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Reaction to fire tests for products — Determination of the gross heat of combustion (calorific value)

Essais de réaction au feu de produits — Détermination du pouvoir calorifique supérieur (valeur calorifique)

Reference number
ISO 1716:2010(E)

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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 2.

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 document 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 Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

This third edition cancels and replaces the second edition (ISO 1716:2002), which has been technically revised.

Reaction to fire tests for products — Determination of the gross heat of combustion (calorific value)

WARNING — The attention of all persons concerned with managing and carrying out this test is drawn to the fact that fire testing may be hazardous and that there is a possibility that toxic and/or harmful gases may be evolved during the test. Operational hazards may also arise during the testing of specimens, such as the possibility of an explosion, and during the disposal of test residues.

WARNING — An assessment of all the potential hazards and risks to health should be made and safety precautions should be identified and provided. Written safety instructions should be issued. Appropriate training should be given to relevant personnel. Laboratory personnel should ensure that they follow written instructions at all times.

1 Scope

This International Standard specifies a method for the determination of the gross heat of combustion (Q_{PCS}) of products at constant volume in a bomb calorimeter.

Annex A describes the calculation of the net heat of combustion (Q_{PCI}) when required.

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

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.

ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 13943, *Fire safety — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943, and the following 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

EXAMPLE Metal, stone, timber, concrete, mineral wool with a uniformly dispersed binder and 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, and that has a mass/unit area $\geqslant 1{,}0\ \mathrm{kg/m^2}$ or a thickness $\geqslant 1{,}0$ mm

3.6
non-substantial component
material that does not constitute a significant part of a non-homogeneous product and that has a layer with a mass/unit area $< 1{,}0\ \mathrm{kg/m^2}$ and a thickness $< 1{,}0$ mm

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
calorific value (deprecated)
thermal energy produced by combustion of unit mass of a given substance

NOTE The heat of combustion is expressed in megajoules per kilogram.

[ISO 13943:2008]

3.10
gross heat of combustion
Q_{PCS}
heat of combustion of a substance when the combustion is complete and any produced water is entirely condensed under specified conditions

NOTE The gross heat of combustion is expressed in megajoules per kilogram.

3.11
net heat of combustion
Q_{PCI}
heat of combustion of a substance when the combustion is complete and any produced water is in the vapour state under specified conditions

NOTE 1 The net heat of combustion may be calculated from the gross heat of combustion.

NOTE 2 The net heat of combustion is expressed in megajoules per kilogram.

3.12
latent heat of vaporization of water
q
heat which is required to change water from a liquid to a gas

NOTE The latent heat of vaporization is expressed in megajoules per kilogram.

3.13
surface density
mass per unit area

NOTE The surface density is expressed in kilograms per square metre.

4 Principle

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 vaporization of water.

This is a test method for determining an absolute value of the heat of combustion for a product and it does not take into account any inherent variability of the product.

5 Test apparatus

5.1 General

The test apparatus (bomb calorimeter) shall be as illustrated in Figure 1, and as detailed in 5.2 to 5.5. Additional equipment shall be in accordance with 5.6 to 5.11.

5.2 Calorimetric bomb, constructed with the following characteristics

The calorimetric bomb shall be constructed as follows:

a) volume: (300 ± 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 sulfur are used, it shall resist pitting and inter-crystalline corrosion by acids produced during combustion.

5.3 Calorimeter

5.3.1 Jacket, consisting of 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 as small as possible area 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 such 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 9.2).

5.3.2 Calorimetric vessel, consisting of a polished metal container designed to accommodate the bomb. The dimensions shall be such that the bomb can be immersed in water (see 8.3.7).

5.3.3 Stirrer, driven by a constant-speed motor. 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.

5.4 Temperature measuring device, 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 gently tap the thermometer to ensure that the mercury column does not stick.

5.5 Crucible, 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.

NOTE 1 The following wall thickness is recommended:

- metal: 1,0 mm;
- silica: 1,5 mm.

NOTE 2 Several shapes of crucible have proved satisfactory.

5.6 Timing device, capable of recording the time elapsed to the nearest second and accurate to within 1 s in 1 h.

5.7 Electric power source, with the voltage to the firing circuit not exceeding 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.

5.8 Pressure gauge and needle-valve, 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.

5.9 Two balances, with the following characteristics:

- one is an analytical balance with an accuracy of 0,1 mg;
- the other is a balance with an accuracy of 0,1 g.

5.10 Device for making the “cigarette”, as shown in Figure 2.

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

5.11 Device for making the pellet.

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

6 Reagents and materials

6.1 Distilled or demineralized water.

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

WARNING — Oxygen prepared by electrolysis can contain a small percentage of hydrogen, which makes it unsuitable for this use.

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

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

6.5 Cigarette-making paper, which is preglued and of minimum dimensions 55 mm × 50 mm with a known heat of combustion.

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

6.6 Firing wire made of pure iron, 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 (5.5), 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.

6.7 Thread, made of white cellulosic cotton (see 6.6).

7 Test specimens

7.1 General

In order to assess a product, each of its components shall be evaluated, 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.

If two or more non-substantial layers are adjacent to each other, and when added together they comply with the definition for a substantial component, then each individual layer shall be tested separately and they shall be assessed together as substantial. The total calorific value of the adjacent layers, which are considered substantial, shall be calculated by adding together the relative percentage of the measured calorific value for each component (see Annex D).

