INTERNATIONAL STANDARD

ISO 17554

Second edition
2014-12-15

Reaction to fire tests — Mass loss measurement

Essais de réaction au feu — Mesurage de la perte de masse

Reference number
ISO 17554:2014(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

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.

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

This second edition cancels and replaces the first edition (ISO 17554:2005), which has been technically revised.

INTERNATIONAL STANDARD **ISO 17554:2014(E)**

Reaction to fire tests — Mass loss measurement

1 Scope

This International Standard specifies a small-scale method for assessing the mass loss rate of essentially flat specimens exposed in the horizontal orientation to controlled levels of radiant heating with an external igniter under well-ventilated conditions. The mass loss rate is determined by measurement of the specimen mass and is derived numerically. The time to ignition (sustained flaming) is also measured in this test. Mass loss rate can be used as an indirect measure of heat release rate for many products. However, this is not an apparatus for determining heat release rates, and the measured mass loss rates for some products are not so closely linked to their heat release rates. Such products need to be tested in accordance with ISO 5660-1 for correct assessment of heat release.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5660-1, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method)*

ISO 13943, *Fire safety — Vocabulary*

ISO 14934-3; *Fire tests — Calibration of heat flux meters – Part 3: Secondary calibration method*

ISO 14697:2007, *Reaction-to-fire tests — Guidance on the choice of substrates for building and transport products*

3 Terms and definitions

For the purposes of this document, 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 3.9

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 1 to entry: Convective heating is negligible in the horizontal specimen orientation. For this reason, the term “irradiance” is used instead of “heat flux” throughout this International Standard, 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
product
material, composite, or assembly about which information is required

3.8
specimen
representative piece of the product which is to be tested together with any substrate or treatment

Note 1 to entry: For certain types of product, for example, products that contain an air gap or joints, it is sometimes not possible to prepare specimens that are representative of the end-use conditions (see Clause 7).

3.9
sustained flaming
existence of flame on or over the surface of the specimen for periods of over 10 s

3.10
transitory flaming
existence of flame on or over the surface of the specimen for periods of between 1 s and 10 s

4 Symbols

Symbol	Designation	Unit
A_s	initially exposed surface area of the specimen	m^2
m	mass of the specimen	g
m_I	mass of the specimen at the start of the test	g
m_f	mass of the specimen at the end of the test	g
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 the specimen	$\mathrm{g \cdot s^{-1}}$
$\dot{m}_\mathrm{max}$	maximum value of the mass loss rate	$\mathrm{g \cdot s^{-1}}$
$\dot{m}_{A,10-90}$	average mass loss rate per unit area between 10 % and 90 % of mass loss	$\mathrm{g \cdot m^{2} \cdot s^{-1}}$
m_{10}	mass of the specimen at 10 % of total mass loss	g
m_{90}	mass of the specimen at 10 % of total mass loss	g
$\dot{m}_{180}$	average mass loss rate over the period starting at t_ig and ending 180s later	$\mathrm{g \cdot s^{-1}}$

Symbol	Designation	Unit
$\dot{m}_{300}$	average mass loss rate over the period starting at t_{ig} and ending 300s later	$\mathrm{g{\cdot}s^{-1}}$
t	time	s
t_{ig}	time to ignition (onset of sustained flaming)	s
Δt	sampling time interval	s

5 Principle

The test method is used to assess the mass loss rate that the product undergoes under the test conditions. This rate is determined on small representative specimens burning in a well-ventilated environment.

