
**Reaction-to-fire tests — Heat release,
smoke production and mass loss rate —**
Part 1:
Heat release rate (cone calorimeter method)

*Essais de réaction au feu — Débit calorifique, taux de dégagement de
fumée et taux de perte de masse —*

Partie 1: Débit calorifique (méthode au calorimètre conique)



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 part of ISO 5660 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 5660-1 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

This second edition cancels and replaces the first edition (ISO 5660-1:1993), which has been technically revised.

ISO 5660 consists of the following parts, under the general title *Reaction-to-fire tests — Heat release, smoke production and mass loss rate*:

- *Part 1: Heat release rate (cone calorimeter method)*
- *Part 2: Smoke production rate (dynamic measurement)*
- *Part 3: Guidance on heat and smoke release rate*

Annexes A, B, C, D, E and F of this part of ISO 5660 are for information only.

Reaction-to-fire tests — Heat release, smoke production and mass loss rate —

Part 1:

Heat release rate (cone calorimeter method)

1 Scope

This part of ISO 5660 specifies a method for assessing the heat release rate of a specimen exposed in the horizontal orientation to controlled levels of irradiance with an external igniter. The heat release rate is determined by measurement of the oxygen consumption derived from the oxygen concentration and the flow rate in the combustion product stream. The time to ignition (sustained flaming) is also measured in this test.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 5660. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 5660 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 554:1976, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 13943:2000, *Fire safety — Vocabulary*

ISO/TR 14697:1997, *Fire tests — Guidance on the choice of substrates for building products*

3 Terms and definitions

For the purposes of this part of ISO 5660, the terms and definitions given in ISO 13943 and the following apply.

3.1

essentially flat surface

surface whose irregularity from a plane does not exceed ± 1 mm

3.2

flashing

existence of flame on or over the surface of the specimen for periods of less than 1 s

3.3

ignition

onset of sustained flaming as defined in 3.10

3.4

irradiance

(at a point on a surface) quotient of the radiant flux incident on an infinitesimal element of surface containing the point, and the area of that element

NOTE Convective heating is negligible in the horizontal specimen orientation. For this reason, the term "irradiance" is used instead of "heat flux" throughout this part of ISO 5660 as it best indicates the essentially radiative mode of heat transfer.

3.5

material

single substance or uniformly dispersed mixture

EXAMPLE Metal, stone, timber, concrete, mineral fibre and polymers.

3.6

orientation

plane in which the exposed face of the specimen is located during testing, with either the vertical or horizontal face upwards

3.7

oxygen consumption principle

proportional relationship between the mass of oxygen consumed during combustion and the heat released

3.8

product

material, composite or assembly about which information is required

3.9

specimen

representative piece of the product which is to be tested together with any substrate or treatment

NOTE For certain types of product, for example products that contain an air gap or joints, it may not be possible to prepare specimens that are representative of the end-use conditions (see clause 7).

3.10

sustained flaming

existence of flame on or over the surface of the specimen for periods of over 10 s

3.11

transitory flaming

existence of flame on or over the surface of the specimen for periods of between 1 s and 10 s

4 Symbols

See Table 1.

Table 1 — Symbols and their designations

Symbol	Designation	Unit
A_s	Initially exposed surface area of the specimen	m^2
C	Orifice flow meter calibration constant	$m^{1/2} \cdot g^{1/2} \cdot K^{1/2}$
Δh_c	Net heat of combustion	$kJ \cdot g^{-1}$
$\Delta h_{c,eff}$	Effective net heat of combustion	$MJ \cdot kg^{-1}$
m	Mass of the specimen	g
Δm	Total mass loss	g
m_t	Mass of the specimen at the end of the test	g
m_s	Mass of the specimen at sustained flaming	g
$\dot{m}_{A,10-90}$	Average mass loss rate per unit area between 10 % and 90 % of mass loss	$g \cdot m^{-2} \cdot s^{-1}$
m_{10}	Mass of the specimen at 10 % of total mass loss	g
m_{90}	Mass of the specimen at 90 % of total mass loss	g
$\dot{m}$	Mass loss rate of specimen	$g \cdot s^{-1}$

Table 1 — Symbols and their designations (*continued*)

Symbol	Designation	Unit
$\dot{m}_e$	Mass flow rate in exhaust duct	$\text{kg} \cdot \text{s}^{-1}$
Δp	Orifice meter pressure differential	Pa
$\dot{q}$	Heat release rate	kW
$\dot{q}_A$	Heat release rate per unit area	$\text{kW} \cdot \text{m}^{-2}$
$\dot{q}_{A,\text{max}}$	Maximum value of the heat release rate per unit area	$\text{kW} \cdot \text{m}^{-2}$
$\dot{q}_{A,180}$	Average heat release rate per unit area over the period starting at t_{ig} and ending 180 s later	$\text{kW} \cdot \text{m}^{-2}$
$\dot{q}_{A,300}$	Average heat release rate per unit area over the period starting at t_{ig} and ending 300 s later	$\text{kW} \cdot \text{m}^{-2}$
$Q_{A,\text{tot}}$	Total heat released per unit area during the entire test	$\text{MJ} \cdot \text{m}^{-2}$
r_o	Stoichiometric oxygen/fuel mass ratio	1
t	Time	s
t_d	Delay time of the oxygen analyser	s
t_{ig}	Time to ignition (onset of sustained flaming)	s
Δt	Sampling time interval	s
t_{10}	Time at 10 % of total mass loss	s
t_{90}	Time at 90 % of total mass loss	s
T_e	Absolute temperature of gas at the orifice meter	K
X_{O_2}	Oxygen analyser reading, mole fraction of oxygen	1
$X_{\text{O}_2}^0$	Initial value of oxygen analyser reading	1
$X_{\text{O}_2}^1$	Oxygen analyser reading, before delay time correction	1

5 Principle

This test method is based on the observation that, generally, the net heat of combustion is proportional to the amount of oxygen required for combustion. The relationship is that approximately $13,1 \times 10^3$ kJ of heat are released per kilogram of oxygen consumed. Specimens in the test are burned under ambient air conditions, while being subjected to a predetermined external irradiance within the range of 0 kW/m² to 100 kW/m² and measurements are made of oxygen concentrations and exhaust gas flow rates.

The test method is used to assess the contribution that the product under test can make to the rate of evolution of heat during its involvement in fire. These properties are determined on small representative specimens.

6 Apparatus

A schematic representation of the apparatus is given in Figure 1. The individual components are described in detail in 6.1 to 6.5.

With minor modifications to the apparatus, specimens may be tested in the vertical orientation. Annex D gives guidance on these modifications.

6.1 Cone-shaped radiant electrical heater

The active element of the heater shall consist of an electrical heater rod, capable of delivering 5 000 W at the operating voltage, tightly wound into the shape of a truncated cone (see Figure 2). The heater shall be encased on the outside with a double-wall stainless-steel cone, filled with a refractory fibre blanket of nominal thickness 13 mm and nominal density 100 kg/m³. The irradiance from the heater shall be maintained at a preset level by controlling the average temperature of three thermocouples (type K stainless-steel sheathed thermocouples have proved suitable but Inconel or other high-performance materials are also acceptable), symmetrically positioned and in contact with, but not welded to, the heater element (see Figure 2). Either 3,0 mm outside diameter sheathed thermocouples with

exposed hot junction or 1,0 mm to 1,6 mm outside diameter sheathed thermocouples with unexposed hot junction shall be used. The heater shall be capable of producing irradiance on the surface of the specimen of up to 100 kW/m². The irradiance shall be uniform within the central 50 mm × 50 mm area of the exposed specimen surface, to within ± 2 %.

6.2 Radiation shield

The cone heater shall be provided with a removable radiation shield to protect the specimen from the irradiance prior to the start of a test. The shield shall be made of non-combustible material, with a total thickness not exceeding 12 mm. The shield shall be one of the following, either

- a) water-cooled and coated with a durable matt black finish of surface emissivity $\epsilon = 0,95 \pm 0,05$, or
- b) not water-cooled, which may be either metal with a reflective top surface or ceramic in order to minimize radiation transfer.

The shield shall be equipped with a handle or other suitable means for quick insertion and removal. The cone heater base plate shall be equipped with a mechanism for moving the shield into position.

6.3 Irradiance control

The irradiance control system shall be properly tuned so that it maintains the average temperature of the heater thermocouples during the calibration described in 10.1.2 at the preset level to within ± 10 °C.

6.4 Weighing device

The weighing device shall have an accuracy of ± 0,1 g or better, measured according to the calibration procedure described in 10.2.2. The weighing device shall be capable of measuring the mass of specimens of at least 500 g. The weighing device shall have a 10 % to 90 % response time of 4 s or less, as determined according to the calibration described in 10.1.3. The output of the weighing device shall not drift by more than 1 g over a 30-min period, as determined with the calibration described in 10.1.4.

6.5 Specimen holder

The specimen holder is shown in Figure 3. The specimen holder shall have the shape of a square pan with an opening of (106 ± 1) mm × (106 ± 1) mm at the top, and a depth of (25 ± 1) mm. The holder shall be constructed of stainless steel with a thickness of $(2,4 \pm 0,15)$ mm. It shall include a handle to facilitate insertion and removal, and a mechanism to ensure central location of the specimen under the heater and proper alignment with the weighing device. The bottom of the holder shall be lined with a layer of low density (nominal density 65 kg/m³) refractory fibre blanket with a thickness of at least 13 mm. The distance between the bottom surface of the cone heater and the top of the specimen shall be adjusted to be (25 ± 1) mm, except for dimensionally unstable materials for which the distance shall be (60 ± 1) mm (see 7.5).

6.6 Retainer frame

The frame shall be constructed of stainless steel with a thickness of $(1,9 \pm 0,1)$ mm, in the shape of a box with an inside dimension of each side (111 ± 1) mm and a height of (54 ± 1) mm. The opening for the specimen face shall be $(94,0 \pm 0,5)$ mm square as shown in Figure 4. The retainer frame shall have an appropriate means to secure it to the specimen holder with a specimen in position.

6.7 Exhaust gas system with flow measuring instrumentation

The exhaust gas system shall consist of a centrifugal exhaust fan rated for the operating temperatures, a hood, intake and exhaust ducts for the fan, and an orifice plate flow meter (see Figure 5). The distance between the bottom of the hood and the specimen surface shall be (210 ± 50) mm. The exhaust system shall be capable of developing flows up to 0,024 m³/s, under standard conditions of temperature and pressure. The recommended location of the fan is indicated on Figure 5. As an alternative, it is acceptable to locate the fan further downstream and to have the measuring orifice before the fan, provided that the requirements described in the remainder of this clause are fulfilled.