If two or more non-substantial layers are adjacent to each other, and when added together they comply with the definition for a non-substantial component, then each individual layer shall be tested separately and they shall be assessed together as non-substantial. (see Annex D)

WARNING — Any aluminium or other metallic component of a product shall not be tested in the bomb calorimeter, with the risk of serious injury to the operator due to overheating and/or overpressure causing the bomb calorimeter to explode.

7.2 Sampling

7.2.1 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.

7.2.2 Loose-fill material

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

7.2.3 Liquid-applied products

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

The material shall be cured or dried in accordance with the manufacturer's instructions. Care should be taken when drying liquid-applied components due to the potential presence of solvents. The method of curing shall be described in the test report

7.3 Determination of surface density

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

For liquid-applied products, the dried mass shall be determined.

7.4 Grinding

The samples defined in 7.2 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.

In the case of homogeneous material which, when ground, clearly separates into components of different density, so that a 0,5 g sample of the product, when taken from the ground powder, is not representative of the original product with respect to the proportion of the materials present, reduce the sample by any appropriate method, e.g. by sawing the sample into thin discs or by cutting it with a knife into small pieces. If this preparation is not possible, testing shall be conducted on the individual ingredients used in the manufacture of that product. The individual PCS values for these ingredients shall be used together with the proportion by mass of the ingredients in the final product to calculate the overall PCS value for the product.

7.5 Type of specimen

If a fine powder can be obtained by grinding (see 7.4), the test specimen shall be prepared using the crucible method (see 7.9). If a fine powder cannot be obtained by grinding and/or a complete combustion cannot be obtained when using the crucible method, the test shall be conducted by using either the “cigarette” method (see 7.10) or the crucible method utilizing a combustion aid, e.g. paraffin oil.

7.6 Conditioning

The powdered specimen, the benzoic acid and the cigarette-making paper shall be conditioned before testing in accordance with EN 13238 or ISO 554.

7.7 Number of test specimens

Three test specimens shall be tested following the procedure in 8.3. Two additional test specimens shall be tested if the requirements for validity of test results are not met (see Clause 11). More than three specimens may be tested as required for any classification system.

7.8 Determination of mass

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

- 0,5 g of material;
- 0,5 g of benzoic acid;

- combustion aid;
- firing wire, cotton thread and cigarette-making paper, if necessary.

NOTE 1 For some products with a high heat of combustion, the combustion aid and/or benzoic acid can be reduced or excluded.

NOTE 2 For some materials with a low heat of combustion, it can 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, e.g. paraffin oil, and/or the benzoic acid can be reduced or excluded.

7.9 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.

NOTE Some automatic apparatus is supplied with a fixed firing wire. For these items of apparatus, loop down a previously weighed cotton thread to touch the powder in the crucible.

7.10 “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-making paper around the mandrel and glue the two overlapping edges together. No additional glue shall be used since the cigarette-making paper is preglued. 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 allows for easy positioning of the assembly.

d) Remove the mandrel.

e) Insert the previously weighed mixture of specimen and benzoic acid into the cigarette-making 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 that 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.

8 Test procedure

8.1 General

It is recommended that the test be conducted in a room where the temperature remains stable, within ±2 K. Calibration of the apparatus and subsequent testing should be conducted under similar conditions of temperature and pressure. For manual apparatus, the difference between the room temperature and the vessel water temperature shall not vary by more than ±2 K.

8.2 Calibration procedure

8.2.1 Determination of the water equivalent

The water equivalent E, expressed in megajoules per kelvin, 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 calibration 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 as an alternative to benzoic acid powder. The certified value provided shall be used in any calculation of the gross heat of combustion.

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 8.3. The water equivalent E, expressed in megajoules per kelvin, shall be the average of the five determinations. Each individual result shall not deviate by more than 0,5 % from the water equivalent E.

8.2.2 Conditions for recalibration

The procedure given in 8.2.1 shall be carried out at regular intervals, not greater than two months with continuous use of the apparatus, or when any significant part of the system is changed.

8.3 Standard test procedure

WARNING — Aluminium or other metallic components of a product shall not be tested in the bomb calorimeter at the risk of serious injury to the operator due to overheating and/or overpressure causing the bomb calorimeter to explode.

Switch on the apparatus at least 1 h before testing.

8.3.1 Place the specimen in the crucible.

8.3.2 Place the crucible in the holder.

8.3.3 Attach the firing wire and loop it to touch the specimen.

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

8.3.5 Place the holder in the body of the calorimetric bomb.

1 ml of de-ionized water may be introduced into the body of the calorimetric bomb to absorb any acid gases produced.

8.3.6 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 (if not achieved automatically), without removing the air already there.

8.3.7 Place the bomb in the calorimeter vessel.

8.3.8 Introduce into the calorimeter vessel a quantity of distilled or demineralized water that is sufficient to cover the upper surface of the bomb cap and weigh. This quantity of water shall be the same, to the nearest 1 g, as that used in the calibration procedure (see 8.2.1) (if not achieved automatically).

8.3.9 Check that the bomb does not leak (no continuous stream of bubbles).

8.3.10 Place the calorimeter vessel in the water jacket.

8.3.11 Proceed as follows:

a) Set the temperature-measuring device and start the stirrer and the timing device (if this is not achieved automatically).

b) Bring the water in the calorimeter vessel to a temperature that is 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 ±0,01 K for at least 10 min (if this is not achieved automatically). Note this temperature as the initial temperature (T_i).