6 Apparatus

6.1 General

The test apparatus shall consist essentially of the following components:

6.1.1 Cone-shaped radiant electrical heater.

6.1.2 Weighing device for measuring specimen mass.

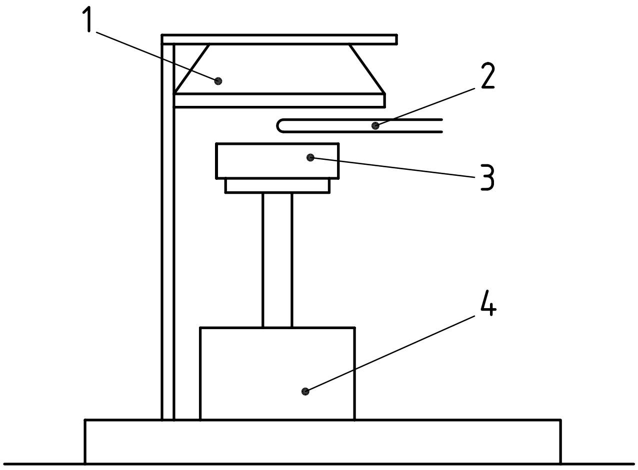
6.1.3 Specimen holder.

6.1.4 Spark ignition circuit.

6.1.5 Heat flux meters.

6.1.6 Data collection and analysis system.

A schematic representing the assembly is given in Figure 1. The apparatus should be located under a suitable exhaust system with a flow rate of less than $0{,}025\mathrm{m^3{\cdot}s^{-1}}$. The individual components are described in detail in the following sections.



Key

1 cone heater

2 spark igniter

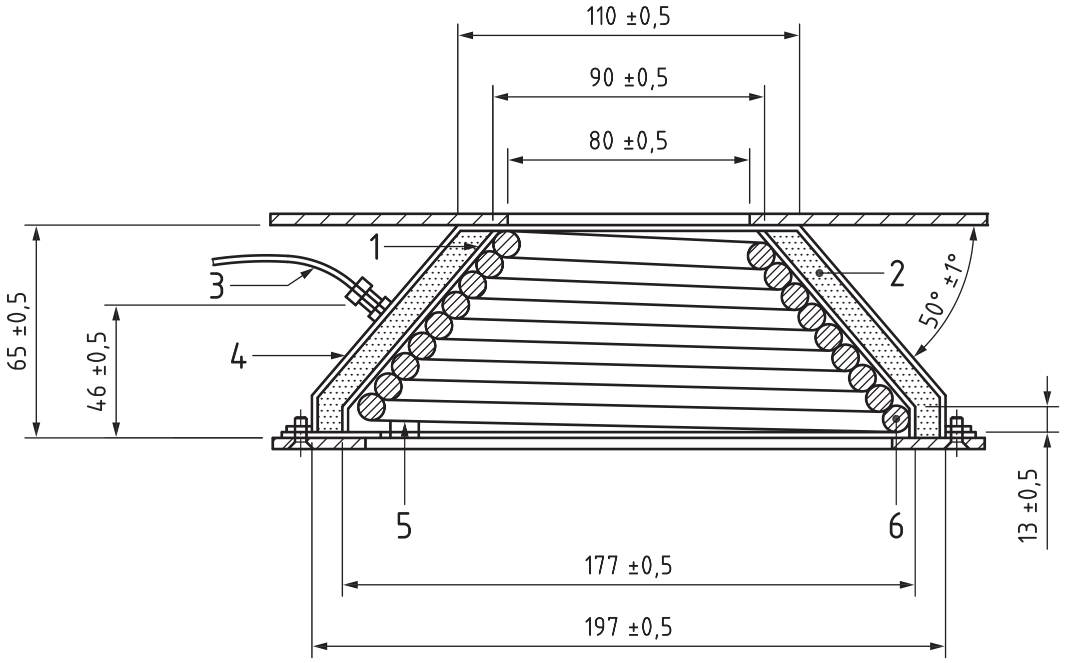
3 specimen
4 load cell

Figure 1 — Schematic of apparatus

6.2 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 ceramic fibre blanket of nominal thickness 13 mm and nominal density 100 kg·m^{-3}. 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[1]) or other high-performance materials are also acceptable) symmetrically disposed and in contact with, but not welded to, the heater element (see Figure 2). Either 3,0 mm outside-diameter sheathed thermocouples with an exposed hot junction or 1,0 mm to 1,6 mm outside-diameter sheathed thermocouples with an unexposed hot junction shall be used. The heater shall be capable of producing irradiances on the surface of the specimen of up to 75 kW·m^{-2}. The irradiance shall be uniform within the central 50 mm × 50 mm area of the exposed specimen surface, to within ±2 %, for an irradiance of 50 kW·m^{-2}.

