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**Reaction to fire tests for building  
products — Determination of the heat  
of combustion**

*Essais de réaction au feu des produits de construction — Détermination  
de la chaleur de combustion*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1716 was prepared by the European Committee for Standardization (CEN) in collaboration with Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Throughout the text of this document, read "...this European Standard..." to mean "...this International Standard...".

This second edition cancels and replaces the first edition (ISO 1716:1973), which has been technically revised.

Annex A forms a normative part of this International Standard. Annexes B to D are for information only.

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## Foreword

This document has been prepared by Technical Committee CEN /TC 127 "*Fire safety in buildings*", the secretariat of which is held by BSI, in collaboration with Technical Committee ISO/TC 92 "*Fire safety*", SC 1 "*Fire initiation and growth*".

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of the Construction Products Directive.

This document shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2002, and conflicting national standards shall be withdrawn by December 2003.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this document: Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

## Introduction

In this test a test specimen of specified mass is burned under standardized conditions, at constant volume, in an atmosphere of oxygen, in a bomb calorimeter calibrated by combustion of certified benzoic acid. The heat of combustion determined under these conditions is calculated on the basis of the observed temperature rise, taking account of heat loss and the latent heat of vaporisation of water.

It should be noted that this is a test method for determining an absolute value of the heat of combustion for a product and does not take into account any inherent variability of the product.

## Safety warning

So that suitable precautions are taken to safeguard health, the attention of all persons concerned in fire tests is drawn to the possibility that toxic or harmful gases may be evolved in the combustion of test specimens. In this test procedure, suitable measures should be taken to prevent harm in the event of an explosion.





## 1 Scope

This European Standard specifies a method for the determination of the heat of combustion of building products at constant volume in a bomb calorimeter.

This European Standard describes a test method for the measurement of the gross heat of combustion (*PCS*). Annex A describes the calculation of the net heat of combustion (*PCI*) when required.

Information on the precision of the test method is given in Annex B.

## 2 Normative references

This European Standard incorporates by dated or undated reference provisions from other publications. These normative references are cited at appropriate places in the text and the publications are listed hereafter. For dated references subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 13238, *Reaction to fire tests for building products — Conditioning procedures and general rules for selection of substrates*

EN ISO 13943:2000, *Fire safety — Vocabulary (ISO 13943:2000)*

## 3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in EN ISO 13943:2000, together with the following terms and definitions, apply.

### 3.1

#### **product**

material, element or component about which information is required

### 3.2

#### **material**

single basic substance or uniformly dispersed mixture of substances e.g. metal, stone, timber, concrete, mineral wool with uniformly dispersed binder, polymers

### 3.3

#### **homogeneous product**

product consisting of a single material having uniform density and composition throughout the product

### 3.4

#### **non-homogeneous product**

product that does not satisfy the requirements of a homogeneous product and which is composed of more than one component, substantial and/or non-substantial

### 3.5

#### **substantial component**

material that constitutes a significant part of a non-homogeneous product, a layer of which having a mass/unit area  $\geq 1,0 \text{ kg/m}^2$  or a thickness  $\geq 1,0 \text{ mm}$

**3.6**

**non-substantial component**

material that does not constitute a significant part of a non-homogeneous product. A layer with a mass/unit area  $< 1,0 \text{ kg/m}^2$  and a thickness  $< 1,0 \text{ mm}$  is considered to be a non-substantial component

Two or more non-substantial layers that are adjacent to each other (i.e. with no substantial component(s) in between the layers) are regarded as one non-substantial component when they collectively comply with the requirements for a layer being a non-substantial component.

**3.7**

**internal non-substantial component**

non-substantial component that is covered on both sides by at least one substantial component

**3.8**

**external non-substantial component**

non-substantial component that is not covered on one side by a substantial component

**3.9**

**heat of combustion**

thermal energy produced by combustion of unit mass of a given substance

NOTE It is expressed in joules per kilogram.

[EN ISO 13943:2000]

**3.10**

**gross heat of combustion, PCS (MJ/kg)**

heat of combustion of a substance when the combustion is complete and any produced water is entirely condensed under specified conditions

[EN ISO 13943:2000]

**3.11**

**net heat of combustion, PCI (MJ/kg)**

heat of combustion of a substance when the combustion is complete and any produced water is in the vapour state under specified conditions

NOTE The net heat of combustion may be calculated from the gross heat of combustion.

[EN ISO 13943:2000]

**3.12**

**latent heat of vaporisation of water (MJ/kg)**

heat which is required to change water from a liquid to a gas

## **4 Test apparatus**

### **4.1 General**

The test apparatus is illustrated in Figure 1, and is detailed in 4.2 to 4.12. All dimensions given in the following description are nominal values, unless tolerances are specified.

## 4.2 Calorimetric bomb

The calorimetric bomb shall be constructed as follows:

- a) volume:  $(300 \pm 50)$  ml;
- b) mass not greater than 3,25 kg;
- c) casing thickness at least 1/10 of the inner diameter of the body.

The lid is intended to receive the crucible and the electric firing device. The lid, including any seals, shall be capable of withstanding an internal pressure of 21 MPa.

NOTE These conditions define a bomb in which 1 g of coal under an initial oxygen pressure no greater than 3 MPa (pressure gauge method) is able to withstand, with a sufficient coefficient of safety, the maximum amount of pressure created under combustion, without a need for a calorimetric bomb of overlarge mass.

The inner surface of the bomb shall be resistant to attack by products of combustion and, even when "fuels" rich in sulphur are used, it shall resist pitting and inter-crystalline corrosion by acids produced during combustion.

## 4.3 Calorimeter

### 4.3.1 Jacket

The jacket shall be a double walled container, which is thermally insulated together with an insulated lid. The jacket is filled with water. The dimensions of the jacket shall be such that there is at least 10 mm space around the calorimetric vessel. The calorimetric vessel shall be supported on an area as small as possible of non-conducting material preferably a 3 point support.

For an adiabatic calorimeter system, a heater and thermometer system shall be incorporated into the vessel so that the water temperature in the jacket is maintained at the same temperature as the water in the calorimetric vessel.

For an isothermal calorimeter system, the temperature of the water in the jacket shall be kept constant. For an isothermal calorimeter, the necessary corrections shall be made (see 8.2).

### 4.3.2 Calorimetric vessel

This shall be a polished metal container designed to accommodate the bomb. The dimensions shall be such that the bomb can be immersed in water(see 7.3 d)).