NOTE 1 With some automatic apparatus, the manufacturer of the apparatus states that a shorter time than 10 min is sufficient. If this is the case, this reduced time may be used on condition that the manufacturer's information is checked and documented by the laboratory.

c) Close the electric circuit to cause combustion.

d) For an 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 ±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 ±0,01 K for at least 10 min. Note this temperature as the maximum temperature (T_m).

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

e) Remove the bomb from the calorimeter, leave to stand for 10 min, then slowly reduce the pressure. Open the bomb. Verify that complete combustion has taken place, i.e. that there is neither a sooty deposit inside the bomb nor traces of residual carbon on the sides of the crucible. Rinse and dry the bomb.

f) If complete combustion has not occurred, a different method of test specimen preparation could be tried. With some materials, a procedure of trial and error is necessary to find the best method.

9 Expression of results

9.1 Corrections for manual apparatus

Correct all the temperatures observed in accordance with the calibration certificate of the thermometer and taking account of the exposed part of the thermometer stem.

9.2 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 factor, c, is given by Equation (1):

$$c = \left(t - t_\mathrm{l}\right) \times T_2 - t_\mathrm{l} \times T_\mathrm{l} \tag{1}$$

where

t is the 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_l is the time passed, in minutes and fractions of a minute, from the start of the main period at temperature T_0 (see Figure 4) until the moment when the temperature increase is 6/10 of the total temperature range ($T_\mathrm{m} - T_\mathrm{i}$) (see 9.3); this moment is calculated by interpolation between the two temperature readings nearest to each other;

T_2 is the average temperature drop, in kelvins per minute, of the final period (see Figure 4);

T_l is the average rise in temperature, in kelvins per minute, of the preliminary period (see Figure 4).

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

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

NOTE 3 A graphical method for the calculation of c is given in Annex C.

9.3 Calculation of the gross heat of combustion of the specimen

The calculation of the gross heat of combustion, Q_PCS, under constant volume, of the test specimen is given by Equation (2), expressed in megajoules per kilogram.

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

$$Q_\mathrm{PCS} = \frac{E(T_\mathrm{m} - T_\mathrm{i} + c) - b}{m} \tag{2}$$

where

Q_PCS is the gross heat of combustion, in megajoules per kilogram;

E is the water equivalent of the calorimeter, the bomb, their accessories and of the water introduced into the bomb, expressed in megajoules per kilogram (see 8.2);

T_i is the initial temperature, in kelvins;

T_m is the maximum temperature, in kelvins;

b is the correction, expressed in megajoules, required for the combustion heat of the “fuels” used during the test, i.e. firing wire, cotton thread, cigarette-making paper and benzoic acid or combustion aid;

c is the temperature correction factor, expressed in kelvins, required for the exchange of heat with the outside (see 9.2); this is nil if an adiabatic jacket is used;

m mass of the test specimen, in kilograms.

Unless a guaranteed value is given for the cotton thread, the cigarette-making paper and/or the combustion aid, their gross heats of combustion shall be measured. The test specimen shall be prepared as specified in 7.9 and the test shall be carried out as specified in 8.3.

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
- nickel = 3,245 MJ/kg
- platinum = 0,419 MJ/kg
- pure iron = 7,490 MJ/kg

9.4 Calculation of the gross heat of combustion of the product

9.4.1 General

Metallic components shall not be tested. Where metallic components are present, their gross heat of combustion shall be deemed to be zero.

A layer which contains perforations may be considered as a non-substantial component, when the area under consideration, with respect to the definition stated above, is to be taken as the whole area (the overall area), including the area of the perforations.

For a product or a component showing an endothermic reaction, a negative Q_{PCS} value will be obtained.

When calculating the Q_{PCS} of a product, the following procedure is adopted.

Firstly, establish the Q_{PCS} of the individual components of a non-homogeneous product or of the Q_{PCS} of a homogeneous product. If any of the three results are negative, they shall be reported, and the mean value calculated from the actual values. For example, 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 Q_{PCS} of the product. For a non-homogeneous product, the mean Q_{PCS} values for each of the components are considered. Any negative Q_{PCS} values of individual components are set to zero for the purpose of calculating the total Q_{PCS} of the product.

For 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 Q_{PCS} of the product is then calculated from these values.

9.4.2 Homogeneous product

9.4.2.1 For an individual sample (see 7.2.1), three test specimens are evaluated. If the spread of the individual values complies with the criteria given in Clause 11, the test is valid and the gross heat of combustion is the mean of these three individual values.

9.4.2.2 If the range of values determined on these three test specimens does not comply with the criteria given in Clause 11, 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 9.4.2.1.

9.4.2.3 If the range of values obtained from 9.4.2.2 does not meet the requirements for the range of validity described in 9.4.2.1, then a new test sample shall be obtained and the whole procedure repeated.

9.4.2.4 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 9.4.2.2, then the same two test specimens are used for both purposes, i.e. a maximum of five specimens are tested.

9.4.3 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 in the same way as for a homogeneous product (see 9.4.2). The gross heat of combustion shall be expressed in both MJ/kg and MJ/m^2 using the mass per unit area of each individual component.

b) Calculate the gross heat of combustion of the non-homogeneous product using the gross heat of combustion (see 9.4.2) and the mass per unit area of each individual component.

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

10 Test report

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

a) a statement that the test was carried out in accordance with this International Standard (ISO 1716:2010);

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) description of method to cure material, where relevant;

m) date of test;

n) water equivalent, expressed in accordance with 8.2;

o) test results, expressed in accordance with Clause 9;

p) observations made during the test;

q) 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.”