Dimensions in millimetres



Key

1 inner shell
2 ceramic fibre packing
3 thermocouple
4 outer shell
5 spacer block
6 heating element

Figure 2 — Conical heater assembly

---

[1] Iconel is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of thisproduct.

6.3 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:

— water cooled and coated with a durable matt black finish of surface emissivity $\varepsilon = 0{,}95 \pm 0{,}05$;

— not water-cooled, which may be either metal with a reflective top surface or metal with a ceramic 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 introducing the shield into position.

6.4 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 10.1.1 at the preset level to within ±10°C.

6.5 Weighing device

The weighing device shall have a resolution of ± 0,1 g and an accuracy of ± 0,3 g or better, measured according to the calibration procedure described in 10.2.1. 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 1 s to 4 s as determined according to the calibration described in 10.1.2. 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 10.1.3.

6.6 Specimen holder and retainer frame

6.6.1 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 ± 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 65kg·m^{-3}) ceramic 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 when testing dimensionally unstable materials for which the distance is (60 ± 1) mm (7.5).

Dimensions in millimetres

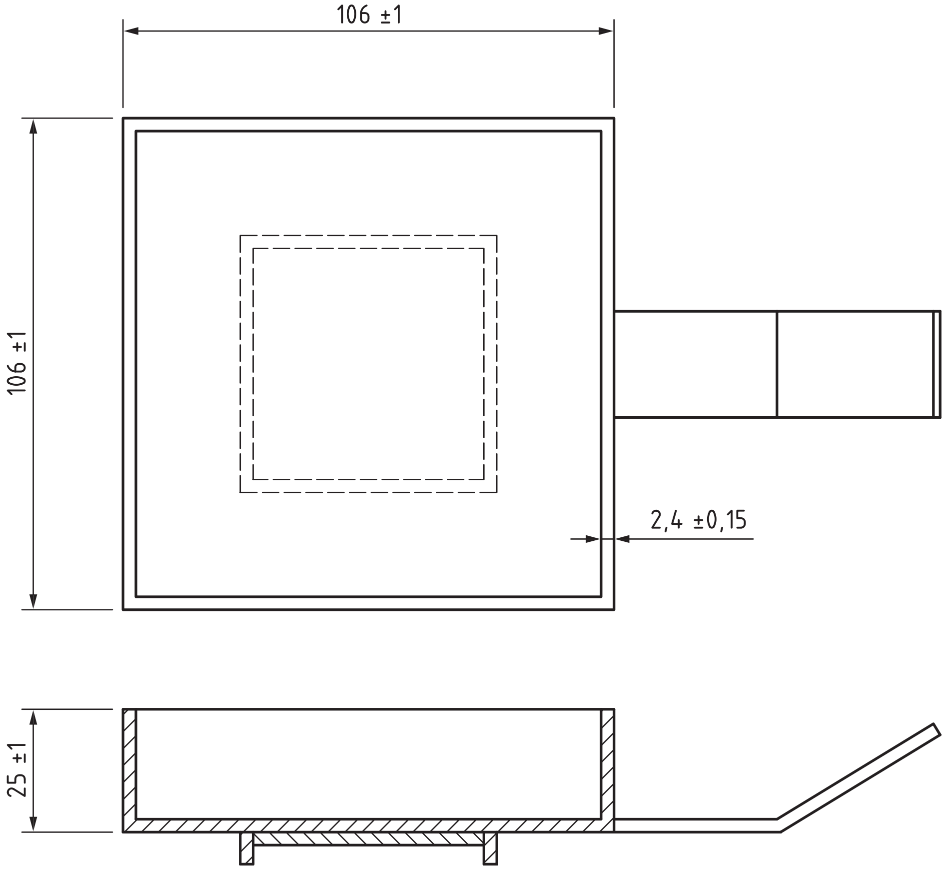
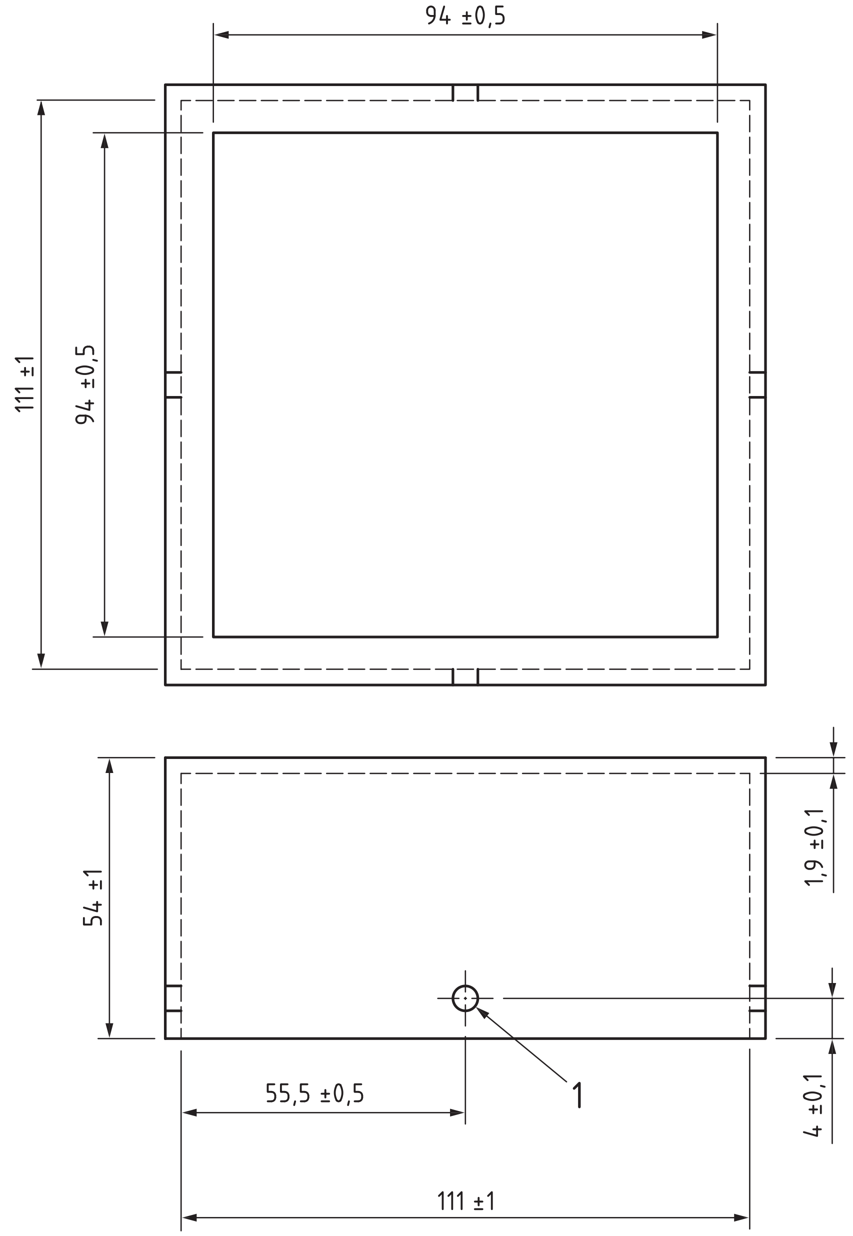


Figure 3 — Specimen holder

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

Dimensions in millimetres



Key

1 tapped holes in 4 places (M3 or 10 × 32 recommended)

Figure 4 — Retainer frame

6.7 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 ± 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 when testing dimensionally unstable materials for which the distance is (48 ± 2) mm (7.5).

