BS EN 15402:2011

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Solid recovered fuels — Determination of the content of volatile matter

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National foreword

This British Standard is the UK implementation of EN 15402:2011. It supersedes DD CEN/TS 15402:2006 which is withdrawn.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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English Version

Solid recovered fuels - Determination of the content of volatile matter

Combustibles solides de récupération - Détermination de la teneur en composés volatils

Feste Sekundärbrennstoffe - Bestimmung des Gehaltes an flüchtigen Substanzen

This European Standard was approved by CEN on 22 January 2011.

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Foreword

This document (EN 15402:2011) has been prepared by Technical Committee CEN/TC 343 “Solid recovered fuels”, the secretariat of which is held by SFS.

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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

This document supersedes CEN/TS 15402:2006.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document differs from CEN/TS 15402:2006 mainly as follows:

a) use of automatic equipments under specific conditions permitted;

b) results of interlaboratory tests supplemented as an informative Annex A;

c) whole document editorially revised.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

The volatile matter is determined as the loss in mass less that due to moisture, when solid recovered fuel 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 recovered fuel during heating to prevent oxidation. The fit of the crucible lid is therefore critical.

The moisture content of the sample is determined at the same time as the volatile matter so that the appropriate correction can be made. Mineral matter associated with the sample can also lose mass under the conditions of the test, the magnitude of the loss being dependent on both the nature and the quantity of the minerals present.

This European Standard is primarily geared toward laboratories, producers, suppliers and purchasers of solid recovered fuels, but is also useful for the authorities and inspection organizations.

The method specified in this European Standard is based on EN 15148 as well as ISO 562.

1 Scope

This European Standard specifies the requirements and a method for the determination of volatile matter of solid recovered fuels.

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.

EN 15357:2011, *Solid recovered fuels — Terminology, definitions and descriptions*

EN 15414-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

EN 15442, *Solid recovered fuels — Methods for sampling*

EN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15357:2011 apply.

4 Principle

A test portion of the general analysis sample is heated out of contact with ambient air at (900 ± 10) °C for about 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.

5 Apparatus

5.1 Furnace

The furnace shall be heated electrically and capable of maintaining a zone with uniform temperature of (900 ± 10) °C. It may be of the stop-ended type or fitted at the back with a flue with a diameter of about 25 mm and a length of about 150 mm (see Figure 1).

NOTE 1 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.

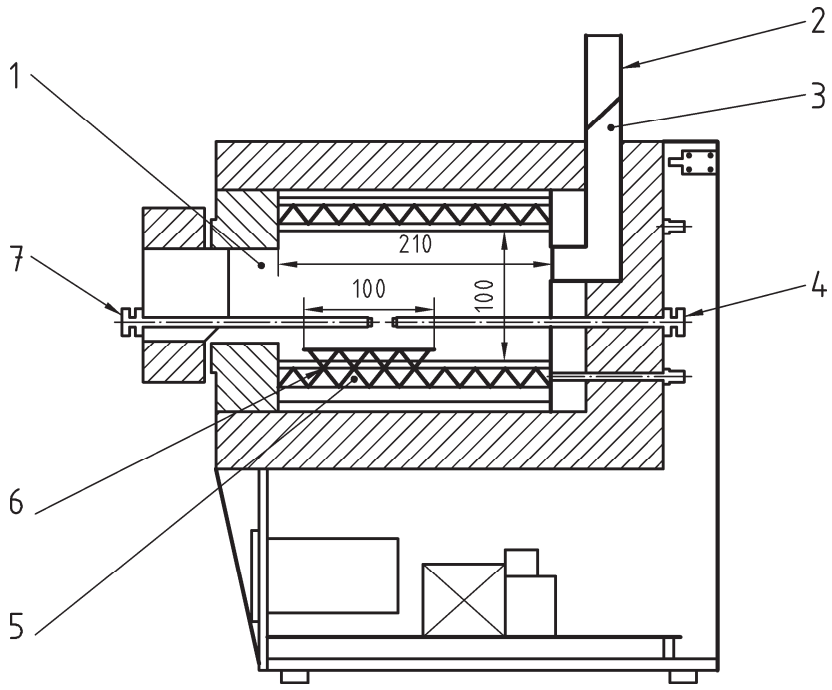
The heat capacity of the furnace shall be such that, with an initial temperature of (900 ± 10) °C, the temperature is regained within about 4 min after insertion of a cold stand and its crucibles. The temperature shall be measured with a thermocouple, as specified in 5.2.