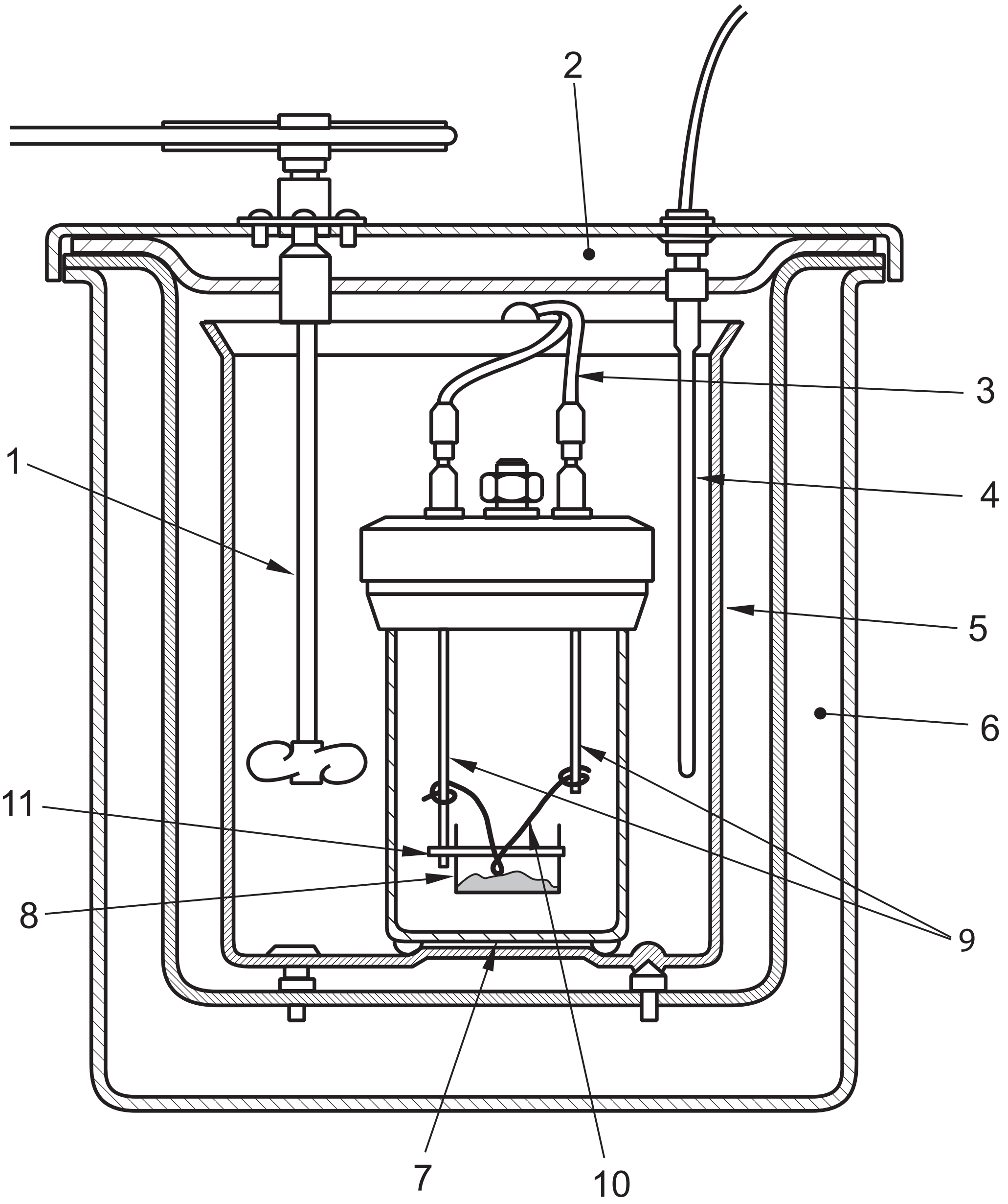
11 Validity of test results

To be validated, test results shall comply with the criteria in the specified range of values given in Table 1.

Table 1 — Criteria for the validity of test results

Gross heat of combustion	Max. and min. of the three replicated tests	Range of validity
Q_{PCS} (MJ/kg)	$\leqslant$ 0,2 MJ/kg	From 0 MJ/kg to 3,2 MJ/kg
	Within 5 %	From 3,2 MJ/kg to 20,0 MJ/kg
	Within 10 %	Greater than 20,0 MJ/kg
Q_{PCS} (MJ/m^2)[a]	$\leqslant$ 0,1 MJ/m^2	From 0 MJ/m^2 to 4,1 MJ/m^2
	Within 5 %	From 4,1 MJ/m^2 to 20 MJ/m^2
	Within 10 %	Greater than 20 MJ/m^2

[a] For non-substantial components only.

