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Building materials – Determination of calorific potential

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 1716 replaces ISO Recommendation R 1716-1971 drawn up by Technical Committee ISO/TC 92, *Fire tests on building materials and structures*.

The Member Bodies of the following countries approved the Recommendation :

Austria	Greece	Poland
Belgium	India	Portugal
Canada	Iran	Romania
Denmark	Israel	South Africa, Rep. of
Egypt, Arab Rep. of	Italy	Sweden
Spain	Korea, Rep. of	Turkey
France	Netherlands	United Kingdom
Germany	Norway	U.S.A.

The Member Bodies of the following countries expressed disapproval of the Recommendation on technical grounds :

Australia
New Zealand

Building materials – Determination of calorific potential

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a test method for the determination of the calorific potential of building materials.

NOTE – For materials containing metals, the calorific potential does not always represent the fire load.

2 DEFINITIONS

2.1 gross calorific potential : The amount of heat released by the complete combustion of a unit of mass of the material. The gross calorific potential has a different value according to whether combustion takes place under constant pressure or constant volume. The gross calorific potential determined under constant volume is considered in this International Standard.

2.2 net calorific potential : The gross calorific potential reduced by the latent heat of vaporization released by the water condensed in the bomb after combustion, i.e. the water formed by the combustion of the hydrogen present in the material, and the water corresponding to the moisture content of the material or the water of crystallization present in the material or both.

3 SYMBOLS

Symbol	Definition
Q_{gr}	Gross calorific potential, in kilojoules per kilogram.
Q_{net}	net calorific potential, in kilojoules per kilogram.
E	water equivalent of the apparatus, in kilograms, determined with an identical calibration test on benzoic acid (gross calorific value, $H_o = 26\,435$ kJ/kg).
W	mass, in kilograms, of the distilled water introduced into the calorimeter vessel.

t_i	temperature of the water in the calorimeter vessel at the beginning of the chief period, in degrees Celsius.
t_m	maximum temperature attained during the chief period, in degrees Celsius.
c	correction, in degrees Celsius, for heat transfer between the calorimeter vessel and the water jacket.
C	corrections, in kilojoules, for heat gains or losses other than to the water jacket.
m	mass of the test specimen, in kilograms.
n	duration, in seconds, of the chief period.
n'	time, in seconds, elapsing from the start of the chief period until the moment when the increase in temperature has become equal to $0,6(t_m - t_i)$.
v'	average temperature change gradient during the preliminary period, in degrees Celsius per second.
v''	average temperature change gradient during the final period, in degrees Celsius per second.
m_a	mass, in kilograms, of the additional substance present.
m_f	mass, in kilograms, of the substance used for firing the specimen.
H_{oa}	gross calorific value, in kilojoules per kilogram, of the additional substance.
H_{of}	gross calorific value, in kilojoules per kilogram, of the substance used for firing the specimen. ¹⁾
w	proportional content of condensed water in the bomb after combustion.
q	latent heat of vaporization released by the water condensed in the bomb.

1) Firing wire :
 nickel-chromium $H_{of} = 1\,403$ kJ/kg
 platinum $H_{of} = 419$ kJ/kg
 pure iron $H_{of} = 7\,490$ kJ/kg
 Cotton wool $H_{of} = 17\,543$ kJ/kg

4 SAMPLING

The sample shall be sufficiently large to be representative of the material, particularly in the case of non-homogeneous materials.

5 DETERMINATION OF GROSS CALORIFIC POTENTIAL

5.1 Additional combustible substances

In order to obtain complete combustion when the gross calorific potential of the materials subjected to this test is relatively low, it may frequently be necessary.

- to increase the gross calorific potential of the specimen by adding a very combustible substance having a known and high gross calorific potential, for instance benzoic acid, or
- to use an envelope made of a very combustible material, having a known and high gross calorific potential, in which the specimen is placed, or
- to use any other method that ensures complete combustion without compromising the precision of the test.