A restrictive orifice with an internal diameter of (57 ± 3) mm shall be located between the hood and the duct to promote mixing.

A ring sampler shall be located in the fan intake duct for gas sampling, (685 ± 15) mm from the hood (see Figure 5). The ring sampler shall contain 12 small holes with a diameter of $(2,2 \pm 0,1)$ mm, to average the stream composition, with the holes facing away from the flow to avoid clogging with soot.

The temperature of the gas stream shall be measured using a 1,0 mm to 1,6 mm outside diameter sheathed-junction thermocouple or a 3 mm outside diameter exposed-junction thermocouple positioned in the exhaust stack on the centreline and (100 ± 5) mm upstream from the measuring orifice plate.

The flow rate shall be determined by measuring the differential pressure across a sharp edge orifice [internal diameter (57 ± 3) mm, thickness $(1,6 \pm 0,3)$ mm] in the exhaust stack, at least 350 mm downstream from the fan, if the latter is located as shown on Figure 5. If the fan is located further downstream than indicated in Figure 5, it is acceptable to locate the orifice plate between the ring sampler and the fan. However, in that case the length of the straight duct section on both sides of the orifice plate shall be at least 350 mm.

6.8 Gas sampling apparatus

The gas sampling apparatus shall incorporate a pump, a filter to prevent entry of soot, a cold trap to remove most of the moisture, a by-pass system set to divert all flow except that required for the gas analysers, a further moisture trap and a trap for CO₂ removal. A schematic view of an example of the gas sampling apparatus is shown in Figure 6. Other arrangements which satisfy the requirements may be used. The transport delay time of the oxygen analyser, t_d , shall be determined according to 10.1.5, and shall not exceed 60 s.

NOTE If an (optional) CO₂ analyser is used, the equations to calculate the heat release rate can be different from those for the standard case (see clause 12 and annex F).

6.9 Ignition circuit

External ignition is accomplished by a spark plug powered from a 10 kV transformer or spark igniter. The spark plug shall have a gap of $(3,0 \pm 0,5)$ mm. The electrode length and location of the spark plug shall be such that the spark gap is located (13 ± 2) mm above the centre of the specimen, except for dimensionally unstable materials for which the distance shall be (48 ± 2) mm (see 7.5).

6.10 Ignition timer

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

6.11 Oxygen analyser

The oxygen analyser shall be of the paramagnetic type, with a range of at least 0 % oxygen to 25 % oxygen. The analyser shall exhibit a drift of not more than 50 parts per million of oxygen over a period of 30 min, and a noise of not more than 50 parts per million of oxygen during this 30-min period, as measured according to 10.1.6. Since oxygen analysers are sensitive to stream pressures, the stream pressure shall be regulated (upstream of the analyser) to minimize flow fluctuations, and the readings from the analyser compensated with an absolute pressure transducer to allow for atmospheric pressure variations. The analyser and the absolute pressure transducer shall be located in an isothermal environment. The temperature of the environment shall be maintained to within 2 °C of a preset value between 30 °C and 70 °C. The oxygen analyser shall have a 10 % to 90 % of full-scale response time of less than 12 s, as measured according to 10.1.5.

6.12 Heat flux meters

The working heat flux meter shall be used to calibrate the heater (see 10.2.5). It shall be positioned at a location equivalent to the centre of the specimen face during this calibration.

This heat flux meter shall be of the Schmidt-Boelter (thermopile) type with a design range of (100 ± 10) kW/m². The target receiving the heat shall be flat, circular, of approximately 12,5 mm in diameter and coated with a durable matt

black finish of surface emissivity $\epsilon = 0,95 \pm 0,05$. The target shall be water-cooled. A cooling temperature which would cause condensation of water on the target surface of the heat flux meter shall not be used.

Radiation shall not pass through any window before reaching the target. The instrument shall be robust, simple to set up and use, and stable in calibration. The instrument shall have an accuracy of within $\pm 3 \%$ and a repeatability to within $\pm 0,5 \%$.

The calibration of the working heat flux meter shall be checked according to 10.3.1, by comparison with two instruments of the same type as the working heat flux meter and of similar range held as reference standards and not used for any other purpose (see annex E). One of the reference standards shall be fully calibrated at a standardizing laboratory at yearly intervals.

6.13 Calibration burner

The calibration burner shall be constructed from tube with a square or circular orifice with an area of $(500 \pm 100) \text{ mm}^2$ covered with wire gauze through which the methane diffuses. The tube is packed with refractory fibre to improve uniformity of flow. The calibration burner is suitably connected to a metered supply of methane of at least 99,5 % purity. The accuracy of the flow meter shall be $\pm 2 \%$ of the readout, corresponding to a heat release rate of 5 kW. The accuracy verification shall be performed according to 10.3.3.

6.14 Data collection and analysis system

The data collection and analysis system shall have facilities for recording the output from the oxygen analyser, the orifice meter, the thermocouples and the weighing device. The data collection system shall have an accuracy corresponding to at least 50 parts per million of oxygen for the oxygen channel, $0,5^\circ\text{C}$ for the temperature measuring channels, 0,01 % of full-scale instrument output for all other instrument channels, and at least 0,1 % for time. The system shall be capable of recording data every second. The system shall be capable of storing a minimum of 720 data per parameter. The raw data recorded for each test shall be stored so that it can be recovered and used to check the accuracy of the software.

6.15 Optional side screens

For operational or safety reasons, it is permitted to guard the heater and sample holder with side screens. However, it shall be demonstrated that the presence of the screens does not affect the ignition time and heat release rate measurements according to the procedure described in 10.1.7.

If the screens form an enclosure, attention is drawn to the fact that there is a possible explosion hazard when the instrument is not operated under conditions prescribed by this part of ISO 5660, in particular for experiments in an oxygen-enriched atmosphere. If an explosion hazard exists, proper precautions shall be taken to protect the operator, e.g. by installing an explosion vent facing away from the operator.

7 Suitability of a product for testing

7.1 Surface characteristics

A product having one of the following properties is suitable for testing:

- a) an essentially flat exposed surface;
- b) a surface irregularity which is evenly distributed over the exposed surface provided that
 - 1) at least 50 % of the surface of a representative 100 mm square area lies within a depth of 10 mm from a plane taken across the highest points on the exposed surface, or
 - 2) for surfaces containing cracks, fissures or holes not exceeding 8 mm in width nor 10 mm in depth, the total area of such cracks, fissures or holes at the surface does not exceed 30 % of a representative 100 mm square area of the exposed surface.

When an exposed surface does not meet the requirements of either 7.1 a) or 7.1 b), the product shall be tested in a modified form complying as nearly as possible with the requirements given in 7.1. The test report shall state that the product has been tested in a modified form, and clearly describe the modification.

7.2 Asymmetrical products

A product submitted for this test can have faces which differ or can contain laminations of different materials arranged in a different order in relation to the two faces. If either of the faces can be exposed in use within a room, cavity or void, then both faces shall be tested.

7.3 Materials of short burning time

For specimens of short burning time (3 min or less), the heat release rate measurements shall be taken at not more than 2 s intervals. For longer burning times, 5 s intervals may be used.

7.4 Composite specimens

Composite specimens are suitable for testing, provided that they are prepared as specified in 8.3 and are exposed in a manner typical of end use conditions.

7.5 Dimensionally unstable materials

Samples that intumesce or deform so that they contact the spark plug prior to ignition, or the underside of the cone heater after ignition, shall be tested with the separation of 60 mm between the base plate of the cone heater and the upper surface of the specimen. In this case the heater calibration (see 10.2.5) shall be performed with the heat flux meter positioned 60 mm below the cone heater base plate. It must be stressed that the time to ignition measured with this separation is not comparable to that measured with the separation of 25 mm.

Other dimensionally unstable products, for example products that warp or shrink during testing, shall be restrained against excessive movement. This shall be accomplished with four tie wires, as described below. Metal wires of $(1,0 \pm 0,1)$ mm diameter and at least 350 mm long shall be used. The sample shall be prepared in the standard way as described in clause 8. A tie wire is then looped around the sample holder and retainer frame assembly, so that it is parallel to and approximately 20 mm away from one of the four sides of the assembly. The ends of the wire are twisted together such that the wire is pulled firmly against the retainer frame. Excess wire is trimmed from the twisted section before testing. The three remaining wires shall be fitted around the specimen holder and retainer frame assembly in a similar manner, parallel to the three remaining sides.

8 Specimen construction and preparation

8.1 Specimens

8.1.1 Unless otherwise specified, three specimens shall be tested at each level of irradiance selected and for each different exposed surface.

8.1.2 The specimens shall be representative of the product and shall be square with sides measuring 100 ± 2 mm.

8.1.3 Products with a normal thickness of 50 mm or less shall be tested using their full thickness.

8.1.4 For products with a normal thickness of greater than 50 mm, the requisite specimens shall be obtained by cutting away the unexposed face to reduce the thickness to 50 mm.

8.1.5 When cutting specimens from products with irregular surfaces, the highest point on the surface shall be arranged to occur at the centre of the specimen.

8.1.6 Assemblies shall be tested as specified in 8.1.3 or 8.1.4 as appropriate. However, where thin materials or composites are used in the fabrication of an assembly, the nature of any underlying construction can significantly affect the ignition and burning characteristics of the exposed surface.

The influence of the underlying layers shall be understood and care taken to ensure that the test result obtained on any assembly is relevant to its use in practice.

When the product is a material or composite which would normally be attached to a well-defined substrate, it shall be tested in conjunction with that substrate using the recommended fixing technique, for example bonded with the appropriate adhesive or mechanically fixed. In the absence of a unique or well-defined substrate, an appropriate substrate for testing shall be selected in accordance with ISO/TR 14697.

8.1.7 Products that are thinner than 6 mm shall be tested with a substrate representative of end-use conditions, such that the total specimen thickness is 6 mm or more.

8.2 Conditioning of specimens

Before the test, specimens shall be conditioned to constant mass at a temperature of $(23 \pm 2) ^\circ\text{C}$, and a relative humidity of $(50 \pm 5) \%$ in accordance with ISO 554.

Constant mass is considered to be reached when two successive weighing operations, carried out at an interval of 24 h, do not differ by more than 0,1 % of the mass of the test piece or 0,1 g, whichever is the greater.

Materials such as polyamides, which require more than one week in conditioning to reach equilibrium may be tested after conditioning in accordance with ISO 291^[1]. This period shall be not less than one week, and shall be described in the test report.