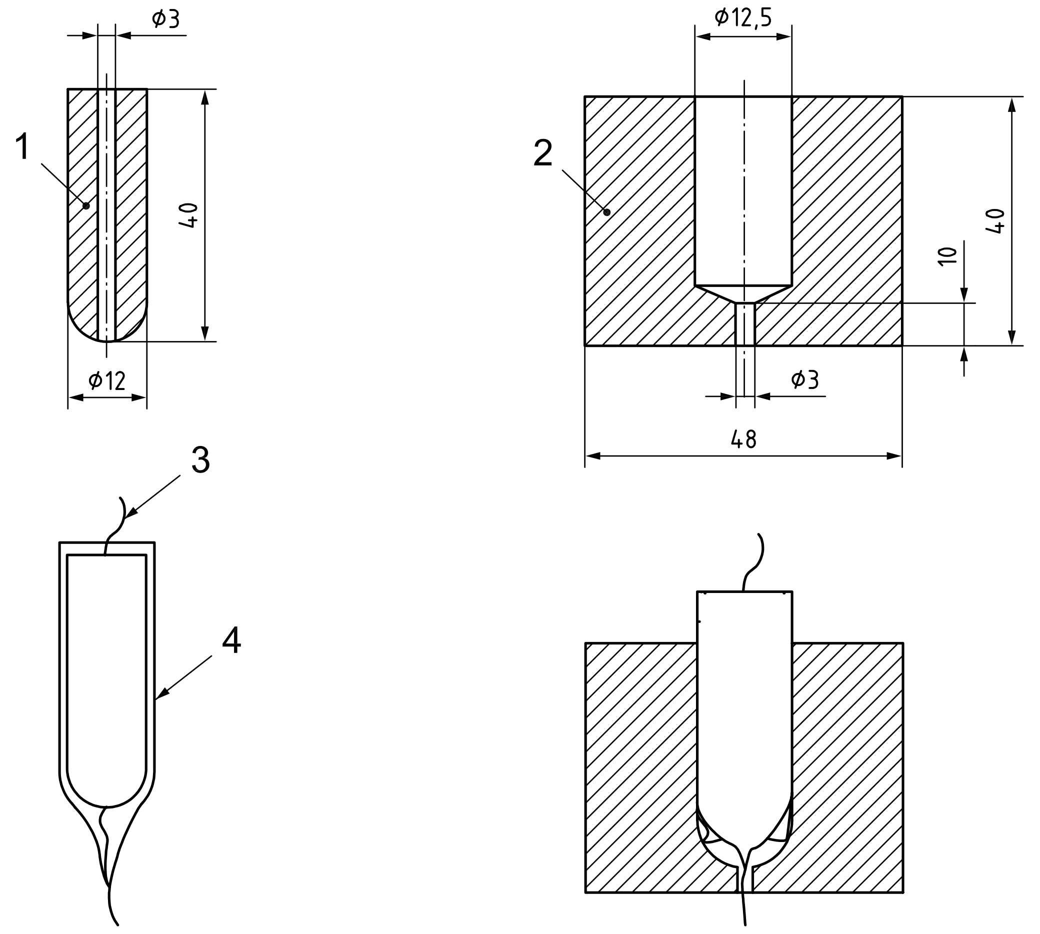


Key

1	stirrer	5	calorimetric vessel	9	electrodes
2	jacket lid	6	jacket	10	firing wire
3	ignition leads	7	calorimetric bomb	11	crucible holder
4	temperature measuring device	8	crucible		

Figure 1 — Test apparatus

Dimensions in millimetres



a) **Shaping the paper over the mandrel**

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

c) **Cigarette completed**

d) **Cigarette placed in the crucible**

Key

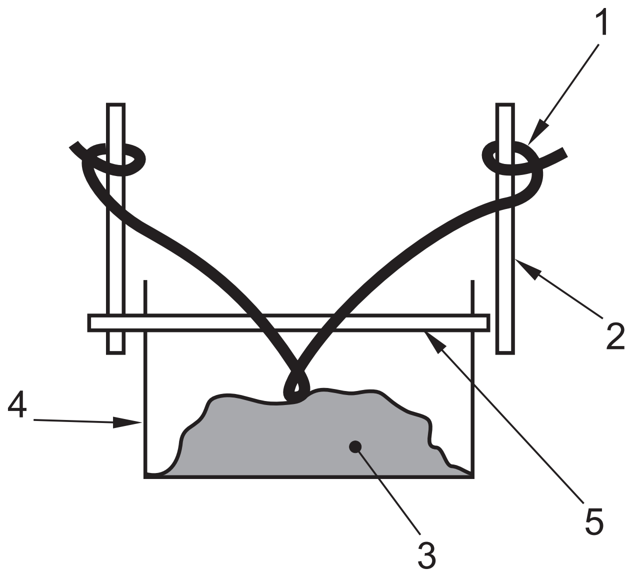
1	mandrel	4	paper	7	crucible
2	mould	5	electrode		
3	firing wire	6	cigarette		

NOTE 1 The paper is kept in place by gluing an overlap of the paper using the preglued cigarette-making paper.

NOTE 2 The two ends of the paper are twisted.

NOTE 3 The “cigarette” is put in the crucible and the firing wire is wrapped tightly around the line of the electrodes