NOTE 2 Observing the temperature is very important in order to compensate for inherent deviations of the temperature measurement and lack of uniformity regarding the temperature distribution.

Usually 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. In the first case, the zone of uniform temperature shall be at least 160 mm × 100 mm; in the latter case, a zone of diameter about 40 mm is sufficient.

A position for the crucible stand shall be chosen within the zone of uniform temperature and this position shall be used for all determinations.

Dimensions in millimetres



Key

1 chamber, 200 mm width
2 flue
3 valve
4 thermocouple
5 heating system
6 zone of uniform temperature
7 check thermocouple

Figure 1 — Example of suitable furnace

5.2 Thermocouple

The thermocouple shall be unsheathed, of wire with a thickness ≤ 1 mm. It should be long enough to reach the centre of the underside of each crucible when placed into the zone of uniform temperature on being inserted through the front or rear of the furnace. The thermo junction shall be placed midway between the base of the crucible in its stand and the floor of the furnace. If the stand holds more than one crucible, the temperature under each crucible shall be checked in the same manner.

If desired, a sheathed thermocouple may be permanently installed in the furnace (5.1) (see Figure 1) with its thermo junction as close as possible to the centre of the zone of uniform temperature; in this case furnace temperature readings shall be correlated at frequent intervals with those of the unsheathed thermocouple which is thus inserted only if necessary.

NOTE The temperature/electromotive force relationship of a thermo junction maintained at elevated temperatures gradually changes with time.

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 from 10 g to 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 such 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

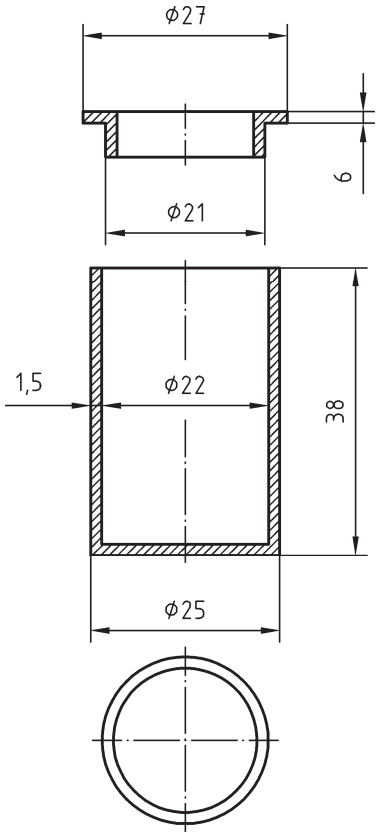


Figure 2 — Silica crucible and lid

If performing multiple determinations on highly swelling solid recovered fuels, it can be necessary to use taller crucibles; these may be up to 45 mm in height without affecting the determined volatile matter, provided that the specified rate of temperature recovery is maintained.

5.4 Crucible stand

The crucible stand which the crucible (5.3) is placed on in the furnace (5.1), shall be such that the appropriate rate of heating is achievable. 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 with a diameter of about 25 mm and a thickness of about 2 mm, resting on the inner projection of its legs

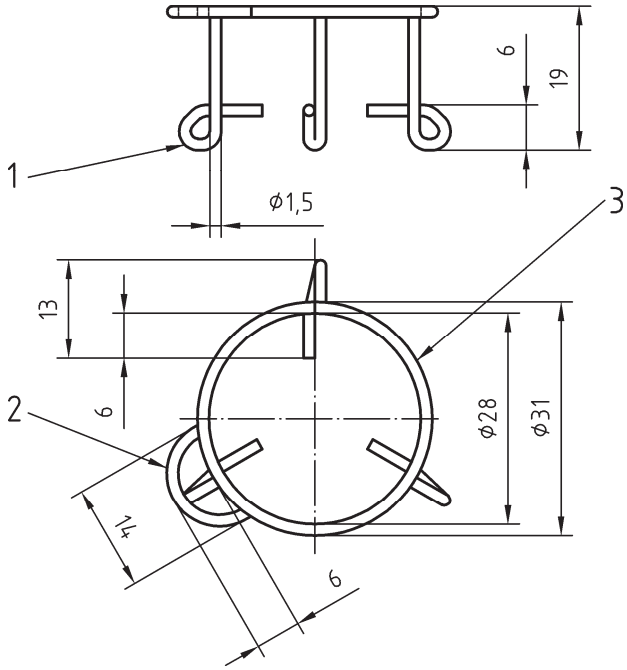
or

b) for multiple determinations, a tray of heat-resistant steel wire as shown in Figure 3 b), of appropriate size, with ceramic plates with a thickness of about 2 mm supporting the crucibles.

