INTERNATIONAL
STANDARD

ISO
1716

Fourth edition
2018-05

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 1716:2018(E)

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This fourth edition cancels and replaces the third edition (ISO 1716:2010), 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 document specifies a method for the determination of the gross heat of combustion (Q_{PCS}) of products at constant volume in a bomb calorimeter.

This method is intended to be applied to solid products.

NOTE Liquids can be tested with similar equipment and using conditions described in ASTM D240[1], as described in IEC 61039[2] using ISO 1928[3] test equipment.

Annex A specifies 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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*

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 following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 Stone, timber, concrete, mineral wool with a uniformly dispersed binder and polymers.

3.3
homogeneous product

product (3.1) consisting of a single *material* (3.2) having uniform density and composition throughout the *product* (3.1)

3.4
non-homogeneous product

product (3.1) that does not satisfy the requirements of a *homogeneous product* (3.3) and which is composed of more than one component, substantial or non-substantial

Note 1 to entry: If a non-homogeneous product cannot be easily separated into its component parts, the individual components shall be provided separately by the sponsor.

3.5
non-substantial component

material (3.2) that does not constitute a significant part of *a non-homogeneous product* (3.4) and that has a layer with a mass/unit area below 1,0 kg/m^2 and a thickness below 1,0 mm

Note 1 to entry: If the non-substantial layers do not comply with the above requirements, together they shall be considered to be substantial.

Note 2 to entry: 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.

3.6
substantial component

material (3.2) that constitutes a significant part of a *non-homogeneous product* (3.4) and that has a layer with a mass/unit area more than or equal to 1,0 kg/m^2 or a thickness more than or equal to 1,0 mm

Note 1 to entry: 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 substantial component when they collectively comply with the requirements for a layer being a substantial component.

3.7
internal non-substantial component

non-substantial component (3.5) that is covered on both sides by at least one *substantial component* (3.6)

3.8
external non-substantial component

non-substantial component (3.5) that is not covered on one side by a *substantial component* (3.6)

3.9
heat of combustion

DEPRECATED: calorific value

Q

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

Note 1 to entry: The heat of combustion is expressed in megajoules per kilogram.

[SOURCE: ISO 13943:2008, 4.174, modified — The Note 1 to entry was changed.]

3.10

gross heat of combustion

Q_{PCS}

heat of combustion (3.9) of a substance when the combustion is complete and any produced water is entirely condensed under specified conditions

Note 1 to entry: The gross heat of combustion is expressed in megajoules per kilogram.

Note 2 to entry: The acronym PCS is derived from the French term “pouvoir calorifique supérieur”.

3.11

net heat of combustion

Q_{PCI}

heat of combustion (3.9) of a substance when the combustion is complete and any produced water is in the vapour state under specified conditions

Note 1 to entry: The net heat of combustion may be calculated from the gross heat of combustion.

Note 2 to entry: The net heat of combustion is expressed in megajoules per kilogram.

Note 3 to entry: The acronym PCI is derived from the French term “pouvoir calorifique inférieur”.

3.12

latent heat of vaporization of water

q

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

Note 1 to entry: The latent heat of vaporization is expressed in megajoules per kilogram.

3.13

surface density

mass per unit area

Note 1 to entry: The surface density is expressed in kilograms per square metre.

