BRITISH STANDARD

BS EN 15148:2009

Solid biofuels — Determination of the content of volatile matter

ICS 75.160.10

BSI
British Standards

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National foreword

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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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Solid biofuels - Determination of the content of volatile matter

Biocombustibles solides - Méthode de détermination de la teneur en matières volatiles

Feste Biobrennstoffe - Bestimmung des Gehaltes an flüchtigen Substanzen

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Foreword

This document (EN 15148:2009) has been prepared by Technical Committee CEN/TC 335 “Solid biofuels”, the secretariat of which is held by SIS.

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of an identical text or by endorsement, at the latest by May 2010, and conflicting national standards shall be withdrawn at the latest by May 2010.

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Introduction

The volatile matter content is determined as the loss in mass, less that due to moisture, when 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.

1 Scope

This European 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 utilising solid biofuels.

The volatile matter content is determined as the loss in mass, less that due to moisture, when solid biofuel is heated out of contact with air under standardized conditions.

2 Normative references

The following referenced documents are indispensable for the application 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.

CEN/TS 14588:2003, *Solid biofuels – Terminology, definitions and descriptions*

CEN/TS 14780, *Solid biofuels — Methods for sample preparation*

EN 14774-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 14588:2003 shall apply.

4 Principle

A test portion of the general analysis sample is heated out of contact with ambient air at (900 ± 10) °C for 7 min. 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. This 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 ± 10) °C can be maintained. It may be of the stop-ended type or fitted at the back with a flue (see Figure 1, as example).

NOTE It is important for furnaces with flues 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 heat capacity shall be such that, with an initial temperature of (900 ± 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.

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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.

5.2 Thermocouples

A sheathed thermocouple shall be permanently installed in the furnace (see Figure 1) with its thermojunction as close as possible to the centre of the heating chamber.

An unsheathed thermocouple long enough to reach the centre of the heating chamber. This thermocouple is used for calibration.

5.3 Crucible

The crucible shall be cylindrical, with a well-fitting lid, both of fused silica. The crucible with lid shall have a mass between 10 g and 14 g and dimensions approximating to 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.

5.4 Crucible stand

The crucible stand on which the crucible is placed in the furnace, shall be such that the appropriate rate of heating can be achieved. For example, it may consist of the following:

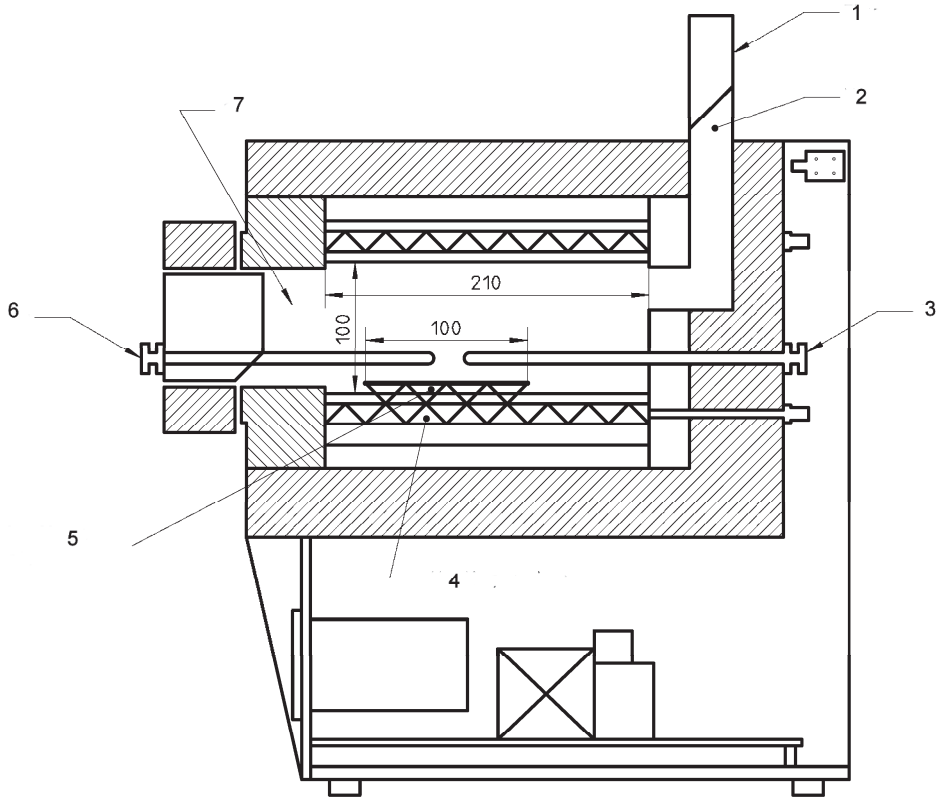
a) for single determinations, a ring of heat-resistant steel wire as shown in Figure 3 a) with ceramic discs, 25 mm diameter and 2 mm thick, resting on the inner projection of its legs; or

