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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)

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Contents

Page

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 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 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.