To ascertain after the test that complete combustion has taken place, the residue of the test shall be dried, its mass shall be determined to the nearest 0,1 mg and it shall be left for 1 h in a furnace at a temperature of 900 °C. After cooling in a desiccator to the ambient temperature, a second weighing will show if the combustion was complete.

5.2 Apparatus

5.2.1 High-pressure calorimetric bomb, completely equipped with all its accessories.

5.2.2 Calorimeter vessel for the bomb, with all its accessories.

5.2.3 Water jacket, having its exterior surface polished, with all its accessories.

5.2.4 Stirrer, operated by a constant-speed motor of which the speed can be changed, with all its accessories.

5.2.5 Temperature measuring equipment that permits measurement of the temperature of the calorimeter vessel water with a precision of 0,002 °C.

5.2.6 Crucible of platinum, nickel-chromium, or silica, or any other device made of heat-, corrosion- and oxidation-resistant materials and able to keep the specimen in place.

5.2.7 Firing wire of platinum, pure iron or nickel-chromium.

5.2.8 Ignition circuit having an electrical supply not exceeding 20 V.

5.2.9 Pressure regulator and gauge for fitting into the oxygen line to indicate the pressure in the bomb during filling. The oxygen pressure at the end of the filling shall be 2 451,6 kN/m² (24,52 bar).

5.3 Preparation of test specimens

The sample shall be reduced gradually to the final test specimen using one of the following methods :

5.3.1 For homogeneous materials

Grind the sample and reduce it with the method of cross reduction, grinding to a finer powder as reduction proceeds.

5.3.2 For heterogeneous materials

5.3.2.1 Separate the constituents of the sample as efficiently as possible. Weigh the constituents to establish the ratio of their masses. Grind each of the constituents and proceed as specified in 5.3.1. Prepare a specimen of each constituent having such a mass that the combined specimen obtained by mixing all the specimens thoroughly together has the same composition as the sample.

5.3.2.2 If the sample cannot be separated into its constituents, grind the whole sample and separate the powder by sieving or any other method. Treat the powders as specified in 5.3.2.1.

5.3.2.3 If separation is impossible, proceed as for homogeneous materials, as specified in 5.3.1.

5.3.2.4 If the material or one of its constituents cannot be ground, reduce the material by any appropriate method into small lumps or pieces and treat the specimens obtained as a powder.

5.3.3 For composite materials

Proceed as specified in 5.3.2.1 but preparing a specimen for each of the constituents.

5.3.4 For all materials

Any other methods which will produce a specimen as representative as those mentioned above may be used.

5.4 Conditioning

The specimen shall be conditioned for 20 h in surroundings having a temperature of 20 ± 2 °C and relative humidity of 65 ± 5 %.

5.5 Procedure

Weigh, respectively, the crucible or other device to keep the specimen in place, the firing wire if it burns, the additional substance necessary to realize the complete combustion, etc., to the nearest 0,1 mg. The firing wire must be brought through or around the specimen in such a way that combustion is certain.

Introduce 5 ml of distilled water into the bomb. Close the bomb, connect to the oxygen supply, drive the air out of the bomb with a slow oxygen stream with the relief valve in the open position, close the relief valve and charge slowly with oxygen to a pressure of 2 451,6 kN/m² (24,52 bar). Disconnect the oxygen supply.

Transfer the calorimeter vessel into the water jacket. Lower the bomb into the calorimeter vessel. Place the stirrer and the temperature measuring device into position. Connect the electrodes of the bomb to the firing circuit.

Introduce into the calorimeter vessel a quantity of distilled water sufficient to cover the upper surface of the bomb cap. The mass of the water shall be determined to the nearest 1 g. This quantity shall be the same as that used in the calibration test. The temperature of this water shall be about 1,5 °C below that of the water jacket when the calorimeter is not an adiabatic calorimeter. The temperature rise of the water in the calorimeter vessel due to the firing and complete combustion of the specimen should be of the order of 3 °C.

Start the stirrer and set it at the same speed as that used during the calibration test. After 3 min, start temperature readings, which shall be made at intervals of 1 min for 5 min, or start the temperature recorder. This 5 min period is called the "preliminary period".