### 4.3.3 Stirrer

A stirrer driven by a constant speed motor shall be provided. To prevent the transfer of heat to and from the calorimeter, the driving shaft of the stirrer shall have a thermally insulated section in a gasket between the jacket lid and the jacket. A magnetic stirring device with a similar performance is an acceptable alternative.

## 4.4 Temperature measuring device

The temperature measuring device shall be capable of giving a resolution of 0,005 K.

When using a mercury thermometer, this shall have at least 0,01 K graduations with a device e.g. a lens, for taking readings to within 0,005 K. A mechanical vibrator shall also be used to tap gently the thermometer to ensure the mercury column does not stick.

#### 4.5 The crucible

The crucible may be made of metal, such as platinum, nickel, stainless steel, or silica, with a flat base, 25 mm in diameter (maximum dimension if it is truncated) and 14 mm to 19 mm high. The following wall thickness is recommended:

Metal: 1,0 mm

Silica: 1,5 mm

NOTE Several shapes of crucible have proved satisfactory.

#### 4.6 Timing device

The timing device shall be capable of recording elapsed time to the nearest second and shall be accurate to within 1 s in 1 h.

#### 4.7 Electric power source

The voltage to the firing circuit shall not exceed 20 V for the firing. An ammeter shall be added to the circuit to indicate the breaking of the firing wire. A circuit breaker is a useful addition to the supply circuit.

#### 4.8 Pressure gauge and needle-valve

These shall be attached to the oxygen supply circuit to show the pressure in the bomb while it is being filled; this pressure shall be indicated with a resolution of 0,1 MPa.

#### 4.9 Balances

Two balances are required:

- an analytical balance with an accuracy of 0,1 mg.
- a balance with an accuracy of 0,1 g.

#### 4.10 Device for making the “cigarette”

The device and the procedure for making the “cigarette” are shown in Figure 2. The device for producing the “cigarette” comprises a mould and a metallic mandrel (not aluminium).

#### 4.11 Device for making the pellet

If prefabricated pellets are not available, a suitable device for making the pellet shall be used.

#### 4.12 Reagents

4.12.1 Distilled or de-mineralised water.

4.12.2 Pressurized oxygen, free from any other combustible product (purity  $\geq 99,5$  %).

NOTE Oxygen prepared by electrolysis may contain a small percentage of hydrogen, which makes it unsuitable for this use.

**4.12.3** Powder or pellet of benzoic acid “reference standard for calorimetry” whose gross heat of combustion is guaranteed.

**4.12.4** Combustion aid, with a known heat of combustion e.g. paraffin oil.

**4.12.5** Cigarette making paper, which is pre-glued and of minimum dimensions 55 mm × 50 mm with a known heat of combustion.

NOTE A commercially available cigarette making paper of 55 mm x 100 mm has been found suitable when cut into two equal pieces.

**4.12.6** Firing wire made of pure iron shall be 0,1 mm in diameter e.g. piano wire. Other types of metal wire (e.g. platinum, nickel or chromium) may be used, provided that they break under their own tension when the switch is closed on the firing circuit and the exact heat of combustion for the wire is known. When using a metal crucible (see 4.5), there shall be no contact between the firing wire and the crucible. It is therefore advisable to wrap the metal wire with a cotton thread.

**4.12.7** A thread made of white cellulosic cotton (see 4.12.6).

## 5 Test specimen

### 5.1 General

A product shall be evaluated through each of its components, taking into account the rules for non-substantial components. If a non-homogeneous product cannot be delaminated, its components shall be provided separately. A product can be delaminated when it is possible to separate one component from another without any part of the other component adhering to the component to be evaluated.

### 5.2 Sampling

#### 5.2.1 General

From a representative amount of a homogeneous product, or a component of a non-homogeneous product, compose a sample from at least five randomly selected parts taken from across the thickness. A minimum mass of 50 g shall be taken from a homogeneous product and a substantial component of a non-homogeneous product. A minimum mass of 10 g shall be taken for a non-substantial component of a non-homogeneous product.

#### 5.2.2 Loose-fill material

A sample shall be taken at random from the product of a minimum mass of 50 g.

#### 5.2.3 Liquid applied products

A sample of a minimum mass of 10 g of dried material shall be prepared.

### 5.3 Determination of surface density

Where required, the surface density of each component of a product shall be determined to an accuracy of  $\pm 0,5$  % from a minimum area of 250 mm × 250 mm.

For liquid applied products, the dried weight shall be determined.

### 5.4 Grinding

The samples as defined in 5.2 shall be reduced gradually to provide the final test sample. Grinding shall be carried out in such a way that no thermal decomposition takes place. Grind the sample and reduce it with a method of cross reduction, grinding to a finer powder as reduction proceeds.

If the sample cannot be ground, reduce it by any appropriate method into small granules or pieces and treat the specimens obtained as a powder.

### 5.5 Type of specimen

Each time a fine powder can be obtained by grinding (see 5.4) the test specimen shall be prepared using the crucible method (see 5.8). If a fine powder cannot be obtained by grinding and/or when a complete combustion cannot be obtained when using the crucible method, the test specimen shall be prepared using the "cigarette" method (see 5.9).

### 5.6 Number of test specimens

Three test specimens shall be tested following the procedure in 7.3. Two additional test specimens shall be tested if the requirements for validity of test results are not met (see clause 10). More than three specimens may be tested as required for any classification system.

### 5.7 Determination of mass

Weigh to the nearest 0,1 mg the various elements:

- 0,5 g material;
- 0,5 g benzoic acid;
- firing wire, cotton thread and cigarette paper if necessary.

NOTE 1 For some products with a high heat of combustion, the combustion aid may be reduced or excluded.

NOTE 2 For some materials with a low heat of combustion, it may be necessary to increase the gross heat of combustion of the specimen in order to obtain complete combustion by changing the mass ratio between the material and the benzoic acid from 1:1 to 1:2, or by adding a combustion aid.

### 5.8 Crucible method

The procedure shall be carried out as follows (see Figure 3):

- a) Insert the previously weighed mixture of specimen and benzoic acid into the crucible.
- b) Connect the previously weighed firing wire to the two electrodes.
- c) Loop down the firing wire to touch the powder in the crucible.

### 5.9 "Cigarette" method

The procedure shall be carried out as follows (see Figure 2):

- a) Place a previously weighed firing wire down the centre of the mandrel.
- b) Wrap the previously weighed "cigarette" paper around the mandrel and glue the two overlapping edges together. No additional glue shall be used since the "cigarette" paper is pre-glued. Sufficient paper shall be left free at each end to allow this to be twisted around the firing wire.
- c) Twist the paper around the firing wire at the lower end of the mandrel and insert the whole assembly into the mould. The firing wire shall project through the bottom of the mould.