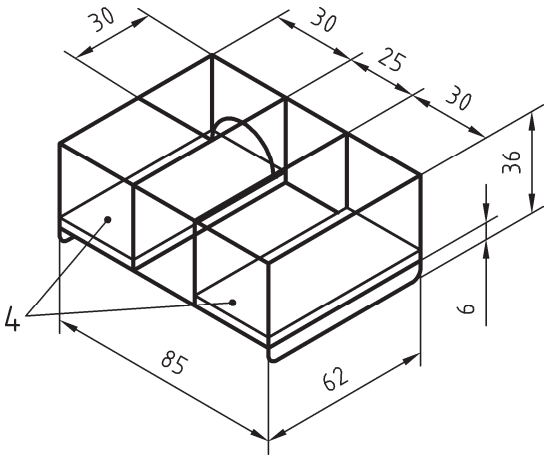
5.5 Balance

The balance shall be readable to the nearest 0,1 mg.

Dimensions in millimetres



a) Suitable for a single determination



b) Suitable for multiple determinations

Key

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

Figure 3 — Crucible stands

6 Sampling and sample preparation

The general analysis sample shall be taken and prepared in accordance with EN 15442 and EN 15443. It shall be well mixed and in moisture equilibrium with the laboratory atmosphere.

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 in accordance with EN 15414-3.

7 Procedure

7.1 Use of automatic equipment

Automatic equipment may be used if the procedure based on such a equipment is validated with reference materials of an adequate type. This equipment shall fulfil all the requirements given in Clauses 5 to 8 regarding sample size, atmosphere, temperatures and weighing accuracy. Deviations from these specifications shall be reported and justified.

7.2 Number of determinations

A minimum of two determinations shall be carried out on the general analysis test sample.

7.3 Furnace temperature checking

Adjust the temperature of the zone in the furnace (5.1), containing either a stand with one crucible and lid (see Figure 3 a)) or a stand with the requisite number of crucibles and lids (see Figure 3 b)), to (900 ± 10) °C as indicated by the correctly located thermocouple (5.2). Check that this temperature is observed at the same height under each crucible (5.3).

NOTE Temperature checking should be made before starting determinations. However, if several analyses are performed per day, a daily temperature check is usually sufficient. The check of the temperature recovery criterion (see 5.1) should be dealt with in a similar way.

7.4 Volatile matter determination

Fill either a stand with one empty crucible (5.3) and lid (see Figure 3 a)) or a stand with the requisite number of empty crucibles and lids (see Figure 3 b)) and insert it into the furnace (5.1). Maintain the temperature at (900 ± 10) °C for about 7 min. Remove the crucible(s) from the furnace and allow to cool to room temperature on a thick metal plate.

As soon as they are cool, weigh each empty crucible and lid and weigh into each crucible, to the nearest 0,1 mg, (1 ± 0,1) g of the general analysis sample. Replace the lid and tap each crucible 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 (420 ± 5) s.

Remove and allow to cool to room temperature. If cool, weigh the crucible(s) to the nearest 0,1 mg in the same manner as for the empty crucible(s).

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

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

8 Calculation

The volatile matter, V, in the sample as analysed, expressed as mass fraction in percent, is given by Equation (1):

$$V = \frac{100\,(m_2 - m_3)}{(m_2 - m_1)} - M \qquad (1)$$

where

m_1 is the mass of the empty crucible (5.3) and lid, in grams;

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

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

M is the mass fraction of moisture in the general analysis sample as analysed, in percent.

Report the result as the mean of duplicate determinations, rounded to the nearest 0,1 % mass fraction. The results of the determination described in this European Standard shall be reported on dry basis.