6.8 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.

6.9 Heat flux meters

The working heat flux meter shall be used to calibrate the heater (10.2.2). It shall be positioned at a location equivalent to the centre of the specimen face during calibration.

This heat flux meter shall be of the Schmidt-Boelter (thermopile) type with a design range of (100 ± 10) $kW{\cdot}m^{-2}$. 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, $\varepsilon = 0{,}95 \pm 0{,}05$. The case of the heat flux meter shall be water-cooled. A cooling temperature, which could 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 a repeatability to within ±0,5 %.

The calibration of the working heat flux meter shall be done according to Annex C or ISO 14934-3 and checked according to 10.3.1, by comparison with two instruments of the same type as the working heat flux meter. They shall be of similar range as the reference standards and not used for any other purpose (see Annex C). One of the reference standards shall be fully calibrated at a standardizing laboratory at yearly intervals.

6.10 Data collection and analysis system

This system shall have facilities for recording the output from the weighing device. It shall have an accuracy corresponding to at least 0,01 % of the full-scale instrument output and at least 0,1 % for the measurement of time.

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^2 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 or 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^2 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 close 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 to this test may have faces which differ or 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, both faces shall be tested.

7.3 Materials of short burning time

For specimens of short burning time (3 min or less), the measurements shall be taken at not more than 2 s intervals. For longer burning time, 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 to 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 shall be performed (10.2.2) with the heat flux meter positioned 60 mm below the cone heater base plate. It shall 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 in Figure 5. Metal wires of (1,0 ± 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.

Materials that distort so extensively that they cannot be held by four wires should be tested using the fine wire grid used in ISO 5660-1. This is made of (0,8 ± 0,1) mm steel wire with a spacing between wires of (20 ± 2) mm and is shown in Figure 5.