4 Principle

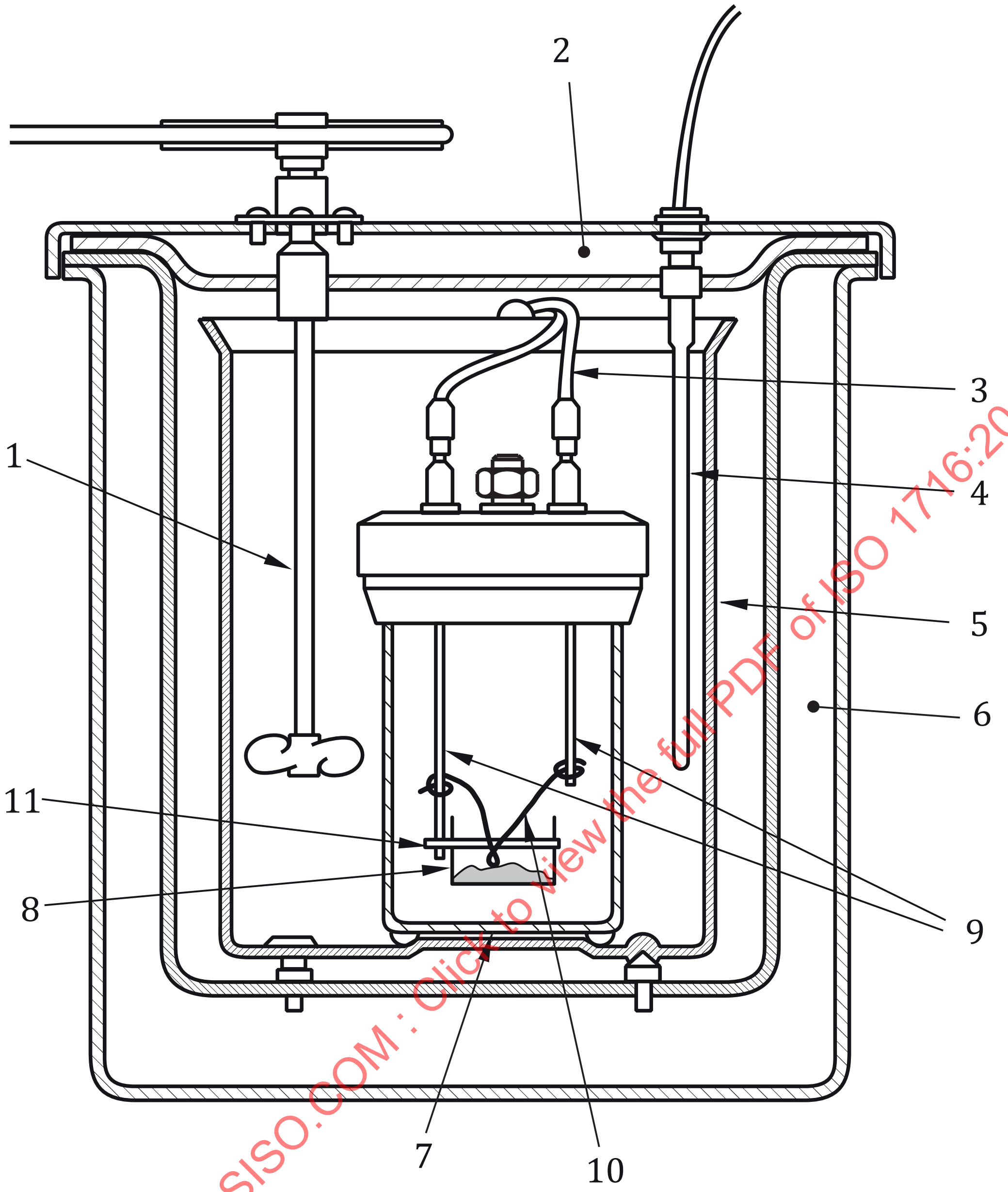
In this test, a test specimen of specified mass is burned under standardized conditions, at constant volume, in an atmosphere of oxygen, in a bomb calorimeter calibrated by combustion of certified benzoic acid. The heat of combustion determined under these conditions is calculated on the basis of the 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

The test apparatus (bomb calorimeter) shall be as illustrated in Figure 1, and as detailed in 5.1 to 5.4. Additional equipment shall be in accordance with 5.5 to 5.10.

Equipment described in 5.1 to 5.4 could also be available as automatic or semi-automatic apparatuses. Any deviation from these subclauses has to be evaluated by the user according to requirements of subsequent subclauses.



Key

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

Figure 1 — Test apparatus

5.1 Calorimetric bomb.

The calorimetric bomb shall be constructed with the following characteristics:

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.

5.2 Calorimeter.

5.2.1 Jacket.

The jacket shall consist 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).

5.2.2 Calorimetric vessel.

The calorimetric vessel shall consist of a polished metal container designed to accommodate the bomb. The dimensions shall be such that the bomb can be immersed in water (see 8.3.8).

5.2.3 Stirrer.

The stirrer shall be 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.

5.3 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 gently tap the thermometer to ensure that the mercury column does not stick.

5.4 Crucible.

The crucible shall 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.

5.5 Timing device.

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

5.6 Electric power source.

The electric power source shall be designed with the voltage to the firing circuit not exceeding 20 V for the firing.

NOTE An ammeter can be added to the circuit to indicate the breaking of the firing wire. A circuit breaker is a useful addition to the supply circuit.

5.7 Pressure gauge and needle-valve.

A pressure gauge and a needle valve 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.