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.

5.5 Balance

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

Dimensions in millimetres



Key

1 Flue
2 Valve
3 Thermocouple
4 Heating system
5 Zone of uniform temperature
6 Check thermocouple
7 Chamber (width 200 mm)

Figure 1 — Example of suitable furnace

Dimensions in millimetres

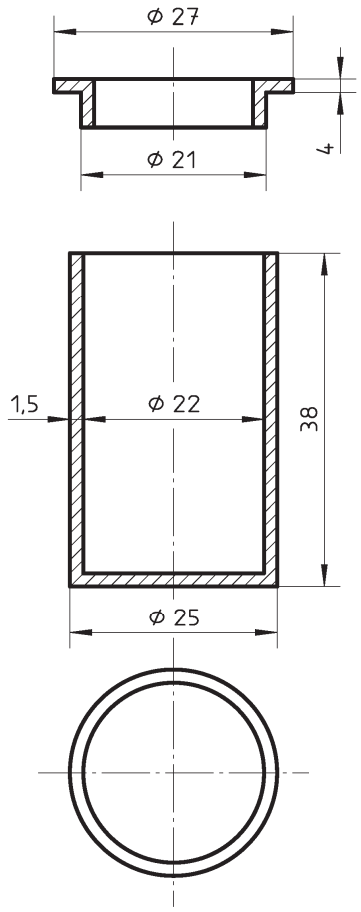
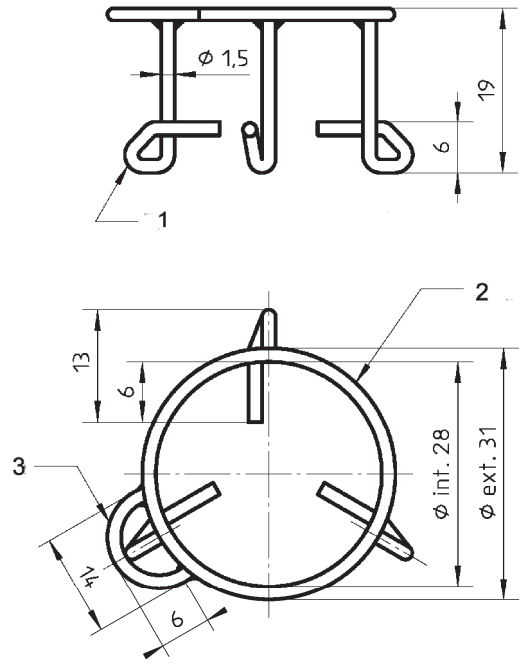
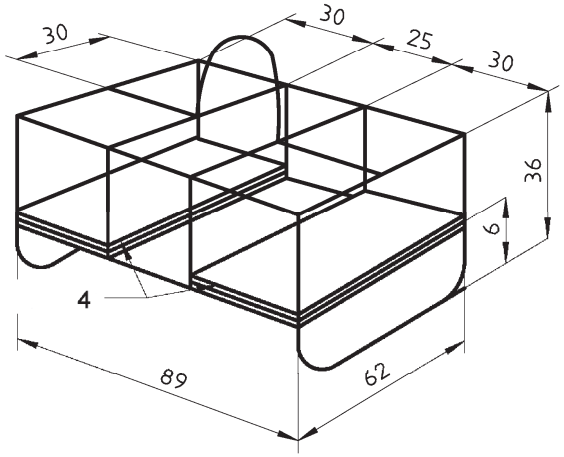


Figure 2 — Silica crucible and lid

Dimensions in millimetres



a) Suitable for a single determination



b) Suitable for multiple determinations

Key

1 Three legs spaced 120° apart
2 Ring
3 Handle
4 Ceramic plates

Figure 3 — Examples of crucible stands

6 Temperature 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 relationship of a thermojunction maintained at elevated temperatures gradually changes with time.