NOTE A clearance of 0,5 mm between the mandrel and the mould should allow easy positioning of the assembly.

- d) Remove the mandrel.
- e) Insert the previously weighed mixture of specimen and benzoic acid into the "cigarette" paper.
- f) Remove the filled "cigarette" from the mould and twist together the ends of the paper to seal the "cigarette".
- g) Weigh the "cigarette" to ensure the total mass does not vary from the masses of the constituents by more than 10 mg.
- h) Put the "cigarette" into the crucible.
- i) Connect the firing wire to the two electrodes.

## 6 Conditioning

The powdered specimen, the benzoic acid and the cigarette paper shall be conditioned before test according to EN 13238.

## 7 Test procedure

### 7.1 General

Carry out the test in a room under normal laboratory conditions. It is recommended that the room temperature remains stable. For manual apparatus, the difference between the room temperature and the vessel water temperature shall not vary by more than  $\pm 2$  K.

### 7.2 Calibration procedure

#### 7.2.1 Determination of the water equivalent

The water equivalent  $E$  (MJ/K) of the calorimeter, the bomb and their accessories shall be determined by making at least five determinations of the gross heat of combustion of pellets of 0,4 g to 1,0 g of certified benzoic acid .

The procedure shall be carried out as follows:

- a) Compress the previously weighed powder of benzoic acid, using a pellet-making machine, to make a pellet or take a prefabricated pellet. Prefabricated certified pellets of benzoic acid may be used under the condition that the certified gross heat of combustion of the pellets is the same as the certified gross heat of combustion of the powder used for the tests.
- b) Weigh the pellet, to the nearest 0,1 mg
- c) Put the pellet into the crucible.
- d) Connect the firing wire to the two electrodes.
- e) Loop down the previously weighed firing wire to touch the pellet.

The test shall be carried out as specified in 7.3. The water equivalent  $E$  expressed in MJ/K shall be the average of the five determinations. Each individual result shall not deviate more than 0,2 % from the water equivalent  $E$ .

### 7.2.2 Conditions for re-calibration

The procedure given in 7.2.1 shall be carried out at regular intervals, not greater than two months, or when any significant part of the system is changed.

### 7.3 Standard test procedure

- a) Check that a good electrical contact is ensured between the two electrodes and the firing wire.

NOTE 1 1 ml de-ionized water may be added to absorb any acid gases produced

- b) Adjust the lid and tighten onto the body of the bomb. Connect the bomb to the bottle of oxygen then carefully open the bottle's tap and fill the bomb until a pressure of 3,0 MPa to 3,5 MPa is achieved without removing the air already there.

- c) Place the bomb in the calorimeter vessel.

- d) Introduce into the calorimeter vessel a quantity of distilled or de-mineralized water sufficient to cover the upper surface of the bomb cap and weigh. This quantity of water shall be the same, to the nearest 1g, as that used in the calibration procedure (see 7.2.1).

- e) Check that the bomb does not leak (no bubbles).

- f) Place the calorimeter vessel in the water jacket.

- g) Proceed as follows:

- i) Set the temperature measuring device and start the stirrer and the timing device.
- ii) Bring the water in the calorimeter vessel to a temperature approximately equal to that of the jacket. Note the temperature of the water in the calorimeter vessel at least every minute until successive readings are identical within  $\pm 0,01$  K for at least 10 min. Note this temperature as the initial temperature, ( $T_i$ ).
- iii) Close the electric circuit to cause combustion.
- iv) For adiabatic calorimeter only: during the rapid temperature rise phase of the water in the calorimetric vessel, the temperature of the water in the jacket shall be maintained as close as possible to that of the calorimetric vessel; the two temperatures shall be to within  $\pm 0,01$  K as they get nearer to the maximum temperature. Note the temperature of the water in the calorimetric vessel at least every minute until successive readings are identical within  $\pm 0,01$  K for at least 10 min. Note this temperature as the maximum temperature ( $T_m$ ).

NOTE 2 These processes may be automated in designs of commercially available equipment.

- h) Remove the bomb from the calorimeter, leave to stand for ten minutes then slowly reduce the pressure. Open the bomb. Verify that complete combustion has taken place i.e. that there is neither sooty deposit inside the bomb nor traces of residual carbon on the sides of the crucible. Rinse and dry the bomb.

- i) If complete combustion has not occurred when using the 'crucible' method, re-test using the "cigarette" method. If complete combustion has not occurred, when using the "cigarette" method, re-test.



## 8 Expression of results

### 8.1 Corrections for manual apparatus

Correct all the temperatures observed according to the calibration certificate of the thermometer and taking account of the exposed part of the thermometer stem.

### 8.2 Corrections for isothermal calorimeter (see annex C).

Correction of the temperature is necessitated by the exchange of heat with the exterior (see NOTES 1, 2 and 3).

This correction is given by the following formula:

$$c = (t - t_1) \times T_2 - t_1 \times T_1 \quad (1)$$

where:

$t$  = time passed in minutes and fractions of a minute from the start of the main period (see Figure 4) until the time when the maximum temperature is reached.

This moment is determined by finding the average of the times at which the temperature stops increasing and starts decreasing.

$t_1$  = time passed in minutes and fractions of a minute from the start of the main period (see Figure 4) until the moment when the temperature increase is 6/10 of the total temperature spread ( $T_m - T_i$ ) (see 8.3).

This moment is calculated by interpolation between the two temperature readings nearest to each other.

$T_2$  = average temperature drop per minute of the final period (see Figure 4).

$T_1$  = average rise in temperature per minute of the preliminary period (see Figure 4).

This difference normally corresponds to an overheating of the calorimeter.

NOTE 1 This is nil if an adiabatic jacket is used.

NOTE 2 This is nil if an automatic correction is made with an automatic apparatus.

NOTE 3 A graphical method for the calculation of this term is given in annex C.

### 8.3 Calculation of the gross heat of combustion of the specimen

The calculation of the gross heat of combustion under constant volume, of the test specimen is given by the following formula expressed in megajoules per kilogram.

With automatic apparatus, gross heat of combustion is directly obtained as the result of the test.

$$PCS = \frac{E(T_m - T_i + C) - b}{m} \quad (2)$$

where:

$PCS$  = gross heat of combustion in MJ/kg

$E$  = water equivalent of the calorimeter, the bomb, their accessories and of the water introduced into the bomb, expressed in MJ/K (see 7.2)

$T_i$  = initial temperature in K.