8.3 Preparation

8.3.1 Specimen wrapping

A conditioned specimen shall be wrapped in a single layer of aluminum foil, of 0,025 mm to 0,04 mm thickness, with the shiny side towards the specimen. The aluminium foil shall be pre-cut to a size to cover the bottom and sides of the specimen and extend 3 mm or more beyond the upper surface of the specimen. The specimen shall be placed in the middle of the foil and the bottom and sides shall be wrapped. The excess foil above the top surface shall be cut if necessary so that it does not extend more than 3 mm above the top surface of the specimen. The excess foil at the corners shall be folded around the corners to form a seal around the top surface of the specimen. After wrapping, the wrapped specimen shall be placed in the specimen holder and covered by a retainer frame. No aluminium foil shall be visible after the procedure is completed.

For soft specimens, a dummy specimen having the same thickness as the specimen to be tested may be used to pre-shape the aluminium foil.

8.3.2 Specimen preparation

All specimens shall be tested with the retainer frame shown in Figure 4. The following steps shall be taken to prepare a specimen for testing:

- a) put the retainer frame on a flat surface facing downwards;
- b) insert the foil-wrapped specimen into the frame with the exposed surface facing downwards;
- c) put layers of refractory fibre blanket (nominal thickness 13 mm, nominal density 65 kg/m³) on top until at least one full layer, and not more than two layers, extend above the rim of the frame;
- d) fit the sample holder into the frame on top of the refractory fibre and press down;
- e) secure the retainer frame to the specimen holder.

9 Test environment

The apparatus shall be located in an essentially draught-free environment in an atmosphere of relative humidity of between 20 % and 80 % and a temperature between 15 °C and 30 °C.

10 Calibration

10.1 Preliminary calibrations

10.1.1 General

The calibrations in this section, except for that in 10.1.7, shall be performed before conducting experiments, when commissioning a Cone calorimeter; or after maintenance, repair or replacement of the heater assembly or irradiance control system (10.1.2), the weighing device (10.1.3 and 10.1.4), the oxygen analyser or other major components of the gas analysis system (10.1.5 and 10.1.6). The calibration tests to determine the effect of side screens in 10.1.7 are conducted at the time the screens are installed. For a new instrument that is delivered with side screens, this shall be done by the manufacturer.

10.1.2 Irradiance control system response characteristics

Turn on power to the cone heater and the exhaust fan. Set an irradiance of $(50 \pm 1) \text{ kW/m}^2$, and an exhaust flow rate of $(0,024 \pm 0,002) \text{ m}^3/\text{s}$. After reaching equilibrium of the heater, record the average heater temperature. Test a specimen of black poly(methyl methacrylate) (PMMA) according to the procedure in clause 11. The PMMA specimen shall have a thickness of at least 6 mm. The average heat release rate recorded over the first 3 min following ignition shall be approximately 530 kW/m^2 . During the test, record the average heater temperature at 5 s intervals.

10.1.3 Weighing device response time

The cone heater shall not be turned on for this calibration. Place an empty specimen holder with a $(250 \pm 25) \text{ g}$ non-combustible weightpiece on the weighing device. The weightpiece accounts for the retainer frame, which is not used during this calibration. Measure the weighing device output, and mechanically or electronically adjust the value to zero. Gently add a second non-combustible weightpiece with a mass of $(250 \pm 25) \text{ g}$ on the holder and record the weighing device output. After equilibrium is reached, gently remove the second weightpiece from the holder, and again record the weighing device output. Determine the response time of the weighing device as the average of the times for the weighing device output to change from 10 % to 90 % of its ultimate deflection.

10.1.4 Weighing device output drift

Set the height of the cone heater to the same position as when testing a specimen with the retainer frame. Place a thermal barrier on the weighing device. Turn on power to the exhaust fan and cone heater. Set an exhaust flow rate of $(0,024 \pm 0,002) \text{ m}^3/\text{s}$ and an irradiance of $(50 \pm 1) \text{ kW/m}^2$. After reaching equilibrium of the heater temperature, remove the thermal barrier and place an empty specimen holder with a $(250 \pm 25) \text{ g}$ weightpiece on the weighing device. The weightpiece accounts for the retainer frame, which is not used during this calibration. After equilibrium is reached, measure the weighing device output and mechanically or electronically adjust the value to zero. Gently add a second weightpiece with a mass of $(250 \pm 25) \text{ g}$ on the specimen holder. After equilibrium is reached, record the weighing device output. After 30 min, record the weighing device output. Calculate the drift of the weighing device output as the absolute value of the difference of the initial and final values.

10.1.5 Oxygen analyser delay and response times

The cone heater shall not be turned on for this calibration. Turn on the exhaust fan, and set an exhaust flow rate of $(0,024 \pm 0,002) \text{ m}^3/\text{s}$. Determine the delay time of the oxygen analyser by delivering a methane flow rate approximately equivalent to 5 kW to the calibration burner. Light the burner outside the hood and allow the flame to

stabilize. Quickly introduce the burner underneath the hood, and leave the burner in position for 3 min. Then, remove the burner from underneath the hood and turn off the methane supply. Record the output of the analyser from the moment of insertion of the burner underneath the hood, until 3 min after removal of the burner. The turn-on delay is the time difference between insertion of the burner and the oxygen reading reaching 50 % of its ultimate deflection. Calculate the turn-off delay similarly. The delay time t_d is the average of at least three turn-on and turn-off delays. The oxygen concentration at a given time shall be taken as the concentration registered after the time interval t_d .

The response time of the oxygen analyser is calculated as the average for the turn-on and turn-off experiments of the time for the oxygen analyser output to change from 10 % to 90 % of its ultimate deflection.

NOTE For the purpose of measurement of the oxygen analyser delay and response time, the methane flow rate need not be controlled accurately, because the delay and response time is not sensitive to the oxygen level.

10.1.6 Oxygen analyser output noise and drift

The cone heater shall not be turned on for this calibration. Turn on the exhaust fan, and set an exhaust flow rate of $(0,024 \pm 0,002)$ m³/s. Feed the oxygen analyser with oxygen-free nitrogen gas. After 60 min, switch to dried ambient air from the exhaust duct at the normal flow rate and pressure as for the sample gases. After reaching equilibrium, adjust the oxygen analyser output to $(20,95 \pm 0,01)$ %. Start recording the oxygen analyser output at 5 s intervals for a period of 30 min. Determine the drift by use of a least-squares fitting procedure to fit a straight line through the data points. For the straight line fit, the absolute value of the difference between the reading at 0 min and at 30 min represents the short-term drift. Determine the noise by computing the root-mean-square deviation around the linear trend line according to the following formula:

$$\text{r.m.s.} = \sqrt{\frac{\sum_{i=1}^n x_i^2}{n}}$$

where x_i is the absolute difference between the data point and the linear trend line.

Record this r.m.s. noise value in terms of parts per million of oxygen.

10.1.7 Effect of side screens

To evaluate the effect of side screens on the test results, six specimens of black cast poly(methyl methacrylate) (PMMA) of thickness $(25 \pm 0,5)$ mm shall be tested at (50 ± 1) kW/m² according to the procedure described in clause 11. The first three tests shall be conducted with the screens removed, the remaining three tests with the screens in place. The screens are permitted if the differences between the average values of t_{ig} , $\dot{q}_{A,180}$ and $\dot{q}_{A,max}$ for the two test series are found to be statistically insignificant according to a two-sided t-test at a significance level of 5 %. This t-test shall be performed for the three variables (t_{ig} , $\dot{q}_{A,180}$, and $\dot{q}_{A,max}$) according to the following procedure:

a) for the two series of three tests, calculate the averages via

$$\bar{x} = \frac{\sum_{i=1}^3 x_i}{3} \quad (1)$$

and

$$\bar{y} = \frac{\sum_{i=1}^3 y_i}{3} \quad (2)$$

b) calculate the pooled standard deviation, s_p , from

$$s_p = \sqrt{\frac{\sum_{i=1}^3 (x_i - \bar{x})^2 + \sum_{i=1}^3 (y_i - \bar{y})^2}{4}} \quad (3)$$

c) calculate the t-test statistic as

$$t_s = \left| \frac{\bar{x} - \bar{y}}{0,816\,5s_p} \right| \quad (4)$$

The t-test is successful if the value of the test statistic does not exceed 2,776, or if the two averages are equal.

10.2 Operating calibrations

10.2.1 General

The following calibrations shall be performed at the start of testing each day, in the order given below. The heater calibration shall also be performed when changing to a different irradiance level.

10.2.2 Weighing device accuracy

The weighing device shall be calibrated with standard weightpieces in the range of the test specimen mass. The cone heater shall be turned off and the apparatus shall be cooled down to ambient temperature before this calibration is performed. Place an empty specimen holder with a (250 ± 25) g weightpiece on the weighing device. The weightpiece accounts for the retainer frame, which is not used during this calibration. Measure the weighing device output, and mechanically or electronically adjust the value to zero. Gently add a weight piece with a mass between 50 g and 200 g on the holder and measure the weighing device output after it reaches a steady value. Repeat this procedure at least four times after adding weightpieces of the same mass range. At the end of the calibration, the total mass of all weightpieces on the holder shall be at least 500 g. The accuracy of the weighing device is determined as the maximum difference between the mass of the weightpieces and the weighing device output recorded during the calibration.

10.2.3 Oxygen analyser

Zero and calibrate the oxygen analyser. This calibration may be performed with the cone heater operating or not, but shall not be performed during heater warm-up. Turn on the exhaust fan, and set an exhaust flow rate of $(0,024 \pm 0,002)$ m³/s. For zeroing, feed the analyser with oxygen-free nitrogen gas, with the same flow rate and pressure as for the sample gases. Adjust the analyser response to $(0,00 \pm 0,01)$ %. Calibration shall be similarly achieved using dried ambient air and adjusting for a response of $(20,95 \pm 0,01)$ %. Carefully monitor analyser flow rates and set them to be equal to the flow rate used when testing specimens. After each specimen has been tested, ensure that a response level of $(20,95 \pm 0,01)$ % is obtained using dried ambient air.

