and leave for 7 min ± 5 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 °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):

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

where

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

m_2 is the mass in g of the crucible and lid and test portion before heating;

m_3 is the mass in g of the crucible and lid and contents after heating;

M_{ad} is the moisture, as a percentage by mass, in the test portion as determined in accordance with ISO 18134-3.

The result shall be calculated to two decimal places and the mean value shall be rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

9.1 Repeatability

The results of duplicate determinations (performed within a short period of time, but not simultaneously) in the same laboratory, by the same operator, using the same apparatus on two representative portions taken from the same general analysis sample shall not differ by more than the value given in Table 1, see References [2], [3], [4], [5], and [6].

9.2 Reproducibility

The mean value of the results of duplicate determinations carried out in two different laboratories on representative portions taken from the same general analysis sample, shall not differ by more than the value given in Table 1, see Reference[2]. The reproducibility is however somewhat depending on the flux of air in the crucible and the furnace. Diligence in the handling during the procedures may improve reproducibility, see References [2], [3], [4], [5], and [6].

Table 1 — Repeatability and reproducibility of the method

Volatile matter	**Maximum acceptable differences between results obtained** (calculated on dry basis)	
	Repeatability limit	**Reproducibility limit**
Solid biofuel	1,0 % of the mean result	3,0 % of the mean result

10 Test report

The test report shall include the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) a reference to this International Standard, i.e. ISO 18123;

d) results of the test on dry basis or alternatively as indicated in Clause 9;

e) any unusual features noted during the determination; which may affect the result;

f) any deviation from this International Standard, or operations regarded as optional.

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