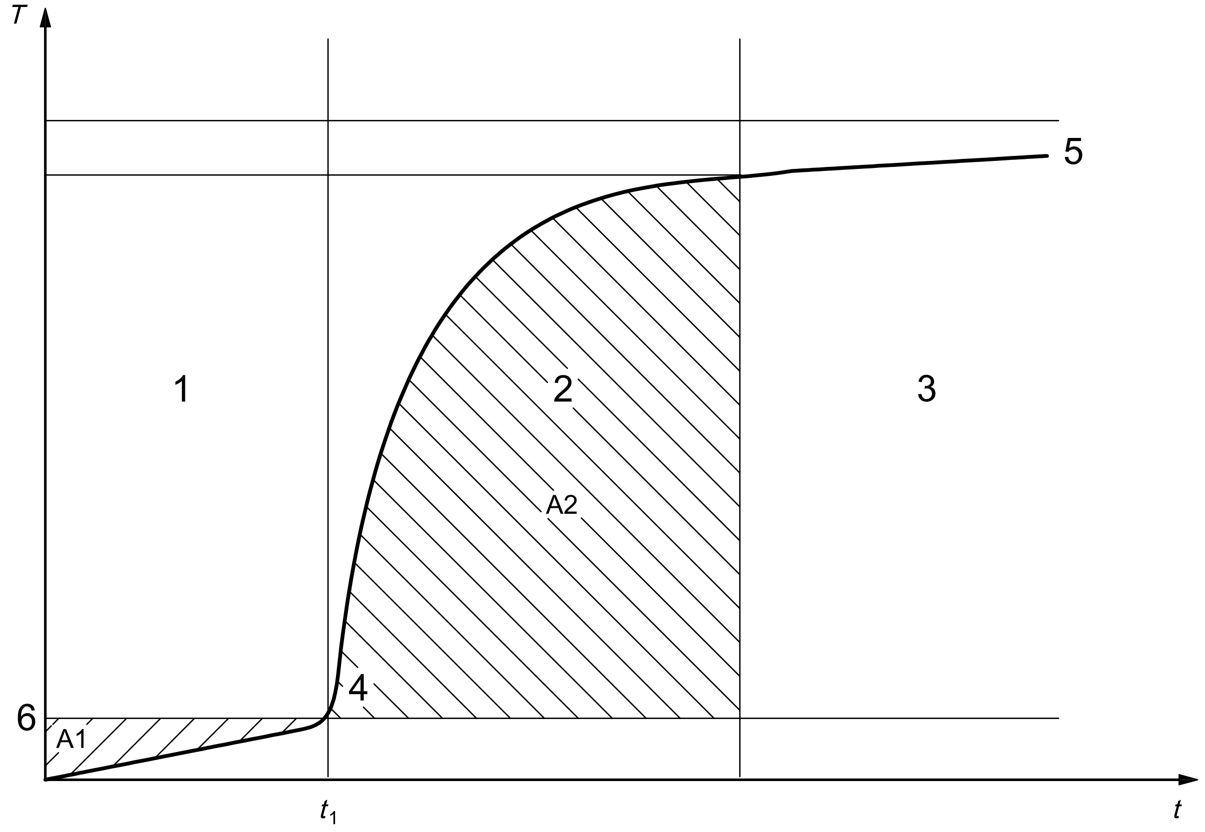
Figure 2 — Method for preparing the “cigarette”



Key

1	firing wire
2	electrodes
3	mixture of benzoic acid and product
4	crucible
5	crucible holder

Figure 3 — Test specimen preparation using crucible method



Key

T	temperature
t	time
1	preliminary period
2	main period
3	final period
4	ignition
5	T (jacket): temperature of the jacket
6	T_0: time when the temperature of the water in the crucible vessel is equal to the temperature of the jacket (see Figure 1) as defined in 8.3.11 b)

Figure 4 — Temperature/time curve

Annex A
(normative)

Calculation of net heat of combustion

The net heat of combustion, Q_{PCI}, is the difference between the gross heat of combustion, Q_{PCS}, and the latent heat of vaporization of the condensed water, q:

$$Q_{\mathrm{PCI}} = Q_{\mathrm{PCS}} - q$$

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, 6 and 7.

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 of the condensed water in the bomb is obtained as follows:

$$q = 2\,449w$$

Annex B
(informative)

Precision of test method

A round-robin exercise, involving 11 European laboratories, was conducted by CEN/TC 127. The protocol used was functionally the same as that described in this International Standard. The products tested in this round-robin exercise were as given in Table B.1.

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

Product	Density kg/m^3	Thickness mm	Mass per unit area g/m^2
Stone wool	145	50	—
Wood fibreboard	50		—
Gypsum fibreboard	110 0	25	—
Phenolic foam		40	—
Flame-retardant (FR) cellulose loose fill	30	—	—
Paint		—	145
PVC/Nitrile rubber (12,9 % chlorine)	65	—	1 235
Acoustic mineral-fibre tiles	wool: 220	18	
— painted glass mat			413,1
— stone wool			4 085
Paper-faced gypsum plasterboard	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, $\bar{m}$, standard deviation, s_r and s_R, repeatability, r, and reproducibility, R, at the 95 % confidence level were calculated in accordance with ISO 5725-2 (see Table B.2) for the two parameters Q_{PCS} with the crucible method in MJ/kg and Q_{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 exclude results identified as “outliers”.

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

Parameter	Statistical mean m	Standard deviation s_r	Standard deviation s_R	Repeatability r	Reproducibility R	$s_r / \overline{m}$	$s_R / \overline{m}$
Q_{PCS} (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 %
Q_{PCS} (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 %

NOTE The percentage values become very high due to division by very low mean values.

It was possible for the two methods to obtain linear models for s_r, s_R, r and R. The coefficients are presented in Table B.3. For the repeatability of Q_{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	Standard deviation s_r	Standard deviation s_R	Repeatability r	Reproducibility R
Q_{PCS} crucible MJ/kg	$= 0{,}07 - 0{,}000\,4 \times Q_{\mathrm{PCS}}$	$= 0{,}09 + 0{,}028\,7 \times Q_{\mathrm{PCS}}$	$= 0{,}20 - 0{,}001\,2 \times Q_{\mathrm{PCS}}$	$= 0{,}26 + 0{,}080\,4 \times Q_{\mathrm{PCS}}$
Q_{PCS} cigarette MJ/kg	$= 0{,}05 + 0{,}004\,1 \times Q_{\mathrm{PCS}}$	$= 0{,}12 + 0{,}032\,8 \times Q_{\mathrm{PCS}}$	$= 0{,}15 + 0{,}011\,4 \times Q_{\mathrm{PCS}}$	$= 0{,}34 + 0{,}091\,8 \times Q_{\mathrm{PCS}}$