10.2.4 Heat release rate calibration

Perform a heat release rate calibration to determine the orifice constant C . This calibration may be performed with the cone heater operating or not, but shall not be performed during heater warm-up. Turn on the exhaust fan, and set an exhaust flow rate of $(0,024 \pm 0,002)$ m³/s. Start collecting baseline data at 5-s intervals for a period of at least 1-min. Introduce methane into the calibration burner using a calibrated flow meter at a flow rate corresponding to $\dot{q}_b = (5 \pm 0,5)$ kW based on the net heat of combustion of methane $(50,0 \times 10^3)$ kJ/kg. After the outputs from all instruments reach equilibrium, collect data at 5-s intervals over a 3-min period. Calculate the orifice constant C according to equation (5) in clause 12, using averages over the 3-min period of the measured values of $\dot{q}_b$, T_e , Δp

and X_{O_2} . $X_{O_2}^0$ is determined as the average of the oxygen analyser output measured during the 1-min baseline measurements.

An alternative procedure for performing this calibration consists of burning a suitable liquid fuel (e.g. ethanol) in a special pan that is placed on the weighing device. The average theoretical heat release rate is then obtained as the total mass of fuel burnt multiplied by the net heat of combustion of the fuel, and divided by the duration of flaming.

10.2.5 Heater calibration

At the start of testing each day or when changing to a different irradiance level, adjust the irradiance control system so that the conical heater produces the required irradiance to within $\pm 2\%$, as measured by the heat flux meter. No specimen or specimen holder shall be used when the heat flux meter is inserted into the calibration position. Operate the cone heater for at least 10 min when stable at set point, and ensure that the controller is within its proportional band before beginning this calibration.

10.3 Less frequent calibrations

10.3.1 Operating heat flux meter calibration

At maximum intervals of 100 working hours, check the operating heat flux meter against the reference heat flux meter using one of the procedures described in annex E. Comparisons shall be made at irradiance levels of (10, 25, 35, 50, 65, 75 and 100) kW/m². The readings from the two meters shall agree to within $\pm 2\%$. If the operating heat flux meter reading is found to disagree with that of the reference meter by a constant factor (to within a $\pm 2\%$ spread) over the whole flux range, a new calibration factor is established for the operating heat flux meter and used for the heater calibration described in 10.2.5. If the operating heat flux meter cannot be brought to within a $\pm 2\%$ agreement over the entire range by the use of a single, new factor, the operating meter shall be replaced.

10.3.2 Linearity of heat release rate measurements

At maximum intervals of 100 working hours, with the instrument calibrated at 5 kW according to 10.2.5, perform a further calibration with a flow rate corresponding to 1 kW $\pm 10\%$ and 3 kW $\pm 10\%$, using the basic procedure as described in 10.2.5. With the value for C from the 5 kW calibration, the measured heat release rate at 1 kW and 3 kW shall be within $\pm 5\%$ of the set value.

10.3.3 Accuracy of calibration burner flow meter

The accuracy of the calibration burner flow meter shall be verified every 6 months or when the calibration factor determined according to 10.2.4 differs by more than 5 % from the value obtained during the first heat release rate calibration following the previous flow meter verification. To verify the accuracy of the flow meter, perform the burner calibration described in 10.2.4, with a reference flow meter in series with the operating flow meter. During the 3-min period of data collection, both flow meters shall agree to within $\pm 3\%$. If the difference between the two measurements exceeds $\pm 3\%$, the operating flow meter shall be recalibrated as recommended by the manufacturer.

11 Test procedure

11.1 General precautions

WARNING — So that suitable precautions are taken to safeguard health, the attention of all concerned in fire tests is drawn to the possibility that toxic or harmful gases can be evolved during exposure of test specimens.

The test procedures involve high temperatures and combustion processes. Therefore, hazards can exist such as burns or the ignition of extraneous objects or clothing. The operator shall use protective gloves for insertion and removal of test specimens. Neither the cone heater nor the associated fixtures shall be touched while hot except with

the use of protective gloves. Care shall be taken never to touch the spark igniter which carries a substantial potential (10 kV). The exhaust system of the apparatus shall be checked for proper operation before testing and shall discharge into a building exhaust system with adequate capacity. The possibility of the violent ejection of molten hot material or sharp fragments from some kinds of specimens when irradiated cannot totally be discounted and it is therefore essential that eye protection be worn.

11.2 Initial preparation

11.2.1 Check the CO₂ trap and the final moisture trap. Replace the sorbent if necessary. Drain any accumulated water in the cold trap separation chamber. The normal operating temperature of the cold trap shall not exceed 4 °C.

If any of the traps or filters in the gas sampling system line have been opened during the check, the gas sampling system should be checked for leaks (with the sample pump on), e.g. by introducing pure nitrogen, at the same flow rate and pressure as for the sample gases, from a nitrogen source connected as close as possible to the ring sampler. The oxygen analyser should then read zero.

11.2.2 Adjust the distance between the base plate of the cone heater and the upper surface of the specimen as specified in 6.5 or 7.5.

11.2.3 Turn on power to the cone heater (see A.4.1) and the exhaust fan. Power to the gas analysers, weighing device and pressure transducer shall not be turned off on a daily basis.

11.2.4 Set an exhaust flow rate of $(0,024 \pm 0,002) \text{ m}^3/\text{s}$.

11.2.5 Perform the required calibration procedures specified in 10.2. Put a thermal barrier on top of the weighing device (for example, an empty specimen holder with refractory fibre blanket or water cooled radiation shield) in place during warm up and between tests to avoid excessive heat transmission to the weighing device.

11.3 Procedure

11.3.1 Start data collection. Collect 1 min of baseline data. The standard scan interval is 5 s, unless a short burning time is anticipated (see 7.3).

11.3.2 Insert the radiation shield in position (see 6.3). Remove the thermal barrier protecting the weighing device (see 11.2.5). Place the specimen holder and specimen, prepared according to 8.3, on the weighing device.

The radiation shield shall be cooler than 100 °C immediately prior to the insertion.

11.3.3 Insert the spark plug and remove the radiation shield in the correct sequence according to the type of shield that is used, as described below.

For type a) shields (see 6.2), remove the shield and start the test. Within 1 s of removing the shield, insert and power the igniter.

For type b) shields (see 6.2), remove the shield within 10 s after the insertion and start the test. Within 1 s of removing the shield, insert and power the igniter.

11.3.4 Record the times when flashing or transitory flaming occurs. When sustained flaming occurs, record the time, turn off the spark, and remove the spark igniter. If the flame extinguishes after turning off the spark, re-insert the spark igniter and turn on the spark within 5 s, and do not remove the spark until the entire test is completed. Report these events in the test report (clause 13).

11.3.5 Collect all data until:

- a) 32 min after the time to sustained flaming (the 32 min consist of a 30-min test period and an additional 2-min post-test period to collect data that will be time-shifted),
- b) 30 min have elapsed and the specimen has not ignited,

- c) X_{O_2} returns to the pretest value within 100 parts per million of oxygen concentration for 10 min, or
- d) the mass of the specimen becomes zero,

whichever occurs first, but in any case, minimum test duration shall be 5 min. Observe and record physical changes to the sample such as melting, swelling and cracking.

11.3.6 Remove specimen and specimen holder. Put a thermal barrier on top of the weighing device.

11.3.7 Three specimens shall be tested and reported as described in clause 13. The 180 s mean heat release readings shall be compared for the three specimens. If any of these mean readings differ by more than 10 % from the arithmetic mean of the three readings, then a further set of three specimens shall be tested. In such cases, the arithmetic mean of the set of six readings shall be reported.

NOTE The test data have limited validity if the specimen melts sufficiently to overflow the sample holder, if explosive spalling occurs, or if the specimen swells excessively and touches the spark igniter or the heater base plate.

12 Calculations

12.1 General

The equations in this clause assume that only O_2 is measured as indicated on the gas analysis system in Figure 6. Appropriate equations for cases where additional gas analysis equipment (CO_2 , CO and possibly H_2O) is used and CO_2 is not removed from the O_2 sampling lines can be found in annex F. If CO_2 is removed from the O_2 sampling lines (even when CO_2 is separately measured), then to equations (5) to (7) shall be used.

12.2 Calibration constant for oxygen consumption analysis

The heat release calibration specified in 10.2.4 shall be performed daily to check for the proper operation of the instrument and to compensate for minor changes in determination of mass flow. A calibration more than 5 % different from the previous one is not normal and suggests instrument malfunction. The calibration constant, C , is calculated using

$$C = \frac{\dot{q}_b}{(12,54 \times 10^3) (1,10)} \sqrt{\frac{T_e}{\Delta p}} \frac{1,105 - 1,5X_{O_2}}{X_{O_2}^0 - X_{O_2}} \quad (5)$$

where $\dot{q}_b$ corresponds to the rate of heat release (in kW) of the methane supplied (see 10.2.4), $(12,54 \times 10^3)$ kJ/kg is $\Delta h_c/r_o$ for methane, 1,10 is the ratio of the molecular masses of oxygen and air.

12.3 Heat release rate

12.3.1 Prior to performing other calculations, calculate the oxygen analyser reading from the recorded analyser data and the delay time, t_d , using the following equation:

$$X_{O_2}(t) = X_{O_2}^1(t + t_d) \quad (6)$$

12.3.2 Calculate the heat release rate, $\dot{q}(t)$, from

$$\dot{q}(t) = (\Delta h_c/r_o) (1,10) C \sqrt{\frac{\Delta p}{T_e}} \cdot \frac{X_{O_2}^0 - X_{O_2}}{1,105 - 1,5X_{O_2}} \quad (7)$$

where $\Delta h_c/r_o$ for the specimen is taken as $(13,1 \times 10^3)$ kJ/kg, unless a more accurate value is known, and $X_{O_2}^0$ is determined as the average of the oxygen analyser output measured during the 1-min baseline measurements.

12.3.3 Heat release rate per unit area can be obtained from

$$\dot{q}_A(t) = \dot{q}(t)/A_s \quad (8)$$

where A_s is the initially exposed area of the sample, 0,008 8 m².

12.4 Exhaust duct flow rate

The mass flow rate, in grams per second, in the exhaust duct is given by

$$\dot{m}_e = C \sqrt{\frac{\Delta p}{T_e}} \quad (9)$$

12.5 Mass loss rate

12.5.1 The mass loss rate, $-\dot{m}$, at each time interval can be calculated using the following five-point numerical differentiation equations.