Ignite the specimen by closing the firing circuit and passing a suitable current through the firing wire. This current shall be the same as that used during the calibration test. The ignition marks the beginning of the "chief period". During this period, note the temperature every 30 s or record it continuously. This period ends when the maximum temperature is reached.

The moment when the maximum temperature is attained also marks the beginning of the "final period"¹⁾. During this period, which lasts 5 min, note the temperature at intervals of 1 min or record it continuously. At the end of this period, note the temperature of the water jacket. Remove the bomb, release its pressure slowly, dismantle and collect the residue to perform the calcination test specified in 5.1.

Wash the bomb with distilled water and collect the wash-water in a beaker to permit its analysis for the determination of the quantity of the different products that were formed.

Calculate the gross calorific potential as specified in 5.6.

5.6 Calculation of results

The gross calorific potential of the specimen is given by the formula

$$Q_{gr} = \frac{4,1868 (E + W) (t_m - t_i + c) - C}{m} \text{ kJ/kg}$$

a) *Correction c* : Calculate this correction using the formula

$$c = (n - n') v'' - n' v'$$

b) *Correction C* :

$$C = \sum C_n (n = 1, 2 \text{ etc.}).$$

In this correction, when necessary, the following shall be included :

1) The amount of heat introduced by the additional substance used to obtain complete combustion :

$$C_1 = m_a H_{oa}$$

2) The amount of heat introduced by the burning of all the substances used for firing :

$$C_2 = m_f H_{of}$$

3) The amounts of heat quantities set free or absorbed by the chemical reactions that have taken place during the combustion in an oxygen atmosphere at 2 451,6 kN/m² in the presence of 5 ml of distilled water and that do not take place when the combustion occurs at atmospheric pressure. The quantities of the substances formed must be determined by the appropriate chemical analysis (titration) of the solution and the corresponding amounts of heat calculated on the basis of the known heats of formation or dissolution.

NOTE — Where elements other than carbon, hydrogen or oxygen are likely to be present, a pure chemical analysis of the material must be made in order to determine what corrections are to be applied under paragraph b) 3) above.

5.7 Number of specimens

The test shall be carried out on three specimens. The results of these three tests shall not differ by more than 167 kJ/kg. If the difference between any two results is greater than 167 kJ/kg the entire test shall be repeated.

5.8 Gross calorific potential

The gross calorific potential of the material is the average of the three test results.

1) Sometimes known as the "after period".

6 DETERMINATION OF NET CALORIFIC POTENTIAL

6.1 Procedure

It is necessary to determine the gross calorific potential and the latent heat of vaporization released by the water, q , condensed in the bomb after combustion.

6.1.1 Gross calorific potential

The gross calorific potential is determined by the average value of three test results, calculated as described in 5.6.

6.1.2 Amount of condensed water

The amount of condensed water in the bomb after combustion is determined by special tests carried out with a specimen of powder prepared and conditioned as described in 5.3 and 5.4 with micro- or macro-analysis equipment for the measurement of the hydrogen content.

6.2 Number of specimens

Tests shall be carried out on two specimens.

6.3 Proportional content of condensed water

The content of condensed water, w , is given by the average of the two values obtained.

6.4 Latent heat of vaporization of the condensed water

The latent heat of vaporization, q , of the condensed water in the bomb is obtained by the formula

$$q = 2\,449 w$$

6.5 Net calorific potential

The net calorific potential is the difference between the gross calorific potential and the latent heat of vaporization of the condensed water, q :

$$Q_{\text{net}} = Q_{\text{gr}} - q$$

7 TEST REPORT

The test report shall include the following particulars :

- a) name of the manufacturer of the material;
- b) name or identification mark of the material;
- c) description of the material;
- d) density of the material;
- e) date of supply of the material and date of tests;
- f) description of the method used in preparing the representative test specimen;
- g) description of the test method;
- h) test results :

1) *Gross calorific potential*

kJ/kg

Test 1

Test 2

Test 3

Average.

2) *Latent heat of vaporization of the water*

Test 1

Test 2

Average.

3) *Net calorific potential*

- i) name of testing body.

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