$T_m$  = maximum temperature in K.

$b$  = correction expressed in MJ required for the combustion heat of the "fuels" used during the test; i.e. firing wire, cotton thread, cigarette paper and benzoic acid or combustion aid.

Unless a guaranteed value is given for the cotton thread and the cigarette paper and/or the combustion aid, their gross heats of combustion shall be measured. The test specimen shall be prepared as specified in 5.8 and the test carried out as specified in 7.3.

Unless a guaranteed value is given for the firing wire, the gross heat of combustion to be considered shall be:

- nickel-chrome = 1,403 MJ/kg
- platinum = 0,419 MJ/kg
- pure iron = 7,490 MJ/kg

$c$  = temperature correction expressed in K required for the exchange of heat with the outside (see 8.2). This is nil if an adiabatic jacket is used

$m$  = mass of the test specimen in kg.

## 8.4 Calculation of the gross heat of combustion of the product

### 8.4.1 General

For a product or a component showing an endothermic reaction a negative *PCS* value will be obtained.

When calculating the *PCS* of a product the following procedure is adopted.

Firstly, establish the *PCS* of the individual components of a non-homogeneous product or of the *PCS* of a homogeneous product. If any of the three results are negative, these shall be reported and the mean value calculated from the actual values, e.g. if the following three results are obtained

- 0,3
- 0,4
- + 0,1

the mean value is - 0,2.

For a homogeneous product, this value is recorded as the *PCS* of the product. For a non-homogeneous product, the mean *PCS* values for each of the components are considered. Any negative *PCS* values of individual components are set to zero for the purpose of calculating the total *PCS* of the product. Metallic components shall not be tested and their gross heat of combustion used to calculate total *PCS* shall be zero.

Example: if there are four components and the following mean values have been obtained

- 0,2
- 15,6
- 6,3
- 1,8

any negative values are now set to zero i.e.

- 0
- 15,6
- 6,3
- 0

and the *PCS* of the product is then calculated from these values.

## 8.4.2 Homogeneous product

**8.4.2.1** For an individual sample (see 5.2.1) three test specimens are evaluated. If the spread of the individual values complies with the criteria given in clause 10, the test is valid and the gross heat of combustion is the mean of these three individual values.

**8.4.2.2** If the range of values determined on these three test specimens does not comply with the criteria given in clause 10, then two further test specimens shall be taken from the same sample and evaluated. The maximum and minimum values of these five results are then discarded and the final three test results are evaluated as described in 8.4.2.1.

**8.4.2.3** If the range of values obtained from 8.4.2.2 does not meet the requirements for range of validity as described in 8.4.2.1, then a new test sample shall be obtained and the whole procedure repeated.

**8.4.2.4** If two further test specimens (after the initial three) are needed for the purpose of any classification procedure and two further test specimens are needed as described in 8.4.2.2, then the same two test specimens are used for both purposes, i.e. a maximum of five specimens are tested.

## 8.4.3 Non-homogeneous product

The gross heat of combustion of the non-homogeneous product shall be determined as follows:

a) determine the gross heat of combustion of each individual component as for a homogeneous product (see 8.4.2). The gross heat of combustion shall be expressed in both MJ/kg and MJ/m<sup>2</sup> using the area weight of each individual component (see 5.3).

b) calculate the gross heat of combustion of the non-homogeneous product using the gross heat of combustion (see 8.4.2) and the area weight of each individual component (see 5.3).

An example for determination of the gross heat of combustion of a non-homogeneous product is given in annex D.

## 9 Test report

The test report shall include the following information as a minimum. A clear distinction shall be made between the data provided by the sponsor and data determined by the test.

- a) reference that the test was carried out in accordance with EN ISO 1716;
- b) any deviations from the test method;
- c) name and address of the testing laboratory;
- d) date and identification number of the report;
- e) name and address of the sponsor;
- f) name and address of the manufacturer/supplier, if known;
- g) date of sample arrival;
- h) identification of the product;
- i) description of the sampling procedure, where relevant;
- j) a general description of the product tested including density, mass per unit area and thickness, together with details of the construction of the product;

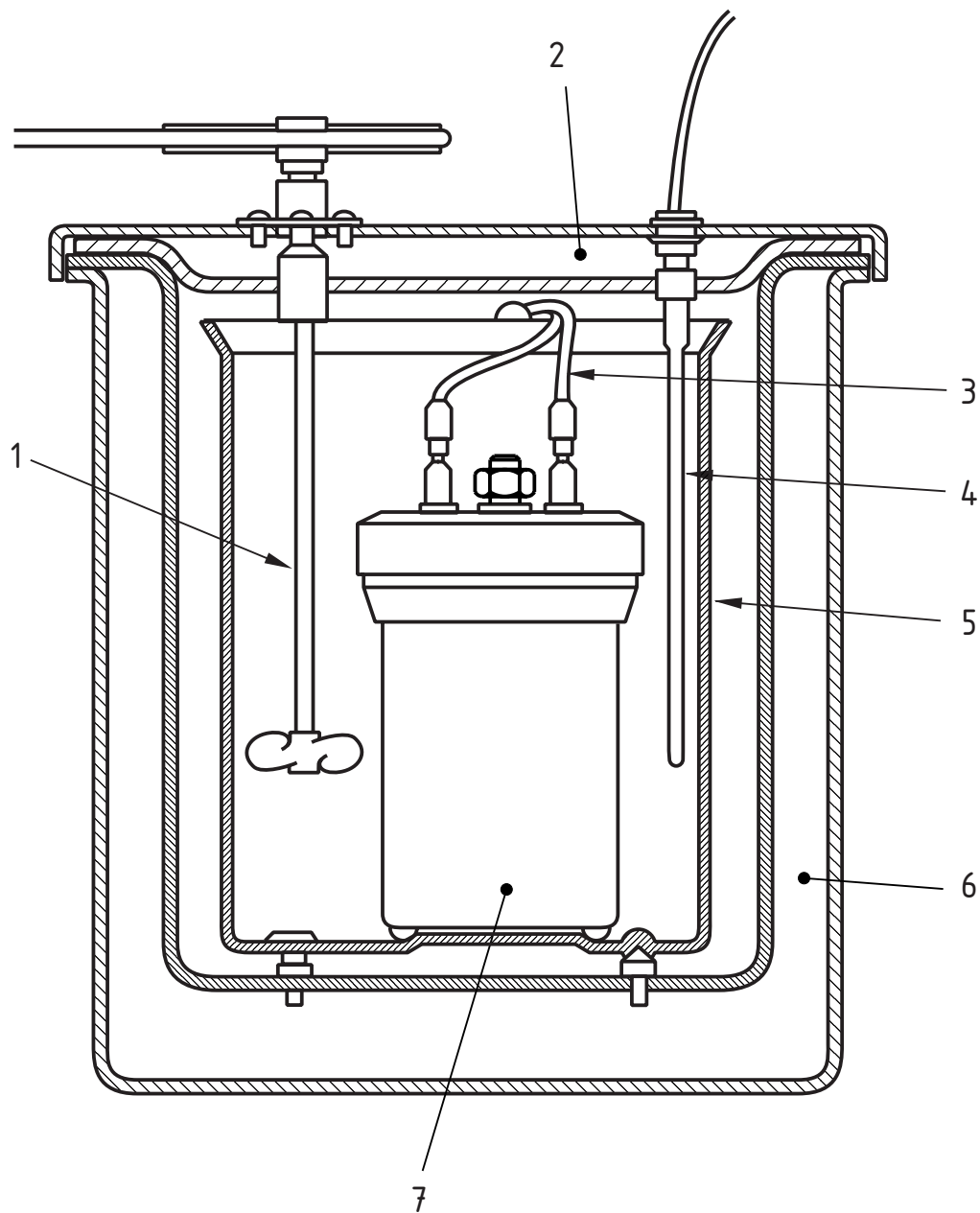
- k) details of conditioning;
- l) date of test;
- m) water equivalent expressed in accordance with 7.2;
- n) test results expressed in accordance with clause 8;
- o) observations made during the test;
- p) the statement 'The test results relate to the behaviour of the test specimens of a product under the particular conditions of the test; they are not intended to be the sole criterion for assessing the potential fire hazard of the product in use'.