For the first scan ($i = 0$):

$$-\left[\dot{m}\right]_{i=0} = \frac{25m_0 - 48m_1 + 36m_2 - 16m_3 + 3m_4}{12\Delta t} \quad (10)$$

For the second scan ($i = 1$):

$$-\left[\dot{m}\right]_{i=1} = \frac{3m_0 + 10m_1 - 18m_2 + 6m_3 - m_4}{12\Delta t} \quad (11)$$

For any scan for which $1 < i < n - 1$ (where n is the total number of scans):

$$-\left[\dot{m}\right]_i = \frac{-m_{i-2} + 8m_{i-1} - 8m_{i+1} + m_{i+2}}{12\Delta t} \quad (12)$$

For the next to last scan ($i = n - 1$):

$$-\left[\dot{m}\right]_{i=n-1} = \frac{-3m_n - 10m_{n-1} + 18m_{n-2} - 6m_{n-3} + m_{n-4}}{12\Delta t} \quad (13)$$

For the last scan ($i = n$):

$$-\left[\dot{m}\right]_{i=n} = \frac{-25m_n + 48m_{n-1} - 36m_{n-2} + 16m_{n-3} - 3m_{n-4}}{12\Delta t} \quad (14)$$

12.5.2 The mass loss rate which includes the "main" burning period, i.e. from 10 % of ultimate mass loss being lost to 90 %, is given by

$$\dot{m}_{A,10-90} = \frac{m_{10} - m_{90}}{t_{90} - t_{10}} \times \frac{1}{A_s} \quad (15)$$

where

$$\Delta m = m_s - m_f$$

$$m_{10} = m_s - 0,10\Delta m$$

$$m_{90} = m_s - 0,90\Delta m$$

NOTE Equations for the effective heat of combustion, $\Delta h_{c,eff}$, are given in annex C.

13 Test report

These test results relate only to the behaviour of the test specimens 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.

The test report shall be as comprehensive as possible and shall include any observations made during the test and comments on any difficulties experienced during testing. The units for all measurements shall be clearly stated in the report. Certain units convenient for reporting are suggested below.

The following essential information shall also be given in the test report:

- a) name and address of test laboratory;
- b) name and address of sponsor;
- c) name and address of manufacturer/supplier;
- d) date of the test;
- e) operator;
- f) trade name and specimen identification code or number;
- g) composition or generic identification;
- h) specimen thickness¹⁾ expressed in millimetres, and mass¹⁾ expressed in grams; with composites and assemblies, a nominal thickness and density of each of the components shall be given, together with the apparent (overall) density of the whole;
- i) colour of the specimens;
- j) details of specimen preparation by the testing laboratory;
- k) specimen mounting, face tested, and any special mounting procedures (i.e. for intumescent specimen) that were used;
- l) orifice flow rate calibration constant C' ;
- m) irradiance¹⁾ expressed in kilowatts per square metre, and exhaust system flow rate¹⁾ expressed in cubic metres per second;
- n) number of replicate specimens tested under the same conditions (this shall be a minimum of three, except for exploratory testing);
- o) time to sustained flaming¹⁾, expressed in seconds;
- p) test duration¹⁾, i.e. the time between the start of the test and the end according to 11.3.5, expressed in seconds;
- q) heat release rate (per unit area)¹⁾ expressed in kilowatts per square metre, represented as a curve, recorded for the entire test;

1) Report these items for each specimen.

- r) average values¹⁾ for the first 180 s ($\dot{q}_{A,180}$) and 300 s ($\dot{q}_{A,300}$) after ignition, or for other appropriate periods and peak¹⁾ $\dot{q}_A$ ($\dot{q}_{A,max}$) values, expressed in kilowatts per square metre.

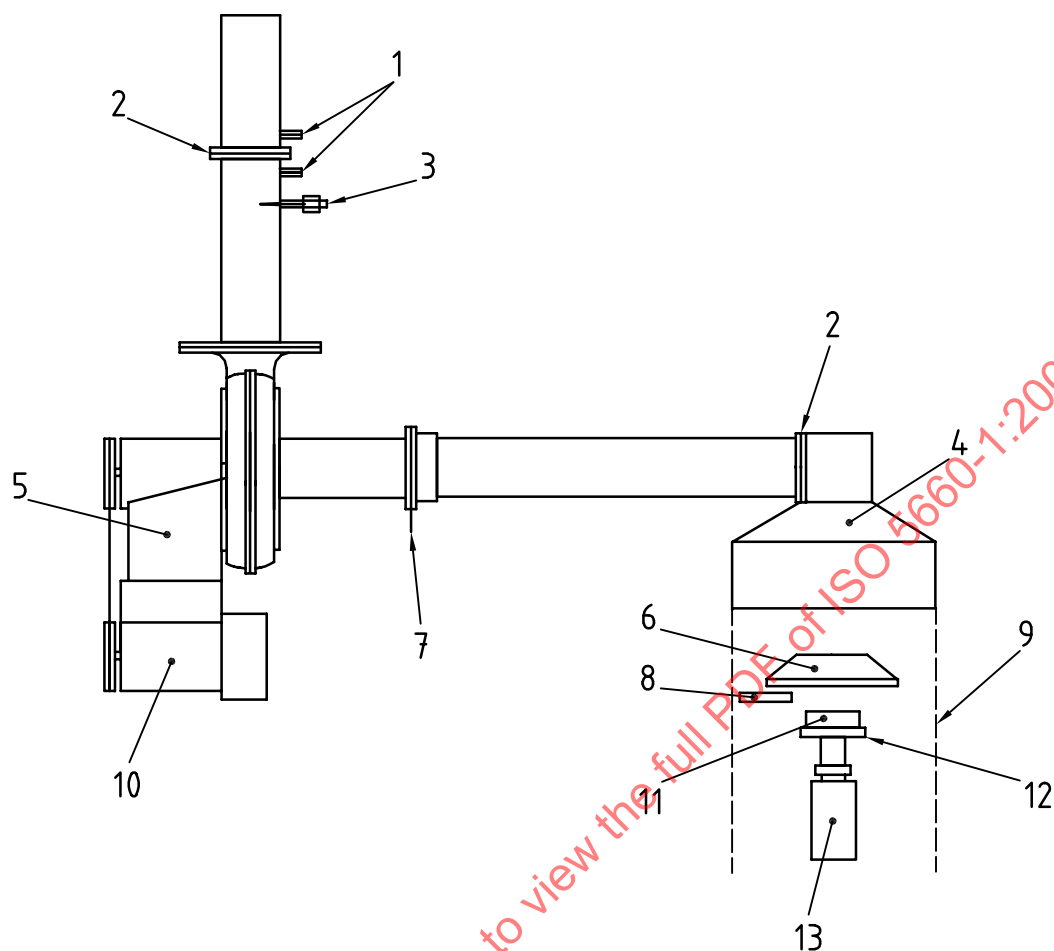
For specimens which do not show sustained flaming, report the above quantities tabulated for periods beginning with the next reading after the last negative heat release rate reading at the beginning of the test. Certain specimens do not show visible, sustained flaming, but do indicate non-zero heat release rate values. There will be negative readings, in general, since before the specimen starts burning the output is $0 \pm \text{noise}$.

Average heat release rate values shall be calculated using the trapezium rule for integration. For example, with a 5 s data collection interval, $\dot{q}_{A,180}$ is obtained as follows:

- 1) sum the rate of heat release rate values for 35 scans following the scan closest to ignition or the first scan after the last negative value; if the test is completed before the 180 s period has elapsed, use the test average instead;
 - 2) add half of the heat release rate measured at the scan closest to ignition or the first scan after the last negative value, and at the 36th scan after the scan closest to ignition or after the first scan after the last negative value;
 - 3) multiply the sum obtained in step 2 by the scan interval (5) and divide by 180;
- s) total heat released by the specimen¹⁾, expressed in megajoules per square metre. The total heat shall be computed beginning at the next reading after the last negative heat release rate reading occurred at the beginning of the test, and continuing until the final reading recorded for the test.

The total heat release can also be computed by using the trapezium rule to calculate integrated values. In this case, the first scan to be used is the one after the last negative heat release rate reading occurring at the beginning of the test;

- t) mass¹⁾ at sustained flaming, m_s , and mass remaining after the test, m_f , both expressed in grams;
- u) sample mass loss¹⁾ expressed in grams per square metre, and the average rate of specimen mass loss, $\dot{m}$, expressed in grams per square metre second ($\text{g/m}^2 \cdot \text{s}$), computed over the period between ignition and the end of the test;
- v) average rate of specimen mass loss per unit area¹⁾, $\dot{m}_{A,10-90}$, expressed in grams per square metre second ($\text{g m}^{-2} \text{s}^{-1}$), computed over the period between 10 % and 90 % of mass loss;
- w) values determined in items o), p), r), s), t), u) and v) averaged for all replicates;
- x) additional observations¹⁾, such as transitory flaming or flashing;
- y) difficulties encountered in testing¹⁾, if any.

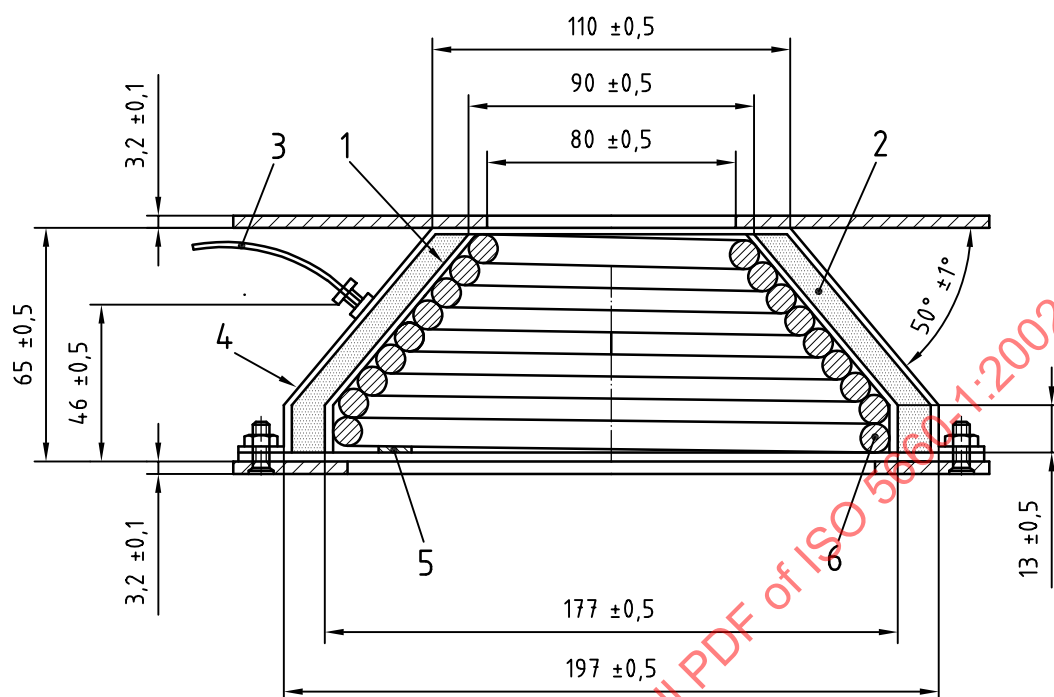


Key

- 1 Pressure ports
- 2 Orifice plate
- 3 Thermocouple (located on stack centreline)
- 4 Hood
- 5 Blower
- 6 Heater
- 7 Gas sampling ring probe
- 8 Spark plug
- 9 Optional screens
- 10 Blower motor
- 11 Retainer frame and specimen
- 12 Specimen holder
- 13 Weighing device