5.8 Balances.

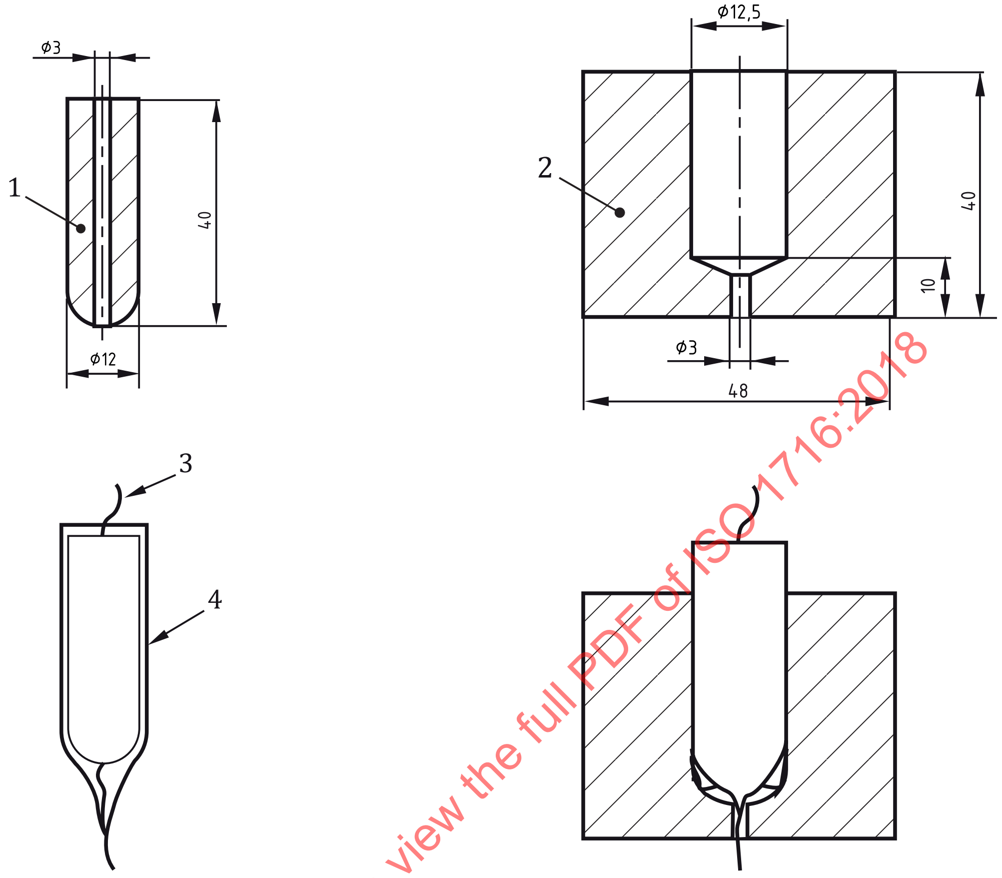
Two balances shall be used with the following characteristics:

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

5.9 Device for making the “cigarette”.

The device shall be designed as shown in Figure 2, and comprise a mould and a metallic mandrel (not aluminium).

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	3	firing wire	5	electrode	7	crucible
2	mould	4	paper	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”

5.10 Device for making the pellet.

The device is suitable if prefabricated pellets are not available.

6 Reagents and materials

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6.1 Distilled or demineralized water.

6.2 Pressurized oxygen.

The oxygen used shall be free from any other combustible product (purity ≥ 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.

Benzoic acid whose gross heat of combustion is guaranteed is used as "reference standard for calorimetry".

6.4 Combustion aid.

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

6.5 Cigarette-making paper.

The cigarette making paper shall be 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.

The firing wire shall be 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 (5.4), 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.

NOTE Some automatic or semi-automatic apparatuses are working with permanent firing wires in combination with single use combustible crucibles. Reference has to be made to the instructions of the manufacturer of such calorimeter systems.

6.7 Thread.

The thread shall be 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 easily separated into its component parts, the individual components shall be provided separately by the sponsor of the test. 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.

Some substantial components cannot be produced without facings. If the facings cannot be delaminated, a section shall be cut from the central part of the core avoiding the facings and any adhesive or other non-substantial component on its surface.

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.

If the product tested is made of non-separable multiple layers in which one layer comprises aluminium or other metallic foils, recommendations of Reference [4] should be applied.

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 sample may be prepared by applying a thin layer of the liquid-applied product to a glass sheet and taking the sample for the test by scrapping the dried product from the glass surface.

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.