9 Precision

9.1 Repeatability limit

The maximum difference to be expected between two independent single test results of one laboratory at a confidence level of 95 % will not exceed the repeatability limit in more than 5 % of cases when measuring the same measurand in the same medium, using the same facilities and fulfilling all requirements of the test method (interlaboratory testing).

Precision data derived from the interlaboratory test are given in Annex A.

9.2 Reproducibility limit

The maximum difference to be expected between two independent single test results of different laboratories at a confidence level of 95 % will not exceed the reproducibility limit in more than 5 % of cases when measuring the same measurand in the same medium, each laboratory using their own facilities and fulfilling all requirements of the test method (interlaboratory testing).

Precision data derived from the interlaboratory test are given in Annex A.

10 Test report

The test report shall include the following information:

a) identification of the laboratory and the testing date;

b) identification of the sample tested;

c) a reference to this European Standard, i.e. EN 15402;

d) method used;

e) test results of the determination performed on dry basis;

f) any deviation from this European Standard;

g) any unusual features observed during the determination which may have affected the test result and details of any operations not included in this European Standard or regarded as optional.

Annex A
(informative)

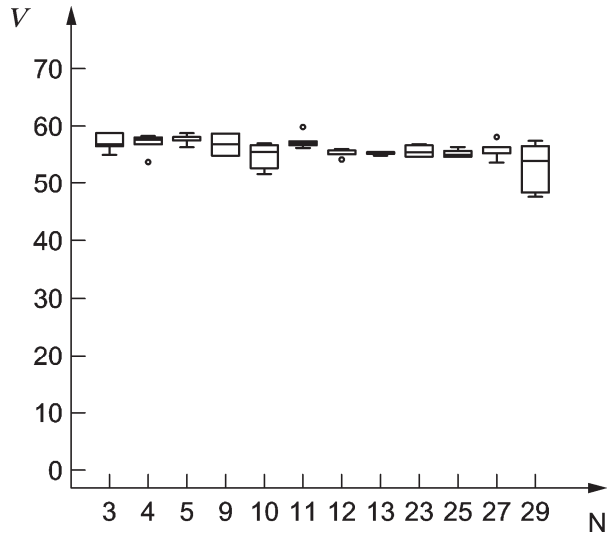
Interlaboratory test results

The statistic evaluation of the interlaboratory test results was carried out in accordance with ISO 5725-5. The precision data obtained are shown in Table A.1.

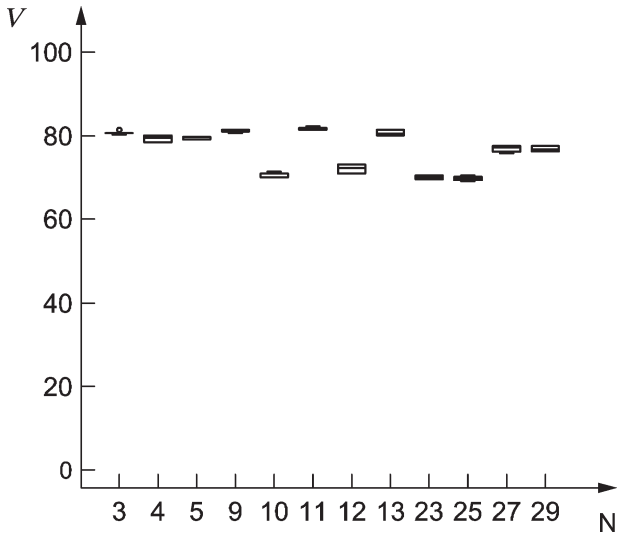
Table A.1 — Precision data

Designation	Shredded tyre	Demolition wood	Dried sludge	Municipal waste	Plastic/ paper fluff
Number of laboratories participating	12	12	12	12	12
Total number of values (without outliers)	69	70	72	69	60
Mean value, in % mass fraction	56,10	76,40	25,90	71,21	66,23
Laboratory effect, in % mass fraction	1,16	1,52	0,18	0,51	0,86
Sample effect, in % mass fraction	0,50	0,09	0,01	0,17	0,19
Repeatability standard deviation, s_r , in % mass fraction	1,05	0,20	0,06	0,27	0,56
Repeatability limit, r: ($r = 2{,}8 \times s_r$) in % mass fraction	2,94	0,56	0,17	0,76	1,57
Reproducibility standard deviation, s_R in % mass fraction	1,83	4,65	1,82	2,75	3,22
Reproducibility limit, R: ($R = 2{,}8 \times s_R$) in % mass fraction	5,12	13,02	5,10	7,70	9,02