Figure 1 — Apparatus

Dimensions in millimetres

**Key**

- 1 Inner shell
- 2 Refractory fibre packing
- 3 Thermocouple
- 4 Outer shell
- 5 Spacer block
- 6 Heating element

Figure 2 — Cone heater

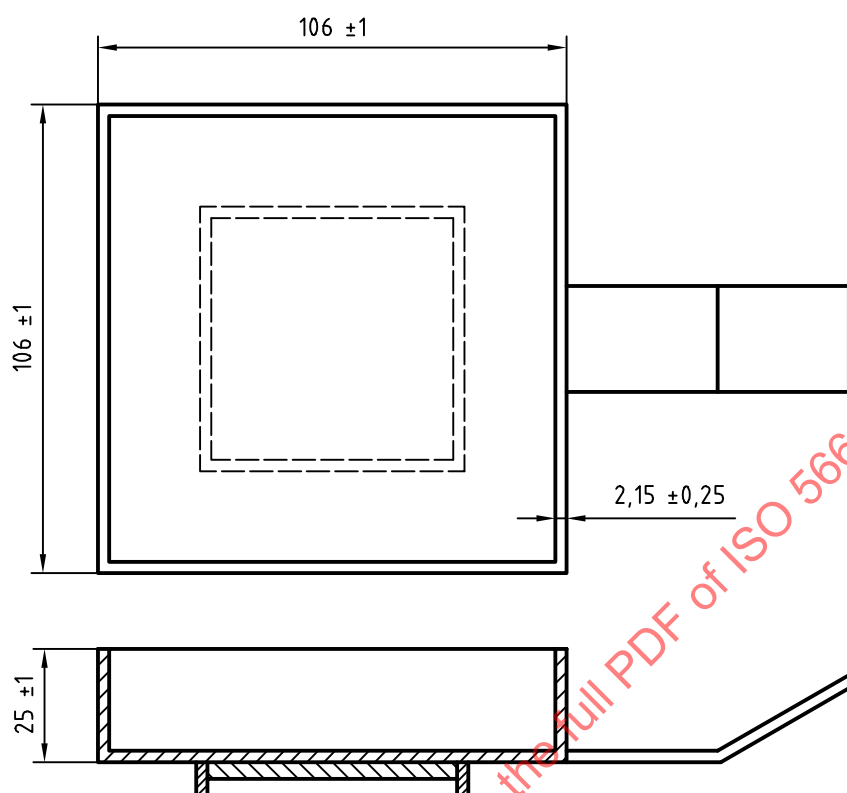
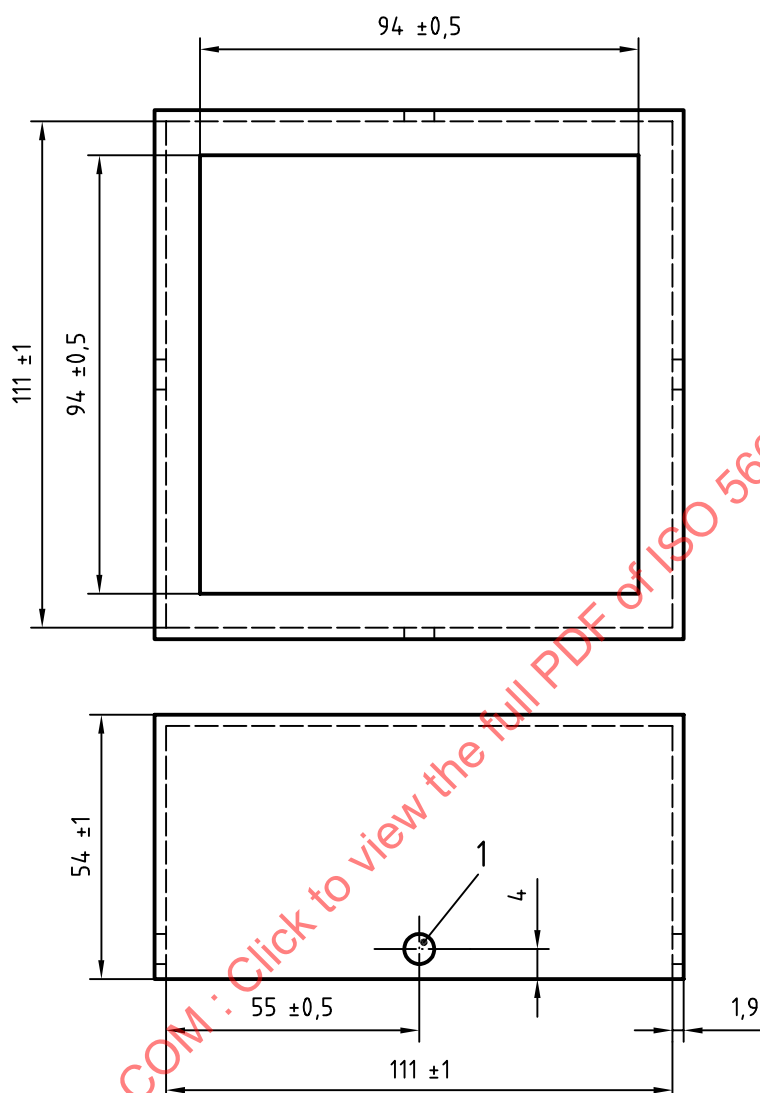


Figure 3 — Specimen holder

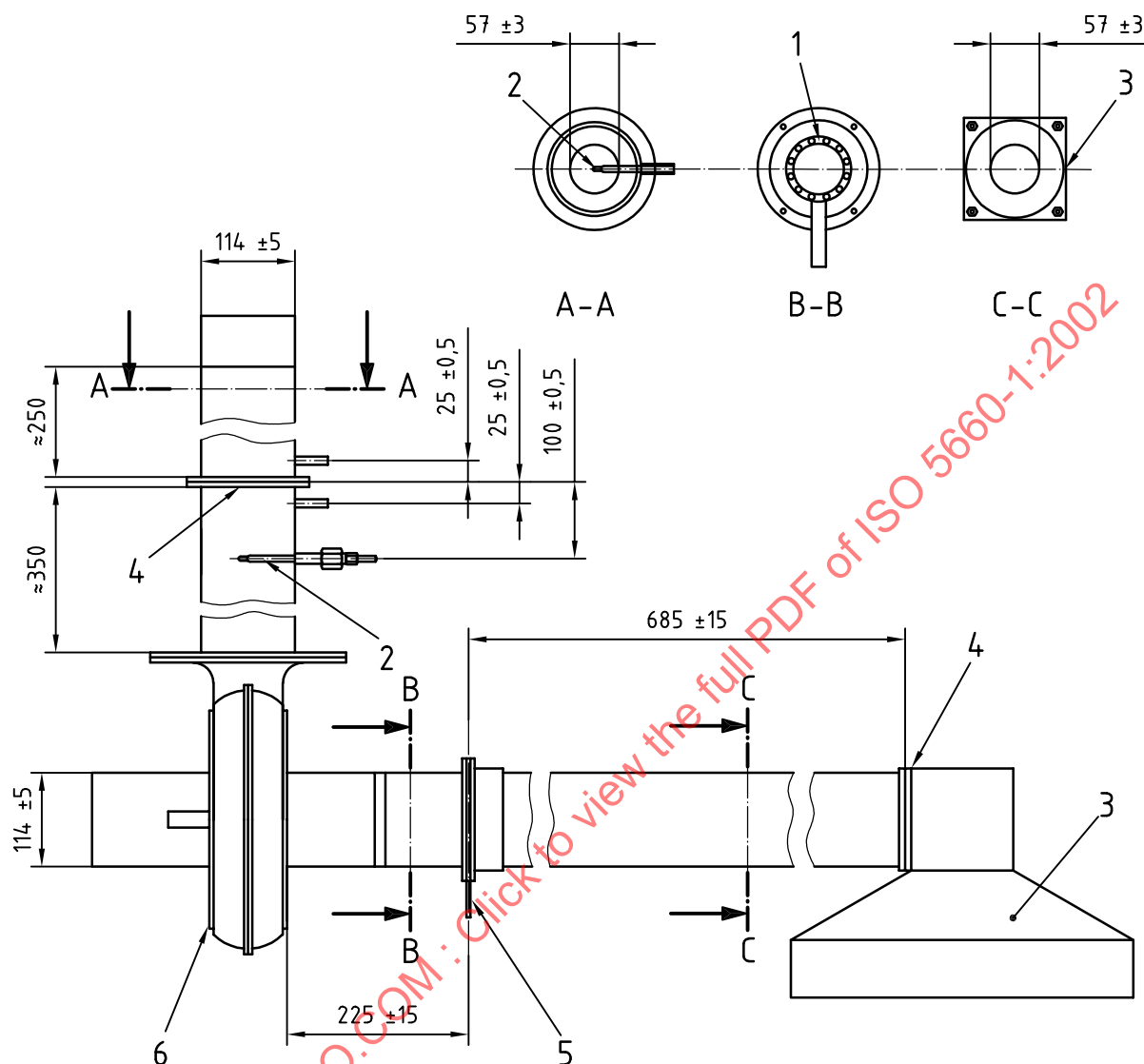
Dimensions in millimetres

**Key**

- 1 10×32 tapped holes in 4 places

Figure 4 — Retainer frame

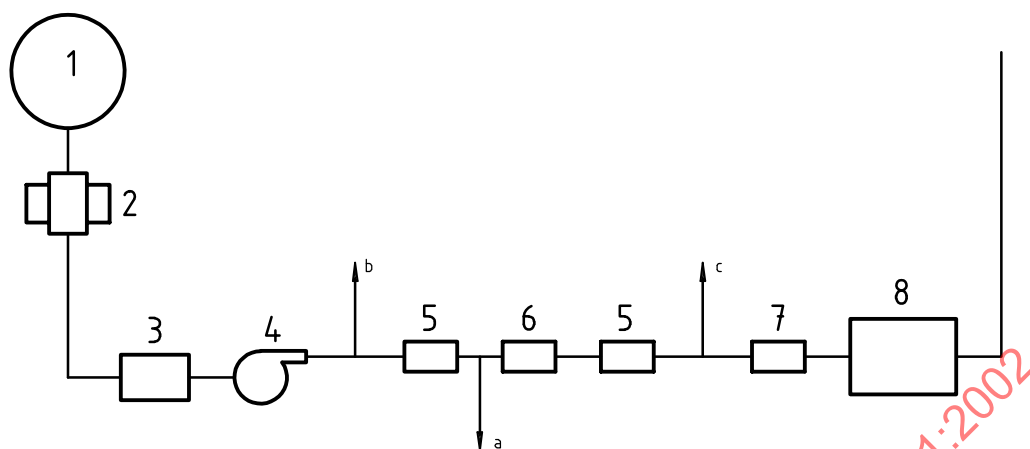
Dimensions in millimetres



Key

- 1 Gas sampling ring probe
- 2 Thermocouple
- 3 Hood
- 4 Orifice plate
- 5 Gas sampling ring probe (sample holes face blower)
- 6 Fan