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 that a laboratory tests a single specimen of a given product and determines that the Q_{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

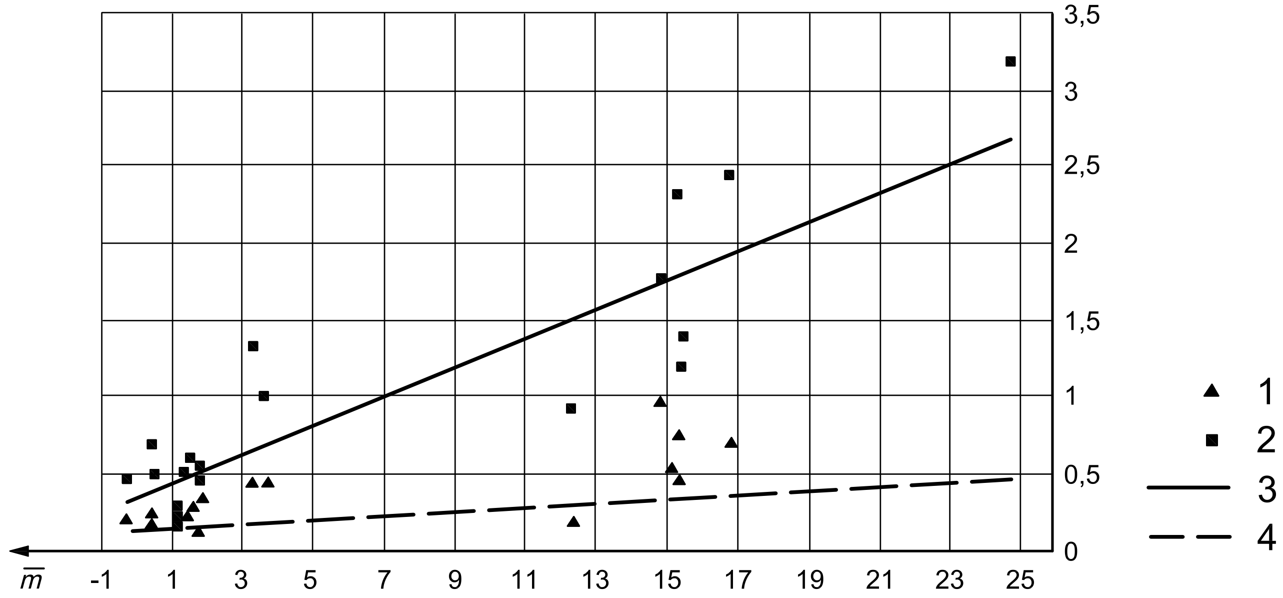
$$r = 0{,}20 - 0{,}001\,2 \times 1{,}57 \approx 0{,}20 \text{ MJ/kg}$$

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

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

$$R = 0{,}26 + 0{,}080\,4 \times 1{,}57 \approx 0{,}39 \text{ MJ/kg}$$

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



Key

1 model r

2 model R

3 reproducibility (line of best fit)

4 repeatability (line of best fit)

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:

$$c = a\left(T - T_0\right)\mathrm{d}t \qquad \text{(C.1)}$$

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:

$$\mathrm{d}c = a\int_{t_1}^{t_\mathrm{m}} (T - T_0)\mathrm{d}t \qquad \text{(C.2)}$$

To calculate the integral, the values of a and T_0 have 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:

$\left(\frac{\mathrm{d}c}{\mathrm{d}t}\right)$ at moments 1 and 2

We can therefore write:

$$\left(\frac{\mathrm{d}c}{\mathrm{d}t}\right)_1 = a\left(T_1 - T_0\right) \qquad \text{(C.3)}$$

$$\left(\frac{\mathrm{d}c}{\mathrm{d}t}\right)_2 = a\left(T_2 - T_0\right) \qquad \text{(C.4)}$$

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

$\left(\frac{\mathrm{d}c}{\mathrm{d}t}\right)_1$ and $\left(\frac{\mathrm{d}c}{\mathrm{d}t}\right)_2$

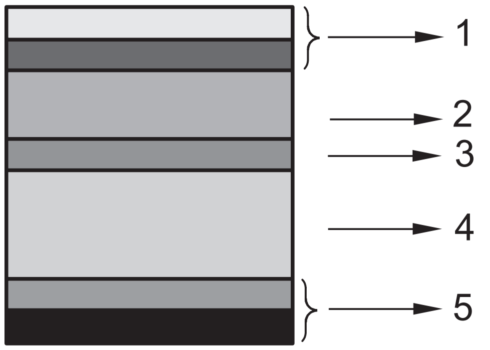
The integral [Equation (C.2)] can then be evaluated by a graph (see Figure 4). It is sufficient to plot the temperature curve as a function of time between moments t_i and t_m and the horizontal straight line with ordinate T_0. The difference between the hatched areas A1 and A2 situated above and below 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
5	external substantial component