## 10 Validity of test results

To be validated, test results shall comply with the following criteria in the specified range of values:

**Table 1 — Criteria for the validity of test results**

<b>Gross heat of combustion</b>	<b>Max – Min of the 3 Replicated tests</b>	<b>Range of validity</b>
<i>PCS</i> (MJ/kg)	$\leq 0,2$ MJ/kg	From 0 MJ/kg to 3,2 MJ/kg
<i>PCS</i> (MJ/m <sup>2</sup> ) <sup>a)</sup>	$\leq 0,1$ MJ/m <sup>2</sup>	From 0 MJ/m <sup>2</sup> to 4,1 MJ/m <sup>2</sup>
<sup>a)</sup> for non-substantial components only.		



**Key**

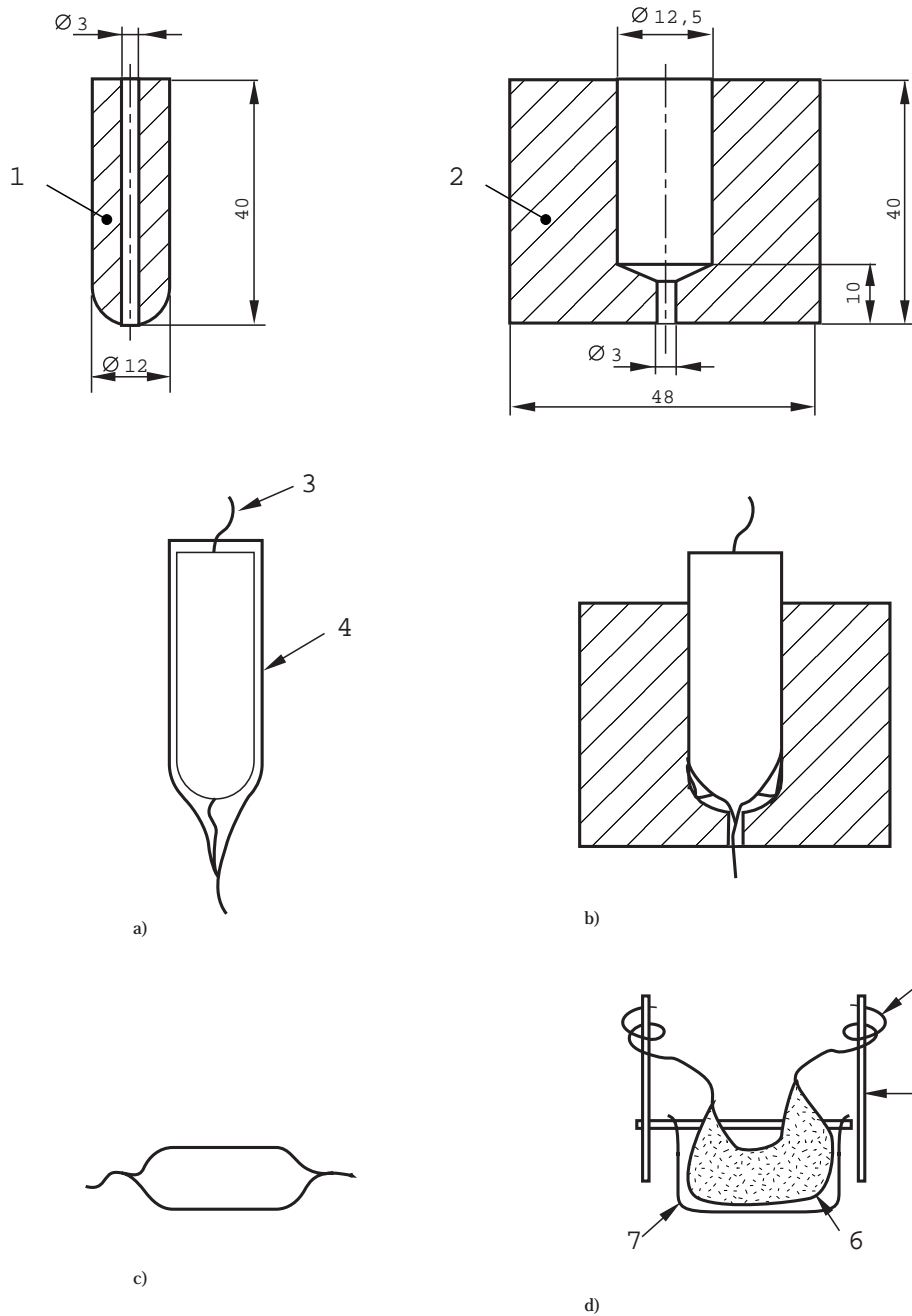
- 1 Stirrer
- 2 Jacket lid
- 3 Ignition leads

- 4 Thermometer
- 5 Calorimetric vessel
- 6 Jacket

- 7 Calorimetric bomb

**Figure 1 — Test apparatus**

Dimensions in mm



a) Shaping the paper over the mandrel. Kept in place by gluing an overlap of the paper using the pre-glued cigarette paper

b) Paper in position in the mould after the mandrel has been removed, ready to be filled

c) Cigarette completed. The two ends of the paper are twisted.