Figure 5 — Exhaust system

**Key**

- 1 Ring sampler
- 2 Particulate filter
- 3 Cold trap and drain
- 4 Pump
- 5 Moisture trap
- 6 CO₂ removal trap
- 7 Flow controls
- 8 Oxygen analyser

^a To optional CO₂ and CO analysers

^b Waste

^c Alternative position for waste

Figure 6 — Gas sampling and measurement system

Annex A (informative)

Commentary and guidance notes for operators

A.1 Introduction

This annex aims to provide the test operator, and perhaps the user of the test results, with background information on the method, the apparatus and the data obtained.

A.2 Heat release rate measurements

A.2.1 Heat release rate is one of the most important variables in determining the hazard from a fire. In a typical fire, many items composed of many surfaces contribute to the development of a fire, thus making its evaluation quite complex. A determination should first be made when each separate surface will ignite, if at all. The size of the fire from any items already burning must be known due to its contribution to the external irradiance on nearby items. Flame spread over each surface should also be evaluated. The heat release rate from the whole surface is then determined knowing the heat release rate per unit area for a given irradiance, as a function of time, as evaluated using this bench scale test. The total fire output then involves a summation over all surfaces for all materials.

A.2.2 Factors which complicate the calculation of the heat output of a fire are

- a) the different burning durations for each individual material involved,
- b) the geometry of each surface, and
- c) the burning behaviour of the material, i.e. melting, dripping or structural collapse.

A.2.3 The test method does not prescribe the irradiance levels. These should be determined separately for each product to be assessed. For given applications and products, a comparison with some full-scale fires is generally necessary to determine the time period over which heat release is to be calculated.

For exploratory testing, it is recommended to use the spark igniter and an irradiance value of 35 kW/m² initially; in the absence of further specifications from the sponsor, tests at 25 kW/m², 35 kW/m² and 50 kW/m² are recommended. Results obtained then suggest whether additional testing at different irradiance levels is desirable.

The test results may not be statistically significant unless the irradiance used is substantially (10 kW/m²) higher than the minimum irradiance level needed for sustained flaming to occur for that specimen.

A.3 Choice of operating principle

A.3.1 A number of apparatuses have been developed for measuring heat release rate. Traditionally, the simplest is a direct measurement of flow enthalpy from a chamber thermally lagged to present an adiabatic environment. A truly adiabatic apparatus, with the use of guard heaters, is possible, but would be prohibitively expensive. A combustion chamber that is insulated in a simpler manner leads to a significant under-measurement of the heat release, so that only an empirical calibration is possible. Furthermore, that calibration may be sensitive to the sootiness of the combustible. A more advanced scheme is an isothermal instrument, with the heat release rate taken to be that which must be supplied by a substitution burner to maintain isothermal conditions. This scheme gives better results, however, its practical implementation is complex and costly.

A.3.2 It is difficult to measure heat directly without some loss. However, it is simple to contain the total products of combustion without loss and to measure the oxygen concentration in that stream. Heat release can be computed from such measurements using the oxygen consumption principle. This principle states that for most common combustibles an amount of heat equal to $(13,1 \times 10^3)$ kJ is released for each kilogram of oxygen consumed from the air stream. This quantity varies by about $\pm 5\%$ for most common combustibles. This principle forms the basis of the

test method detailed in this part of ISO 5660. The method remains useful even if a significant fraction of the products become CO or soot rather than CO₂; in these cases, correction factors can be applied.

Excessively high CO concentrations which could result from a restricted oxygen supply, cannot occur under the normal operating conditions of this test method since oxygen intake is not restricted.

A.4 Heater design

A.4.1 Experience with various rate-of-heat-release measurement techniques suggests that for minimal errors in irradiance, the specimen should see only either a thermostatically controlled heater, or a water-cooled plate or open air. Nearby solid surfaces, if they are not temperature controlled, can rise in temperature due to specimen flame heating and can then act as further sources of radiation back to the specimen which can lead to errors. Furthermore, when oxygen consumption is used as the measurement principle, a gas-fired heater is not desirable because it can contribute a noisy baseline to oxygen readings, even though allowance can be made for its oxygen consumption.

A.4.2 The heater, in the shape of a truncated cone, initially developed for ISO 5657, has been modified to include higher irradiance, temperature control, flow streamline improvement and to be of a more rugged design. In the horizontal orientation, the conical shape approximately follows the fire plume contours while the central hole allows the stream to emerge without impacting on the heater. Air entrainment ensures the flames do not reach the sides of the cone.

A.4.3 Due to the shape of the heater, the apparatus is commonly referred to as the Cone calorimeter.

A.5 Pilot ignition

Ignition of test specimens in many apparatuses is achieved by a gas pilot. This however can present difficulties when assessing heat release due to its contribution to heat output, deterioration of orifices and sooting. Its design is also difficult since it should be centrally located, be resistant to extinguishment by draughts and fire retardants, and, most importantly, additional heat should not be applied to the specimen. An electric spark is free from most of these difficulties and has therefore been adopted as the igniter. The spark igniter requires only occasional cleaning and adjustment of the electrodes.

A.6 Back face conditions

The heat losses through the back face of the specimen can have an influence on the burning rate near the end of its burning time. For reproducible measurements, the loss through the back face should be standardized and this is achieved by using a layer of insulating material.

Annex B (informative)

Resolution, precision and bias

B.1 Resolution

Methane calibration studies have shown typical fluctuations (due mainly to the turbulence of the flame itself) of $\pm 1,5\%$ in the heat release rate with a linearity to within 5 % over a range of 1 kW to 12 kW, and within 2 % over a range of 5 kW to 12 kW. Calibrations with other gases show similar results. Calibration gases can be delivered to the burner at a steady rate. The uniformity of solid-fuel combustion, however, is governed by the pyrolysis at the surface, which can under some circumstances show substantial fluctuations. For instance, the fluctuations for poly(methyl methacrylate) are typically greater than for wood products. With solid materials, therefore, the resolution is determined by the specimen pyrolysis process, rather than by instrument limits.

B.2 Speed of response

The limits to the speed of response of any technique for measuring heat release rate are set by the slowest responding element. With this method this is the oxygen analyser. Response times of the pressure transducer and thermocouple are usually considerably faster.

B.3 Precision

The repeatability limit r and reproducibility limit R in B.3 and B.4 were calculated according to ISO 5725:1986 (now withdrawn) which was valid when the interlaboratory trials were conducted.

NOTE The current version of ISO 5725-1 reports r and R as $1 \times$ the relevant standard deviation, rather than $2,8 \times$ the standard deviation.

A set of interlaboratory trials was conducted by ISO/TC 92/SC 1/WG 5. The protocol used was functionally the same as described in this part of ISO 5660. The materials tested in these trials were: 25 mm black PMMA ($\rho = 1\,180\text{ kg/m}^3$), 30 mm rigid polyurethane foam ($\rho = 33\text{ kg/m}^3$), 13 mm particle board ($\rho = 640\text{ kg/m}^3$), 3 mm hardboard ($\rho = 1\,010\text{ kg/m}^3$), 10 mm gypsum board ($\rho = 1\,110\text{ kg/m}^3$) and 10 mm fire-retardant-treated particle board ($\rho = 750\text{ kg/m}^3$). Three replicates of each material were tested in two orientations (horizontal and vertical) and at two irradiance levels (25 kW/m^2 and 50 kW/m^2) by six to eight laboratories.

Data from these trials were supplemented by data developed during an analogous set of trials conducted by ASTM E05 SC 21 TG 60, again using functionally the same protocol and identical irradiance levels, orientations and number of replicates. Since the findings for r and R in the ASTM trials showed generally similar trends, the data were analysed as a combined data set. ASTM data were excluded in the one instance (i.e. for $\dot{q}_{A,180}$) where instructions to the laboratories differed. Six laboratories tested the following materials: 6 mm fire-retardant-treated ABS ($\rho = 325\text{ kg/m}^3$), 12 mm particle board ($\rho = 640\text{ kg/m}^3$), 6 mm black PMMA ($\rho = 1\,180\text{ kg/m}^3$), 6 mm polyethylene ($\rho = 800\text{ kg/m}^3$), 6 mm PVC ($\rho = 1\,340\text{ kg/m}^3$) and 25 mm rigid polyisocyanurate foam ($\rho = 28,0\text{ kg/m}^3$).

Values for repeatability limit r and reproducibility limit R at the 95 % confidence level were calculated for the complete data set according to ISO 5725:1986 for five variables. Such values for r and R are equal to $2,8 \times$ the appropriate standard deviation and include results identified as "stragglers". The variables were chosen as being representative for the test results: t_{ig} , $\dot{q}_{A,max}$, $\dot{q}_{A,180}$, $Q_{A,tot}$ and $\Delta h_{c,eff}$. A linear regression model (equation II in ISO 5725:1986) was used to describe r and R as a function of the mean over all replicates and over all laboratories

for each of the five aforementioned variables. The regression equations are given below. The range of mean values over which the fit was obtained is also indicated.

