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R 1716

BUILDING MATERIALS

DETERMINATION OF CALORIFIC POTENTIAL

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BRIEF HISTORY

The ISO Recommendation R 1716, *Building materials – Determination of calorific potential*, was drawn up by Technical Committee ISO/TC 92, *Fire tests on building materials and structures*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1716, which was circulated to all the ISO Member Bodies for enquiry in October 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Bodies opposed the approval of the Draft :

Australia
New Zealand

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1716 | October 1971

BUILDING MATERIALS

DETERMINATION OF CALORIFIC POTENTIAL

1. SCOPE

This ISO Recommendation 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 ISO Recommendation.

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 AND DEFINITIONS

Symbol	Definition
Q_{gr}	gross calorific potential in kilojoules per kilogramme.
Q_{net}	net calorific potential in kilojoules per kilogramme.
E	water equivalent of the apparatus in kilogrammes, determined with an identical calibration test on benzoic acid (gross calorific value, $H_o = 26\,435$ kJ/kg).
W	mass in kilogrammes 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 kilogrammes.
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 kilogrammes of the additional substance present.
m_f	mass in kilogrammes of the substance used for firing the specimen.
H_{oa}	gross calorific value of the additional substance in kilojoules per kilogramme.
H_{of}	gross calorific value in kilojoules per kilogramme of the substance used for firing the specimen.*
w	proportional content of condensed water in the bomb after combustion.
q	latent heat of vaporization released by the water condensed in the bomb.

4. SAMPLING

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

5. TEST METHOD – 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 should be dried, its mass should be determined to the nearest 0.1 mg and it should be left for 1 hour in a furnace having a temperature of 900 °C. After cooling in a desiccator to the ambient temperature, a second weighing will show if the combustion was complete.

* 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

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 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 *Crucibles* 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 should be 2451.6 kN/m^2 (24.52 bar).

5.3 Preparation of test specimens

The sample should 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. Grind eventually to a finer powder as reduction goes further.

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 prescribed in clause 5.3.1. Prepare a specimen of each constituent having such a mass that the combined specimen obtained by mixing all 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 seiving or any other method. Treat the powders as indicated in clause 5.3.2.1.

5.3.2.3 If separation is impossible, proceed as for homogeneous materials, as specified in clause 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 described in clause 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 should be conditioned for 20 hours 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 cm^3 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 2451.6 kN/m^2 (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 should be determined to the nearest 1 g. This quantity should be the same as that used in the calibration test. The temperature of this water should 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 minutes, start temperature readings, which should be made at intervals of 1 minute for 5 minutes, or start the temperature recorder. This 5 minute 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 should 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 seconds 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"*. During this period, which lasts 5 minutes, note the temperature every minute 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 described in clause 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 clause 5.6.

5.6 **Calculation of results**

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

$$Q_{gr} = 4.1868\ \frac{(E+W)(t_m - t_i + c) - C}{m} \quad \text{kJ/kg}$$

(*a*) *Correction c* : This correction should be calculated using the following formula :

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

(*b*) *Correction C* : $C = \Sigma C_n$ $(n = 1, 2 \text{ etc.})$.

In this correction, when necessary, the following should 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) Correction for 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 2451.6 kN/m^2 in the presence of 5 cm^3 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.

* Sometimes known as the "after period".