7.2.4 Thin film products

The sample shall have a minimum mass of 10 g. The film shall be cut into small pieces for the test which shall either be tested as a cigarette (7.10) or shall be tested in the crucible. For the crucible method, it is recommended that 0,5 g of the paraffin oil combustion aid is added to the crucible over the surface of the film to stop the film from being ejected from the crucible.

7.3 Determination of surface density

Where required, the surface density of each sample 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 which should be as small as possible, 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 Q_{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 Q_{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 the following elements:

— Approximately 0,5 g of material, with a minimum precision of 0,1 mg;

— Approximatively 0,5 g of benzoic acid, with a minimum precision of 0,1 mg;

— combustion aid, with a minimum precision of 0,1 mg;

— firing wire, cotton thread and cigarette-making paper, if necessary, with a minimum precision of 0,1 mg.

NOTE 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 or by adding a combustion aid (e.g. paraffin oil). The benzoic acid and/or combustion aid can be reduced or excluded if it’s not necessary for combustion of the material.

7.9 Crucible method

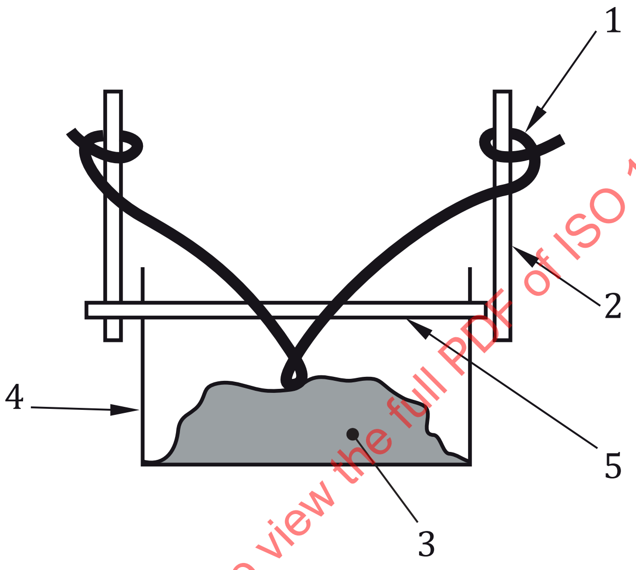
The procedure shall be carried out as follows (see Figure 3).

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.



Key

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

Figure 3 — Test specimen preparation using the crucible method

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 with a minimum precision of 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.

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 and wait at least 1 h before testing, or until stabilization is reached.

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. Absorption of the acid gases can help reduce the amount of pitting within the bomb vessel.

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 can 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, 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, using combustion aid and/or an increased proportion of benzoic acid. 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 Formula (1):

$$c = (t - t_{\mathrm{I}}) \times T_2 - t_{\mathrm{I}} \times T_{\mathrm{I}} \qquad (1)$$