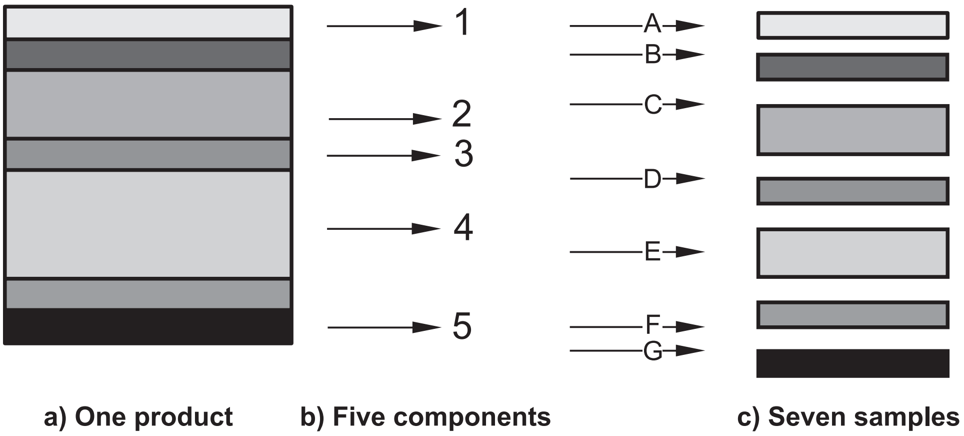
Figure D.1 — Non-homogeneous product to be evaluated

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 7.2 and each sample is ground as specified in 7.4.



Key

A minimum 0,5 m^2 and minimum 10,0 g
B minimum 10,0 g of dried material (glue)
C minimum 0,5 m^2 and minimum 50,0 g
D minimum 10,0 g of dried material (glue)
E minimum 0,5 m^2 and minimum 50,0 g
F minimum 10,0 g of dried material (glue)
G minimum 0,5 m^2 and minimum 50,0 g
1, 2, 3, 4 and 5 are defined in Figure D.1

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 seven components, in kg/m^2, is determined as specified in 8.3. The area weights of the seven components A, B, C, D, E, F and G are: m_A, m_B, m_C, m_D, m_E, m_F and m_G.

The mass per unit area of the product is: $m = m_A + m_B + m_C + m_D + m_E + m_F + m_G$.

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 8.3, i.e. three results for each component, in MJ/kg, are:

Q_{PCSA1} Q_{PCSB1} Q_{PCSC1} Q_{PCSD1} Q_{PCSE1} Q_{PCSF1} Q_{PCSG1}

Q_{PCSA2} Q_{PCSB2} Q_{PCSC2} Q_{PCSD2} Q_{PCSE2} Q_{PCSF2} Q_{PCSG2}

Q_{PCSA3} Q_{PCSB3} Q_{PCSC3} Q_{PCSD3} Q_{PCSE3} Q_{PCSF3} Q_{PCSG3}

These results are analysed for each component as described in Clause 9 and further tests are carried out, if required, giving the following mean values for each component:

– in MJ/kg: Q_{PCSA}, Q_{PCSB}, Q_{PCSC}, Q_{PCSD}, Q_{PCSE}, Q_{PCSF} and Q_{PCSG}

– in MJ/m^2: $Q_{PCSSA} = m_A \times Q_{PCSA}$, $Q_{PCSSB} = m_B \times Q_{PCSB}$, $Q_{PCSSC} = m_C \times Q_{PCSC}$, $Q_{PCSSD} = m_D \times Q_{PCSD}$,

$Q_{PCSSE} = m_E \times Q_{PCSE}$, $Q_{PCSSF} = m_F \times Q_{PCSF}$ and $Q_{PCSSG} = m_G \times Q_{PCSG}$

The gross heat of combustion of the external non-substantial component of the product (1), in MJ/m^2, is

$Q_{\mathrm{PCS}_{\mathrm{Sext}}} = Q_{\mathrm{PCS}_{\mathrm{SA}}} + Q_{\mathrm{PCS}_{\mathrm{SB}}}$

The gross heat of combustion of the external non-substantial component of the product (1), in MJ/kg, is

$Q_{\mathrm{PCSext}} = (Q_{\mathrm{PCS}_{\mathrm{SA}}} + Q_{\mathrm{PCS}_{\mathrm{SB}}})/m_{\mathrm{A}} + m_{\mathrm{B}}$

The gross heat of combustion of the external substantial component of the product (5), in MJ/m^2, is

$Q_{\mathrm{PCS}_{\mathrm{Sext}}} = Q_{\mathrm{PCS}_{\mathrm{SF}}} + Q_{\mathrm{PCS}_{\mathrm{SG}}}$

The gross heat of combustion of the external substantial component of the product (5), in MJ/kg, is

$Q_{\mathrm{PCSext}} = (Q_{\mathrm{PCS}_{\mathrm{SF}}} + Q_{\mathrm{PCS}_{\mathrm{SG}}})/m_{\mathrm{F}} + m_{\mathrm{G}}$

The gross heat of combustion of the whole product (PCS$_{\mathrm{S}}$), in MJ/m^2, is

$Q_{\mathrm{PCS}_{\mathrm{S}}} = Q_{\mathrm{PCS}_{\mathrm{SA}}} + Q_{\mathrm{PCS}_{\mathrm{SB}}} + Q_{\mathrm{PCS}_{\mathrm{SC}}} + Q_{\mathrm{PCS}_{\mathrm{SD}}} + Q_{\mathrm{PCS}_{\mathrm{SE}}} + Q_{\mathrm{PCS}_{\mathrm{SF}}} + Q_{\mathrm{PCS}_{\mathrm{SG}}}$

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

$Q_{\mathrm{PCS}} = Q_{\mathrm{PCS}_{\mathrm{S}}}/m$

Bibliography

[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*

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