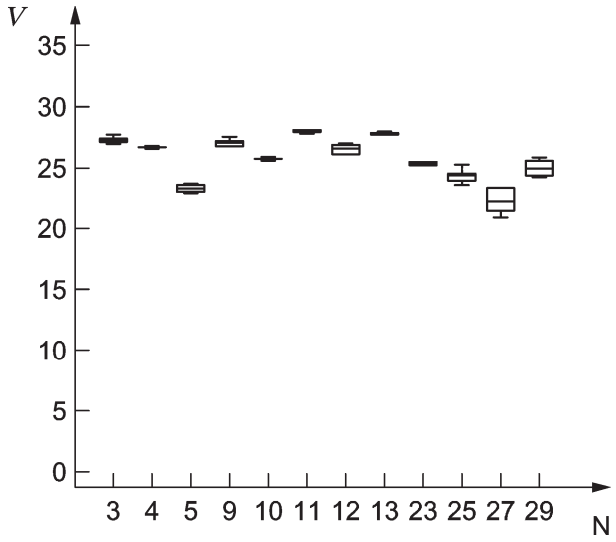
The deviations of the test results between the individual laboratories for each sample type are shown in Figures A.1 a) to A.1 e).



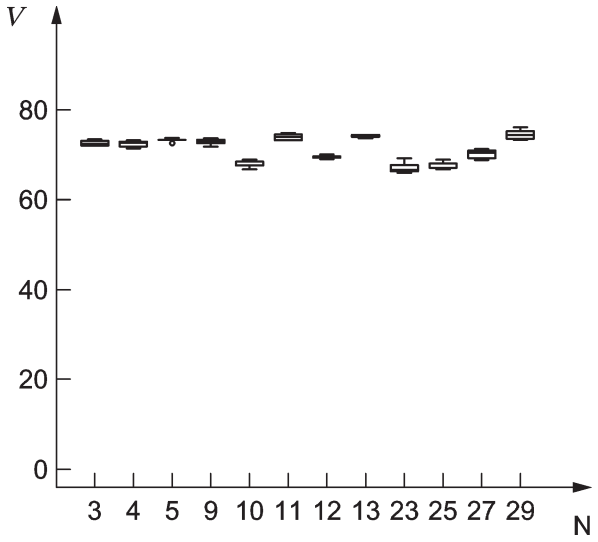
a) Shredded tyre



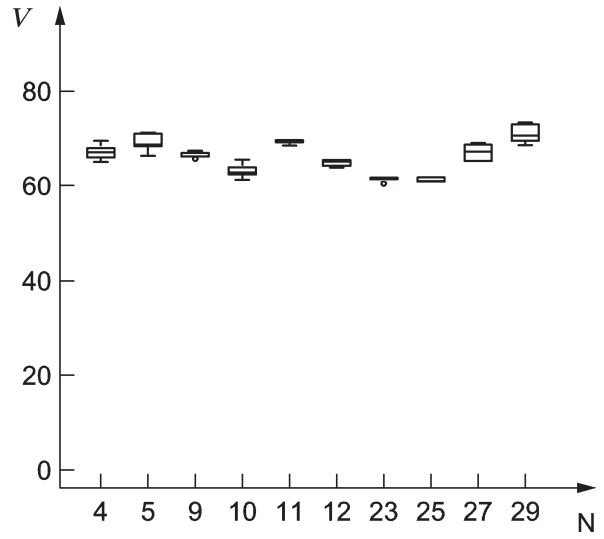
b) Demolition wood



c) Dried sludge



d) Municipal waste



e) Plastic and paper fluffs

Key

V mass fraction of volatile matter in %
N number of the individual laboratory

Figure A.1 — Deviations of the test results between the individual laboratories

Bibliography

[1] EN 15148, *Solid biofuels — Method for the determination of the content of volatile matter*

[2] ISO 562, *Hard coal and coke — Determination of volatile matter*

[3] ISO 5725-5, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

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