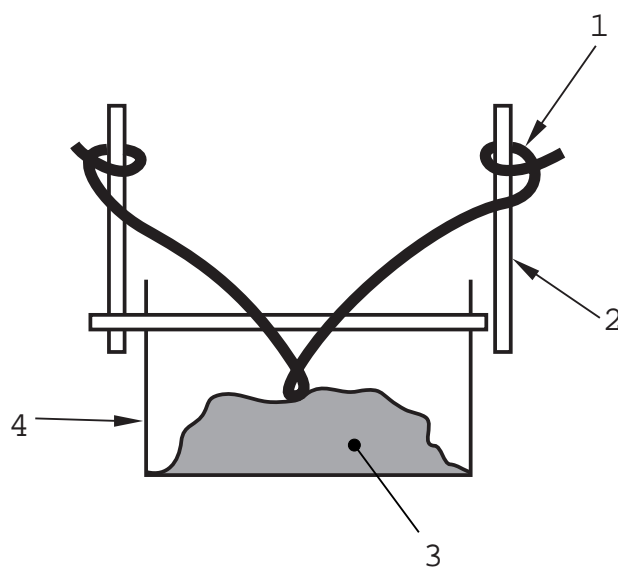
d) The "cigarette" is put in the crucible and the firing wire is wrapped tightly around the line of the electrodes

1 Mandrel  
2 Mould  
3 Firing wire

4 Paper  
5 Electrode  
6 Cigarette

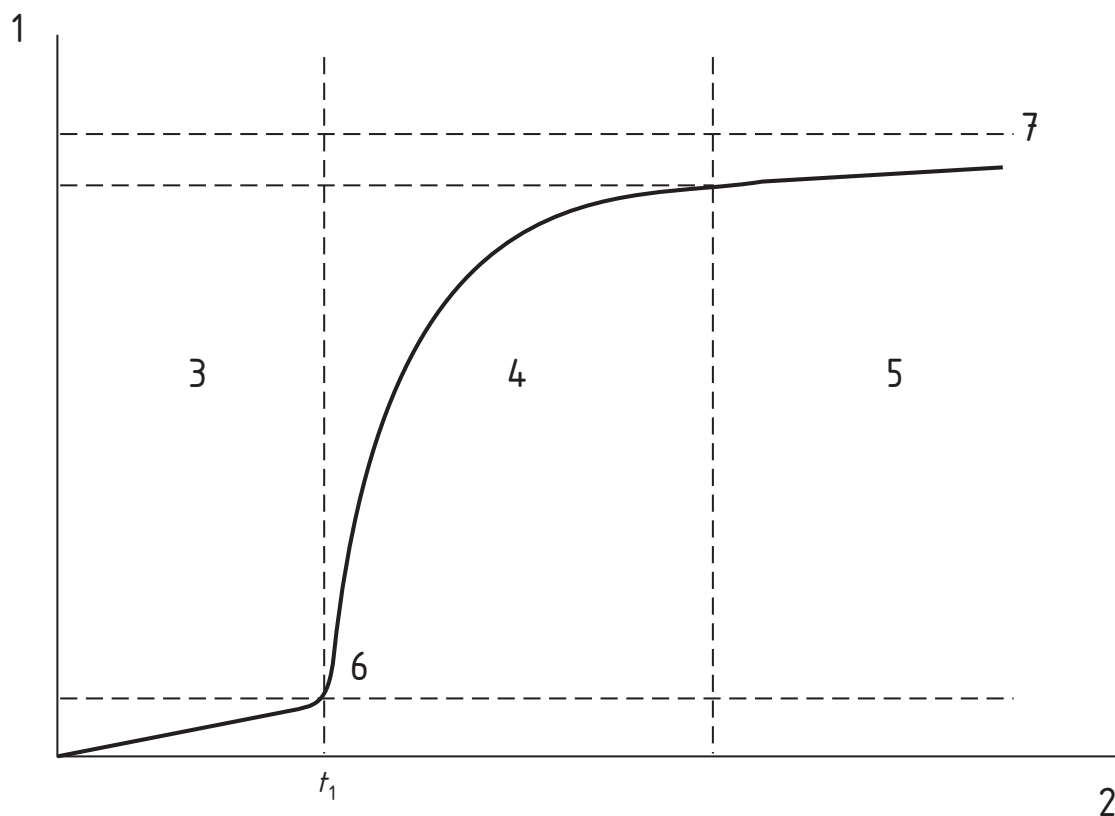
7 Crucible

Figure 2 — Method for preparing the "cigarette"

**Key**

- 1 Firing wire
- 2 Electrodes
- 3 Mixture of benzoic acid and product
- 4 Crucible

**Figure 3 — Test specimen preparation with crucible method**



**Key**

- 1 Temperature
- 2 Time
- 3 Preliminary period
- 4 Main period
- 5 Final period
- 6 Ignition
- 7 T (jacket)

**Figure 4 — Temperature-time curve**



## Annex A (normative)

### Calculation of net heat of combustion

The net heat of combustion ( $PCI$ ) is the difference between the gross heats of combustion ( $PCS$ ) and the latent heat of vaporization of the condensed water ( $q$ )

i.e.  $PCI = PCS - q$

The amount of condensed water in the bomb after combustion is determined by special tests, using analysis equipment for the measurement of the hydrogen content. A specimen of powder is prepared and conditioned as described in clauses 5 and 6.

The number of tests shall be as for the determination of the gross heat of combustion.

The content of condensed water  $w$  is given by the mean of the three results obtained.

The latent heat of vaporization  $q$  of the condensed water in the bomb is obtained as follows

$q = 2\,449 w$ .

## Annex B (informative)

### Precision of test method

A round robin exercise was conducted by CEN/TC 127. The protocol used was functionally the same as described in this standard. The products tested in this round robin were as described in Table B.1.