7 Preparation of the test sample

The test sample is the general analysis sample with a nominal top size of 1 mm or less, prepared in accordance with CEN/TS 14780.

The general analysis sample shall be well mixed and in moisture equilibrium with the laboratory atmosphere, or oven-dry (dried by the same procedure as described in EN 14774-3).

A test portion of the general analysis sample shall be separated for the determination of moisture content at the same time to the determination of volatile matter. The moisture content shall be determined according to EN 14774-3.

8 Procedure

Fill either a stand with one empty crucible and lid (Figure 3a) or a stand with the requisite number of empty crucibles and lids (Figure 3b) and insert in the oven. Maintain at (900 ± 10) °C for 7 min. Remove the crucible(s) from the furnace and allow to cool to room temperature on a plate of thermo-resistant material and store in desiccator.

Weigh cool empty crucible(s) and lid(s) and weigh (1 ± 0,1) g test portion of the general analysis sample into the crucible(s) to the nearest 0,1 mg. Replace the lid(s) and tap the crucible(s) on a clean hard surface until the test portion forms a layer of even thickness on the bottom of the crucible

Place the charged crucible(s) in a cold stand, transfer to the furnace, close the door and leave for 7 min ± 5 s.

Place the crucible(s) on a thermo-resistant plate and allow to cool to 30 °C to 50 °C above room temperature and finally to room temperature in a dessicator. When cool, weigh the crucible(s) to the nearest 0,1 mg.

NOTE 1 The same treatment of the crucible before and after the determination minimizes the effect of any film of water adsorbed on its surface, while the rapid cooling reduces absorption of moisture by the solid biofuel residue.

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

NOTE 3 For some types of solid biofuels it may be necessary to carry out the determination on dry sample in order to avoid loss of material due to violent reactions during the heating process. In this case, the charged crucibles are dried at 105 °C and cooled, according to EN 14774-3, before the transfer in the furnace.

9 Calculation

The volatile matter V_d in the sample, expressed as a percentage by mass on the dry basis, is given by the equation:

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

where

$m1$ is the mass, in g, of the empty crucible and lid;

$m2$ is the mass, in g, of the crucible and lid and test portion before heating;

$m3$ 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 general analysis sample as analysed, determined according to EN 14774-3.

Report the result, as the mean of duplicate determinations, rounded to the nearest 0,1 % (m/m). The results of the determination described in this European Standard are reported on the dry basis.

10 Precision

10.1 Repeatability

The results of the 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 test portions taken from the same general analysis sample, shall not differ by more than the value given in Table 1. See [2] in the bibliography.

10.2 Reproducibility

The means of the results of duplicate determinations, performed in each of two different laboratories on representative test portions taken from the same general analysis sample shall not differ by more than the values given in Table 1.

Table 1

Volatile matter	**Maximum acceptable differences between results obtained** (calculated to dry basis)	
	Repeatability limit	**Reproducibility critical difference**
Solid biofuel	2,0 % of the mean result	4,0 % of the mean result

11 Test report

The test report shall include the following information:

a) identification of the laboratory and the testing date;

b) identification of the product or sample tested;

c) reference to this European Standard;

d) any deviations from this European Standard;

e) test results of the determination on dry basis;

f) conditions and observations, i.e. unusual features, during the test procedure, which may affect the results.

The test results shall be expressed with relevant symbols.

Bibliography

[1] ISO 562, *Hard coal and coke – Determination of volatile matter*

[2] OFI – VT 2007 (interlaboratory comparison tests, round robins), Final Report , Vienna, December 2007

[3] ASTM D6374-99(2004), Standard Test Method for Volatile Matter in Green Petroleum Coke Quartz Crucible Procedure

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