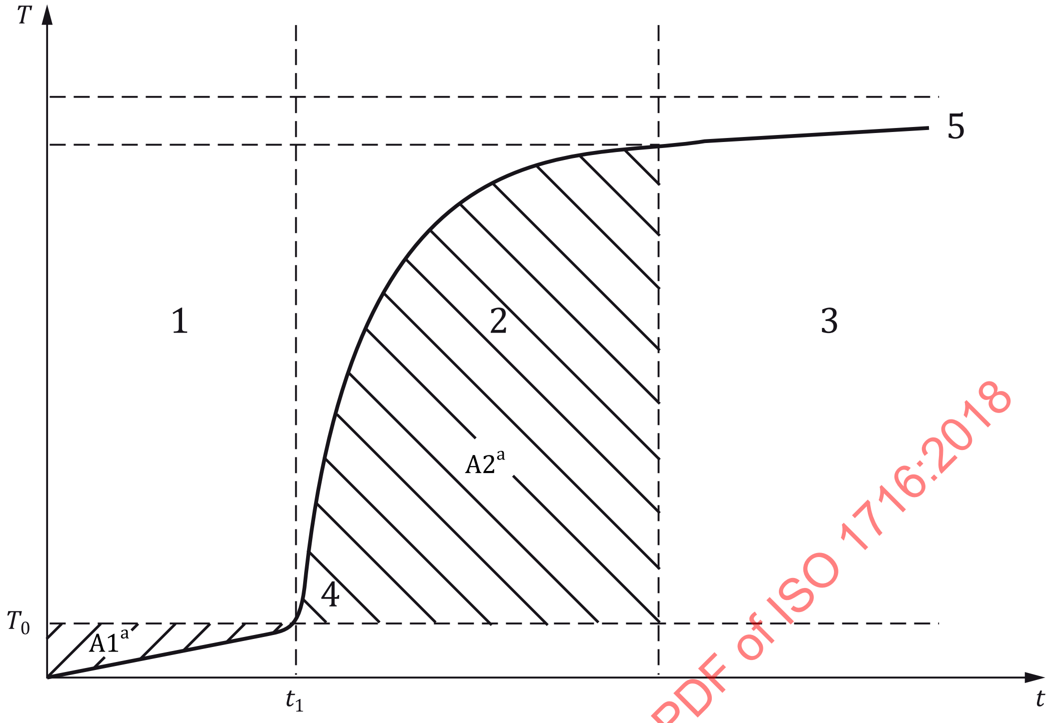
where

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).



Key

T	temperature
t	time
1	preliminary period
2	main period
3	final period
4	ignition
5	temperature/time curve of the jacket
t_1	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)

[a] A1 and A2 respectively represent the hatched areas of temperature/time curve situated above and below the line of ordinate T_0.

Figure 4 — Temperature/time curve

NOTE 1 *c* is null if an adiabatic jacket is used.

NOTE 2 *c* is null 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 Formula (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.

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

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 null if an adiabatic jacket is used;

m is the 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, related to the mass of wire consumed, shall be:

— nickel-chrome = 5,872 MJ/kg;

— nickel = 4,10 MJ/kg[5];

— pure iron = 7,39 MJ/kg[5].

NOTE Platinum wires do not oxidize during the test. The correction factor is then only dependent on the electrical energy used by the system to heat the wire.

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 $Q_{PCS\ S}$ 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 document (i.e. ISO 1716:2018);

b) any deviations from the test method;

c) the name and address of the testing laboratory;

d) the date and identification number of the report;

e) the name and address of the sponsor;

f) the name and address of the manufacturer/supplier, if known;

g) the date of sample arrival;

h) the identification of the product;

i) a 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) a description of method to cure material, where relevant;

m) the date of test;

n) the 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 following 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	Acceptance criteria	Range of validity
Q_{PCS} (MJ/kg)	≤0,2 MJ/kg	From any negative value to 3,2 MJ/kg
	Within 5 % of the average of the 3 results	From 3,2 MJ/kg to 20,0 MJ/kg
	Within 10 % of the average of the 3 results	Greater than 20,0 MJ/kg
Q_{PCS} (MJ/m^2)[a]	≤0,1 MJ/m^2	From any negative value to 4,1 MJ/m^2
	Within 5 % of the average of the 3 results	From 4,1 MJ/m^2 to 20 MJ/m^2
	Within 10 % of the average of the 3 results	Greater than 20 MJ/m^2

[a] For non-substantial components only.

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:

$$q = 2\,449\,w$$

Annex B
(informative)

Precision of test method

B.1 CEN/TC 127 round-robin exercise

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 document. 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	1 100	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, $\overline{m}$, 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[6] (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	$= 0{,}07 - 0{,}000\,4 \times Q_{\text{PCS}}$	$= 0{,}09 + 0{,}028\,7 \times Q_{\text{PCS}}$	$= 0{,}20 - 0{,}001\,2 \times Q_{\text{PCS}}$	$= 0{,}26 + 0{,}080\,4 \times Q_{\text{PCS}}$
Q_{PCS} cigarette MJ/kg	$= 0{,}05 + 0{,}004\,1 \times Q_{\text{PCS}}$	$= 0{,}12 + 0{,}032\,8 \times Q_{\text{PCS}}$	$= 0{,}15 + 0{,}011\,4 \times Q_{\text{PCS}}$	$= 0{,}34 + 0{,}091\,8 \times Q_{\text{PCS}}$

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 (see Figure B.1).