**Table B.1 — Products included in the round robin exercise**

Product	Density (kg/m <sup>3</sup> )	Thickness (mm)	area weight (g/m <sup>2</sup> )
Stone wool	145	50	
Wood fibre board	50		
Gypsum fibre board	1100	25	
Phenolic foam		40	
FR cellulose loose fill	30	-	
Paint			145
PVC/Nitrile rubber (12,9% chlorine)	65		1 235
Acoustical mineral fibre tiles	wool:220	18	
* Painted glass mat			413,1
* Stone wool			4 085
Paper faced gypsum plaster board	700	12,5	
* Paper (dark colour)			220
* Gypsum			8 700
* Paper (light colour)			230
Faced glass wool	80	15	
* Painted glass mat			313,2
* Glass wool			1 092,8
* Glass fleece			55,4

Values of statistical means ( $m$ ), standard deviation ( $S_r$  and  $S_R$ ), repeatability ( $r$ ) and reproducibility ( $R$ ) at 95 % confidence level were calculated according to ISO 5725-2<sup>1)</sup> (Table B.2) for the two parameters  $PCS$  with the crucible method in MJ/kg and  $PCS$  with the cigarette method in MJ/kg. Such values for  $r$  and  $R$  are equal to 2,8 times the appropriate standard deviation. The values include results identified as 'stragglers' but excludes results identified as "outliers".

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1) ISO 5725-2: 1994 Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

Table B.2 — Statistical results of the round robin exercise

	Statistical Mean ( <i>m</i> )	Standard deviation ( <i>S<sub>r</sub></i> )	Standard Deviation ( <i>S<sub>R</sub></i> )	<i>r</i>	<i>R</i>	<i>S<sub>r</sub>/m</i>	<i>S<sub>R</sub>/m</i>
<i>PCS</i> (MJ/kg) Crucible	From -0,32 To 24,82	from 0,04 to 0,35	From 0,07 to 1,13	from 0,12 to 0,98	from 0,19 to 3,16	From 0,17 % to 21,3 %	From 2,72 % to 60,40 %
<i>PCS</i> (MJ/kg) Cigarette	From -0,31 to 25,18	from 0,03 to 0,34	From 0,09 to 1,17	from 0,10 to 0,95	from 0,25 to 3,27	from 0,37 % to 23,41 %	from 3,16 % to 70,40 %

It was possible for the two methods to obtain linear models for *S<sub>r</sub>*, *S<sub>R</sub>*, *r* and *R*. The coefficients are presented in Table B.3. The graphic for *PCS* with the crucible method is presented as an example in Figure B.1. For the repeatability of *PCS*, the results lead to models which are more or less meaningless even if they are statistically correct. More complicated models than simple linear models could better fit to these parameters but this was not considered in this round robin exercise.

Table B.3 — Statistical models of the round robin exercise

Parameters	<i>S<sub>r</sub></i>	<i>S<sub>R</sub></i>	<i>R</i>	<i>R</i>
<i>PCS</i> crucible MJ/kg	$= 0,07 - 0,0004 \times PCS$	$= 0,09 + 0,0287 \times PCS$	$= 0,20 - 0,0012 \times PCS$	$= 0,26 + 0,0804 \times PCS$
<i>PCS</i> cigarette MJ/kg	$= 0,05 + 0,0041 \times PCS$	$= 0,12 + 0,0328 \times PCS$	$= 0,15 + 0,0114 \times PCS$	$= 0,34 + 0,0918 \times PCS$

When the models correctly fit to the parameters, they may be a tool to “predict” a result. This can be illustrated by means of an example. Suppose a laboratory tests a single specimen of a given product and determines that the *PCS* with the crucible method is 1,57 MJ/kg. If the same laboratory conducts a second test on the same product, the value of *r* is evaluated as:

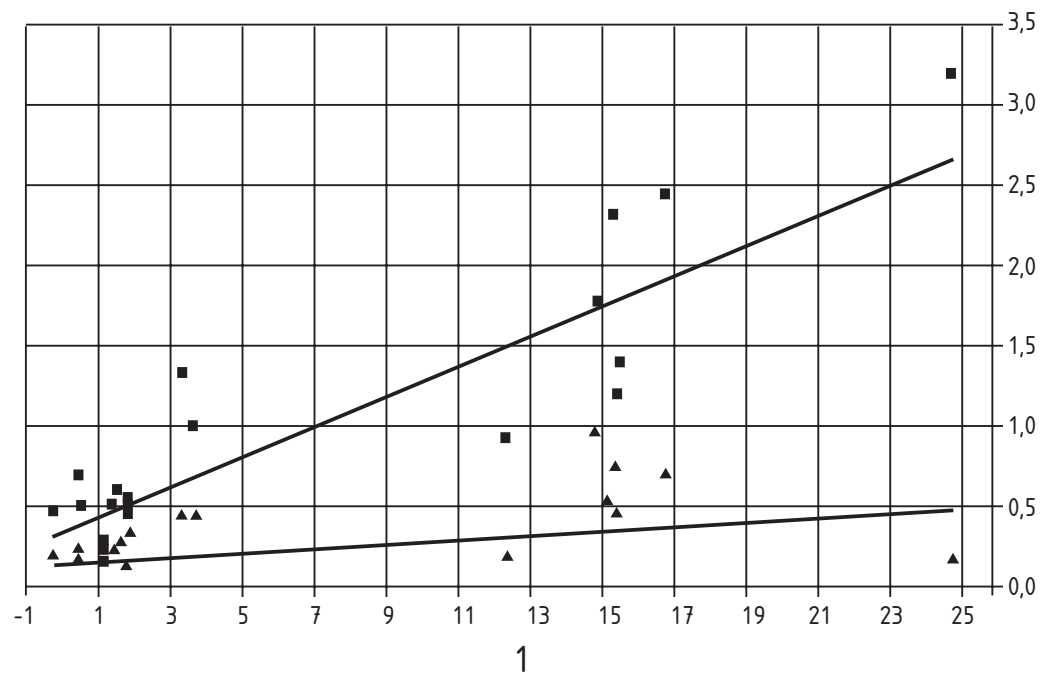
$$r = 0,20 - 0,0012 \times 1,57 \approx 0,20 \text{ MJ/kg}$$

Then the probability is 95 % that the result of the second test will fall between 1,77 MJ/kg and 1,37 MJ/kg.

Suppose now that the same product is tested by a different laboratory. The value *R* is evaluated as

$$R = 0,26 + 0,0804 \times 1,57 \approx 0,39 \text{ MJ/kg}$$

Then the probability is 95 % that the results from the test at that laboratory will fall between 1,18 MJ/kg and 1,96 MJ/kg.