The results for t_{ig} in the range of 5 s to 150 s were:

$$r = 4,1 + 0,125t_{ig} \quad (B.1)$$

$$R = 7,4 + 0,220t_{ig} \quad (B.2)$$

The results for $\dot{q}_{A,max}$ in the range of 70 kW/m² to 1 120 kW/m² were:

$$r = 13,3 + 0,131\dot{q}_{A,max} \quad (B.3)$$

$$R = 60,4 + 0,141\dot{q}_{A,max} \quad (B.4)$$

The results for $\dot{q}_{A,180}$ in the range of 70 kW/m² to 870 kW/m² were:

$$r = 23,3 + 0,037\dot{q}_{A,180} \quad (B.5)$$

$$R = 25,5 + 0,151\dot{q}_{A,180} \quad (B.6)$$

The results for $Q_{A,tot}$ in the range of 5 MJ/m² to 720 MJ/m² were:

$$r = 7,4 + 0,068Q_{A,tot} \quad (B.7)$$

$$R = 11,8 + 0,088Q_{A,tot} \quad (B.8)$$

The results for $\Delta h_{c,eff}$ in the range of 7 kJ/g to 40 kJ/g were:

$$r = 1,23 + 0,050\Delta h_{c,eff} \quad (B.9)$$

$$R = 2,42 + 0,055\Delta h_{c,eff} \quad (B.10)$$

The meaning of these equations is best illustrated by means of an example. Suppose a laboratory tests a single sample of a certain material and determines that the time to ignition is 100 s. If the same laboratory now conducts a second test on the same material, the value of r is evaluated as:

$$r = 4,1 + 0,125 \times 100 = 17 \text{ s}$$

Then the probability is 95 % that the result of the second test will fall between 83 s and 117 s. Suppose now that the same material is tested by a different laboratory. The value of R is evaluated as:

$$R = 7,4 + 0,220 \times 100 = 29 \text{ s}$$

Then the probability is 95 % that the results from the test at that laboratory will fall between 71 s and 129 s.

B.4 Precision (test procedures for materials that intumesce or deform)

A set of interlaboratory trials on materials that intumesce or deform when subjected to heat was conducted by ISO/TC 61/SC 4/WG 3. The protocol used was functionally the same as described in this standard, with a distance between sample surface and heater base plate of 60 mm (instead of the standard 25 mm) as specified in 7.5. The materials tested in these trials were: 9,6 mm black PMMA, 4 mm PVC, 3 mm fire-retardant polypropylene, 5,8 mm polycarbonate and 7,8 mm polycarbonate. Three replicates of each material were tested in the horizontal orientation, and at 50 kW/m² by 10 laboratories.

Values for repeatability limit r and reproducibility limit R at the 95 % confidence level were calculated according to ISO 5725:1986 for three variables: t_{ig} , $\dot{q}_{A,max}$ and $Q_{A,tot}$. A linear regression model (equation II in ISO 5725:1986) was used to describe r and R as function of the mean over all replicates and over all laboratories for each of the three aforementioned variables. The regression equations are given below. The range of mean values over which the fit was obtained is also indicated.

The results for t_{ig} in the range of 27 s to 167 s were:

$$r = 2,3 + 0,255t_{ig} \quad (B.11)$$

$$R = 2,3 + 0,652t_{ig} \quad (B.12)$$

The results for $\dot{q}_{A,max}$ in the range of 83 kW/m² to 855 kW/m² were:

$$r = 36,6 + 0,064\dot{q}_{A,max} \quad (B.13)$$

$$R = 36,6 + 0,330\dot{q}_{A,max} \quad (B.14)$$

The results for $Q_{A,tot}$ in the range of 27 MJ/m² to 319 MJ/m² were:

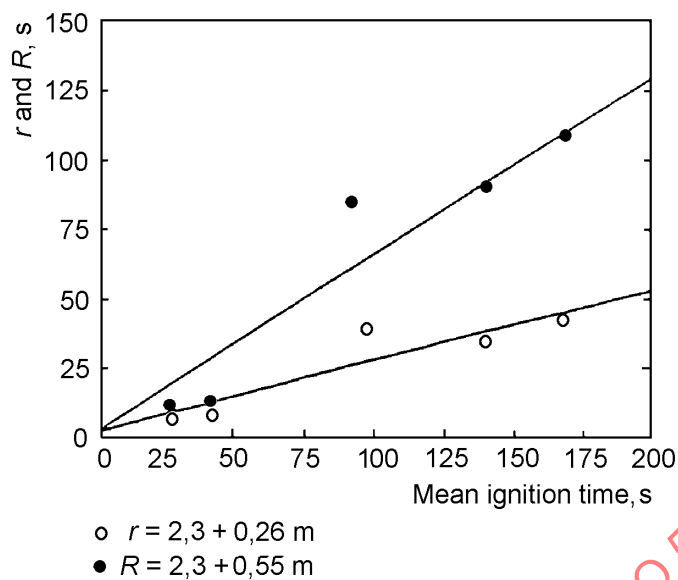
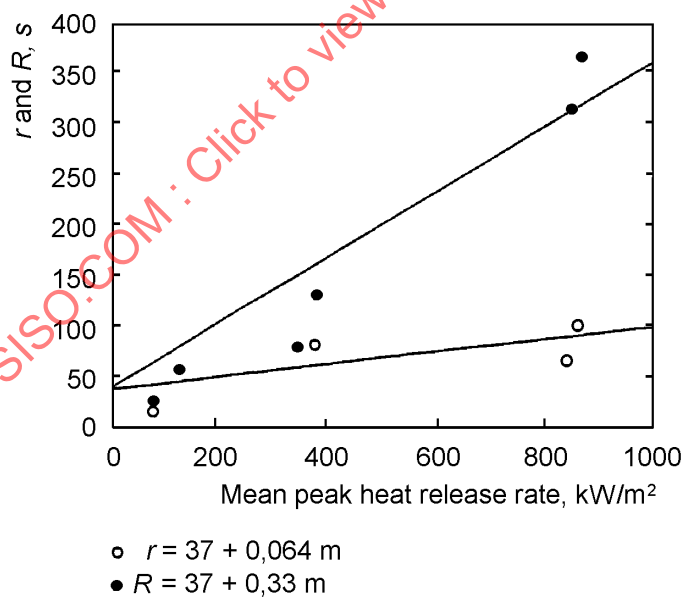
$$r = 15,5 + 0,008Q_{A,tot} \quad (B.15)$$

$$R = 15,5 + 0,125Q_{A,tot} \quad (B.16)$$

A comparison of equations (B.1) and (B.2) with (B.11) and (B.12) reveals that the repeatability and reproducibility of the ignition time is worsened by increasing the distance between the sample surface and the heater base plate to 60 mm. The repeatability of the remaining two variables does not seem to be affected [see equations (B.3) and (B.7) vs. (B.13) and (B.15)], but the reproducibility is slightly worse for the 60 mm distance [see equations (B.4) and (B.8) vs. (B.14) and (B.16)].

B.5 Bias

For solid specimens of unknown chemical composition, as used in building materials, furnishings, etc., it has been documented that the use of the oxygen consumption standard value of $\Delta h_c/r_o = (13,1 \times 10^3)$ kJ/kg oxygen results in an expected error band of ± 5 %. For homogeneous materials with only a single pyrolysis mechanism, this uncertainty can be reduced by determining Δh_c from oxygen bomb measurements and r_o from ultimate elemental analysis. For most testing, this is not practical since specimens are usually composites, non-homogeneous or exhibit several degradation reactions. For reference materials, however, careful determination of $\Delta h_c/r_o$ can make this source of uncertainty substantially less.

Figure B.1 — Values of r and R of t_{ig} for intumescent materialFigure B.2 — Values of r and R of $\dot{q}_{A,max}$ for intumescent material

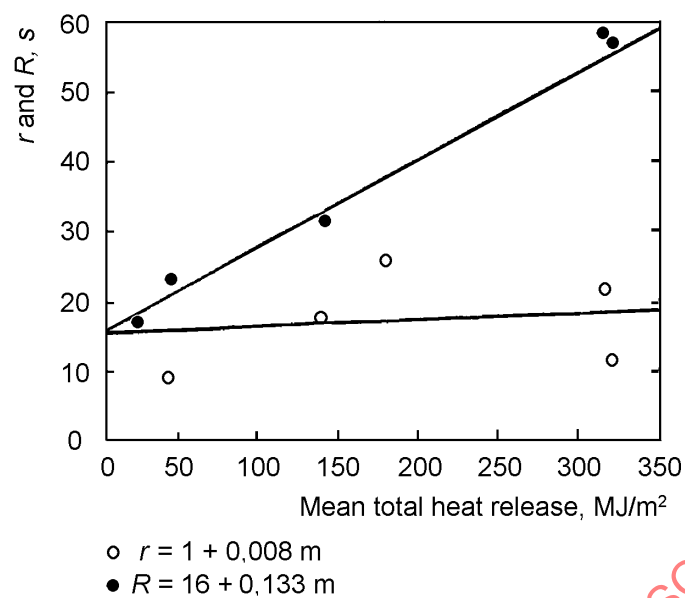


Figure B.3 — Values of r and R of $Q_{A,tot}$ for intumescent material

Annex C (informative)

Mass loss rate and effective heat of combustion

C.1 Effective heat of combustion

The effective heat of combustion is a constant during combustion of homogeneous specimens having only a single mode of degradation and is less than the value of the theoretical net heat of combustion. Examples of a material with a single mode of degradation and, therefore, a constant effective heat of combustion include most organic liquids. Cellulosic products, by contrast, typically show more than one mode of degradation and a varying effective heat of combustion. For materials having more than one mode of degradation, or for composites or non-homogeneous materials, the effective heat of combustion is not necessarily constant. Effective heat of combustion and mass loss rate can be used to provide additional information on the fire behaviour of materials.

NOTE For materials containing absorbed water or molecularly bound water, mass loss measured will not fully represent heat of combustion.

C.2 Symbols

$\Delta h_{c,eff}$, the effective net heat of combustion, is expressed in megajoules per kilogram (MJ/kg).

C.3 Calculation

The mass loss rate, $-\dot{m}$, computed for each time interval starting from time of ignition (see 12.5.1), can be used to determine a time-varying value of the effective heat of combustion:

$$\Delta h_{c,eff} = \frac{\dot{q}(t)}{-\dot{m}} \quad (C.1)$$

As the mass loss rate requires numerical differentiation for its determination and is, therefore, noisier than measurements obtained directly from instrument readings, it is better to calculate average values of $\Delta h_{c,eff}$. To obtain such averages, the numerator and denominator in equation (C.1) must be averaged separately rather than calculating the average of the ratio. For instance $\Delta h_{c,eff}$ averaged over the whole test is obtained as:

$$\Delta h_{c,eff} = \frac{\sum \dot{q}(t) \Delta t}{m_s - m_f} \quad (C.2)$$

The summation is taken over the entire test length from the time of ignition.