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

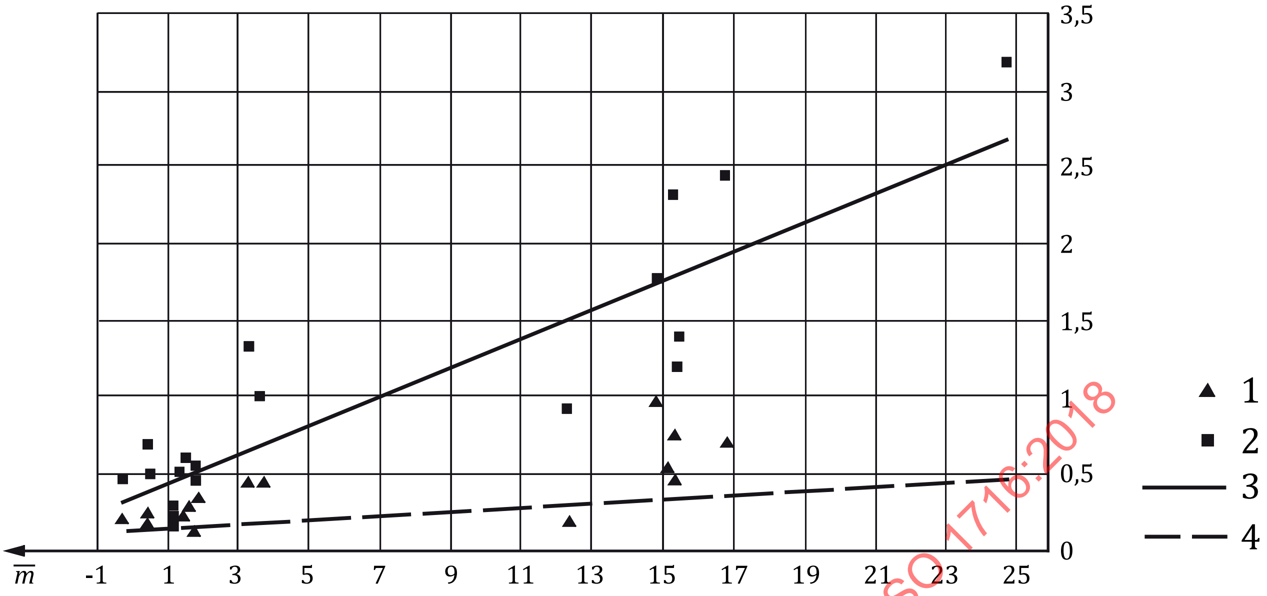
$$r = 0{,}20 - 0{,}001\,2 \times 1{,}57 \approx 0{,}20 \text{ MJ/kg}$$

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 Q_{PCS} with the crucible method, in MJ/kg

B.2 EGOLF round-robin exercise, 2008-2009

A round-robin exercise has been performed in the framework of EGOLF in 2008-2009, involving 35 laboratories[7].

The first product for the round robin exercise was a copolyamide in powder form. This material was selected because it is homogeneous, and no special preparation of the samples is necessary. Furthermore, the product is having a Q_{PCS} value greater than or equal to 30 MJ/kg, and therefore no combustion aid is necessary for the tests. The second material was a mineral wool with a nominal density of 15 kg/m^3. The Q_{PCS} value should be between 1,5 MJ/kg and 2,0 MJ/kg. The third material was a black glass tissue with a nominal area weight of 60 g/m^2. The Q_{PCS} value should be between 3,0 MJ/kg and 4,0 MJ/kg.

The data were analysed according to ISO 5725-2[6]. In the first round 32 labs carried out the tests with Material 1 Procedure 1 and 2. After this 34 labs determined the gross heat of combustion of Material 2 and Material 3 in a second round. The 35 participating labs used different types of calorimeter, produced by different manufacturers. The working methods were adiabatic, isoperibolic or isothermal. Due to the test results of this round robin exercise there was no significant influence of the equipment of the labs. Also the used combustion aids like benzoic acid, paraffin oil, PE-bag, etc. did not lead to different test results.

The number of outliers and stragglers due to the between-laboratory consistency is less than the number due to the within-laboratory consistency. Reasons for the outliers and stragglers of the test results of some labs could not be found in the test reports of the labs. Also the photos taken of the test materials before and after the tests show no significant deviations from the given test procedure.

All statistical values from the EGOLF round robin exercise are clearly less than the maximum values in ISO 1716:2010, Table B.2. This shows that the labs can carry out this reaction to fire test in a very proper way.

Annex C
(informative)

Calculation by graph of the corrective term, *c*, necessarybecause 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 d*t*, the calorimeter undergoes a positive or negative d*c* cooling from the outside tied to the temperature by the Newton relation:

$$\mathrm{d}c = a(T - T_0)\,\mathrm{d}t \tag{C.1}$$

where *a* is 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:

$$c = a\int_{t_1}^{t_m}\left[T - T_0\right]\mathrm{d}t \tag{C.2}$$

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:

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

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

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

$$\left[\frac{\mathrm{d}c}{\mathrm{d}t}\right]_1 \text{ and } \left[\frac{\mathrm{d}c}{\mathrm{d}t}\right]_2$$

The integral [Formula (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*.