- ▲ 2
- 3
- 4
- 5

**Key**

- 1 Estimated mean  $m$
- 2 Model  $r$
- 3 Model  $R$
- ▲  $R$
- $R$

**Figure B.1 — Statistical model for PCS with the crucible method in MJ/kg**

## Annex C (informative)

### Calculation by graph of the corrective term “c” necessary because of the cooling of the calorimeter

$T$  is the temperature of the calorimeter and  $t$  the time. Designate the temperature of the outside air, near to the calorimeter, presumed constant for the duration of the experiment, as  $T_0$ .  $T$  increases from the initial value  $T_1$  at the beginning of the experiment to a final value of  $T_2$  always greater than  $T_1$ . During each moment of time  $dt$ , the calorimeter undergoes a positive or negative  $dc$  cooling from the outside tied to the temperature by the Newton relation :

$$c = a.(T - T_0) dt \quad (C.1)$$

with  $a$  being a constant for a given calorimeter (cooling constant); the temperature correction for heat exchange with the outside between the start of the main period  $t_1$  and the moment  $t_m$  when the maximum temperature is reached, is given by the integral;

$$dc = \alpha \int_{t_1}^{t_m} (T - T_0) dt \quad (C.2)$$

To calculate the integral,  $a$  and  $T_0$  has to be known. At the end of the preliminary period (moment 1) and at the end of the final period (moment 2) the variations in temperature of the calorimeter are approximately linear and correspond to the exchanges with the outside. Measurement of these variations therefore gives:

$$\left[ \frac{dc}{dt} \right] \text{ at moments 1 and 2}$$

We can therefore write:

$$\left[ \frac{dc}{dt} \right]_1 = \alpha(T_1 - T_0) \quad (C.3)$$

$$\left[ \frac{dc}{dt} \right]_2 = \alpha(T_2 - T_0) \quad (C.4)$$

This system of equations provides the values of  $a$  and  $T_0$  as a function of  $T_1$  and  $T_2$

$$\left[ \frac{dc}{dt} \right]_1 \text{ and } \left[ \frac{dc}{dt} \right]_2$$

The integral (C.2) can then be evaluated by a graph(see Figure 4). It is sufficient to plot the temperature curve as a function of time between moments  $t_1$  and  $t_m$  and the horizontal straight line with ordinate  $T_0$ . The difference between the hatched areas A1 and A2 situated above and below the line of ordinate  $T_0$  multiplied by the cooling constant  $a$  represents the corrective term  $C$ .

## Annex D (informative)

### Example of determination of the gross heat of combustion of a non-homogeneous product

#### D.1 Non-homogeneous product to be tested

Consider a non-homogeneous product constituted of substantial components and internal and external non-substantial components as defined in clause 3 (see Figure D.1).



#### Key

- 1 External non-substantial component
- 2 Substantial component
- 3 Internal non-substantial component
- 4 Substantial component

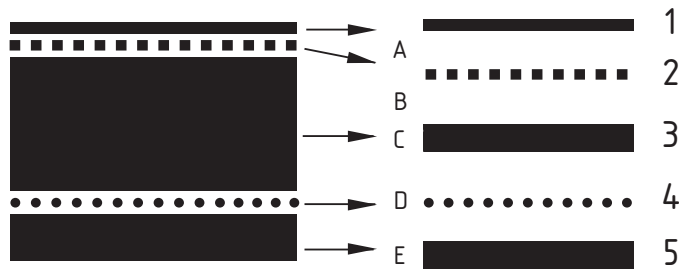
**Figure D.1 — Non-homogeneous product to be evaluated**

#### D.2 Sampling of the non-homogeneous product

##### D.2.1 Delamination of the product

The product is evaluated through each of its constituting components. Each component is obtained by delamination or is provided separately.

The samples are taken as described in 5.2 and each sample is ground as specified in 5.4.



One product	Five components	Five samples
1	Minimum 0,5 m <sup>2</sup> and minimum 10,0 g	
2	Minimum 10,0 g of dried material (glue)	
3	Minimum 0,5 m <sup>2</sup> and minimum 50,0 g	
4	Minimum 10,0 g of dried material (glue)	
5	Minimum 0,5 m <sup>2</sup> and minimum 50,0 g	

**Figure D.2 — Sampling of the non-homogeneous product**

### D.2.2 Determination of the area weight of each component

The area weight of each of the five components, in kg/m<sup>2</sup>, is determined as specified in 5.3. The area weights of the five components A, B, C, D and E are:  $M_A$ ,  $M_B$ ,  $M_C$ ,  $M_D$ ,  $M_E$ . The area weight of the product is:  $M = M_A + M_B + M_C + M_D + M_E$ .

### D.3 Determination of the gross heat of combustion of each component

The gross heat of combustion of each component is determined as described in 7.3 i.e. 3 results for each component, in MJ/kg, are:

$PCS_{A1}$	$PCS_{B1}$	$PCS_{C1}$	$PCS_{D1}$	$PCS_{E1}$
$PCS_{A2}$	$PCS_{B2}$	$PCS_{C2}$	$PCS_{D2}$	$PCS_{E2}$
$PCS_{A3}$	$PCS_{B3}$	$PCS_{C3}$	$PCS_{D3}$	$PCS_{E3}$

These results are analyzed for each component as described in clause 8 and further tests carried out if required, giving a mean value for each component:

— in MJ/kg:  $PCS_A$ ,  $PCS_B$ ,  $PCS_C$ ,  $PCS_D$ ,  $PCS_E$

— in MJ/m<sup>2</sup>:  $PCS_{SA} = M_A \times PCS_A$ ,  $PCS_{SB} = M_B \times PCS_B$ ,  $PCS_{SC} = M_C \times PCS_C$ ,  $PCS_{SD} = M_D \times PCS_D$ ,  $PCS_{SE} = M_E \times PCS_E$

The gross heat of combustion of the external non-substantial component of the product in MJ/m<sup>2</sup> is

$$PCS_{S_{ext}} = PCS_{SA} + PCS_{SB}$$

## ISO 1716:2002(E)

The gross heat of combustion of the external non-substantial component of the product in MJ/kg is

$$PCS_{\text{ext}} = (PCS_{\text{SA}} + PCS_{\text{SB}}) / M_A + M_B$$

The gross heat of combustion of the product ( $PCS_S$ ) in MJ/m<sup>2</sup> is

$$PCS_S = PCS_{\text{SA}} + PCS_{\text{SB}} + PCS_{\text{SC}} + PCS_{\text{SD}} + PCS_{\text{SE}}$$

The gross heat of combustion of the product ( $PCS$ ) in MJ/kg is

$$PCS = PCS_S / M$$





**ISO 1716:2002(E)**

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**ICS 13.220.50; 91.100.01**

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