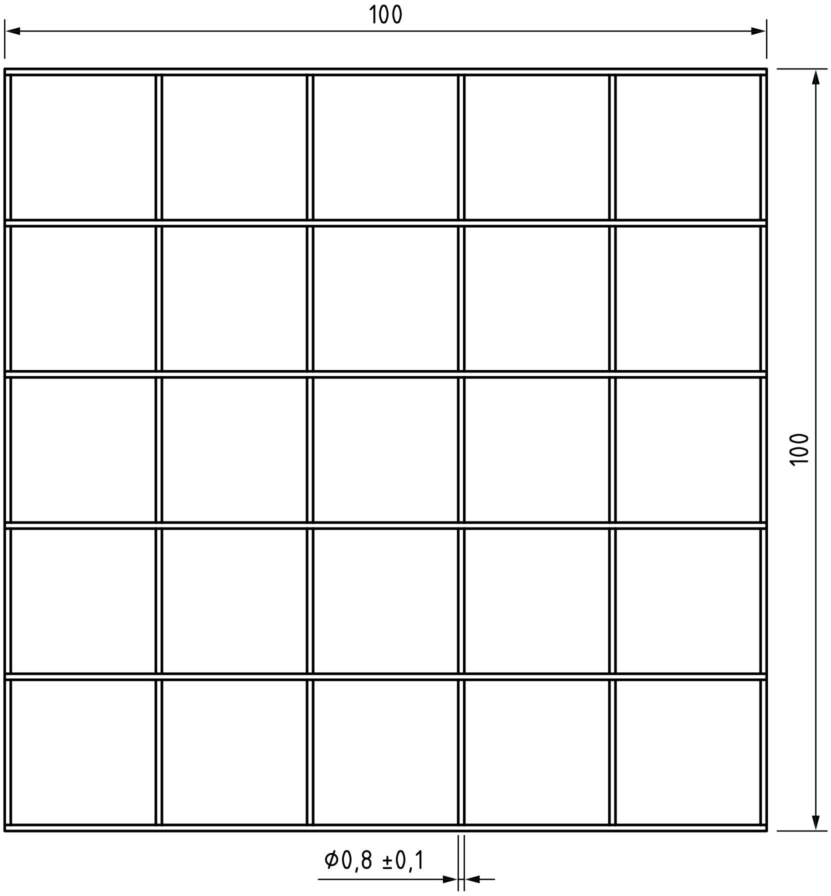


Figure 5 — Fine wire grid made from 0,8 mm wire

Materials that intumesce in a fluid phase such that molten materials overflow the retainer frame or seep between the edge frame and the specimen holder invalidate the test. These materials should be tested without the retainer frame and should be housed in 0,1 mm thick aluminium tray wrappings which extends 10 mm above the top edge of the test specimen.

7.6 Materials that require testing under compression

Materials, such as fibres, which need to be both physically restrained or compressed to be tested at installed densities should be tested in a suitable cage holder (Figure 6) which provides appropriate artificial boundaries to enable the materials to be tested. The cage holder is made of a steel wire cage constructed from (1,0 ± 0,1) mm steel wire with (9 ± 1) mm spacing (8.3.3).

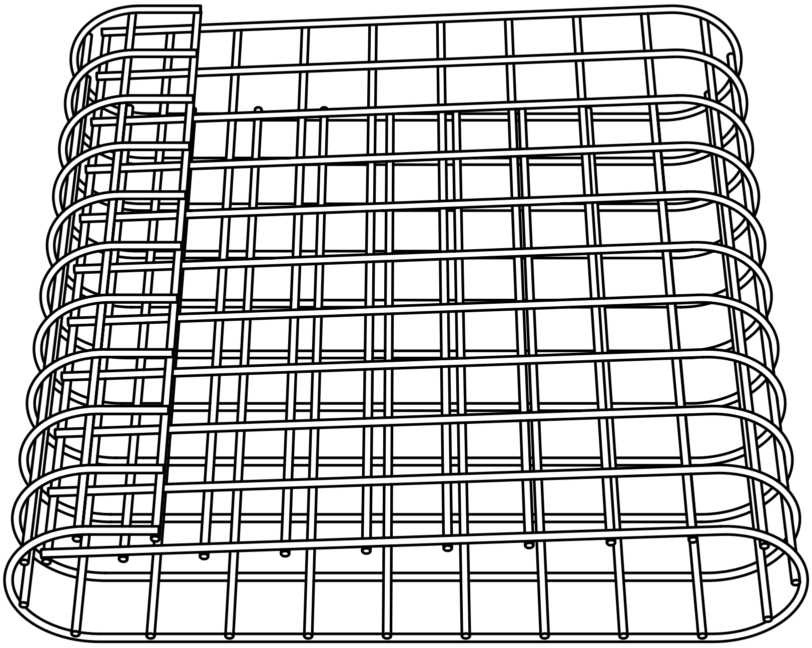


Figure 6 — Wire cage for materials that need testing under compression

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^{0}_{-2} mm

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

8.1.4 For products with 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 presence of air or an air gap, or 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 shall be 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 that would normally be attached to a well-defined substrate, then 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 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 ± 2) °C and a relative humidity of (50 ± 5) % in accordance with ISO 554.

NOTE 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 of conditioning to reach equilibrium, may be tested after conditioning for a period specified by the sponsor. This period shall not be 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 aluminium foil, of 0,025 mm to 0,04 mm thickness, with the shiny side towards the specimen. The aluminium foil shall be cut to a size to cover the bottom and the sides of the specimen and extended 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 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 (other than those that intumesce in a fluid phase or those that require testing under compressing in specimen cages, as in 8.3.3), 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 down.

b) Insert the foil-wrapped specimen into the frame with the exposed surface facing down.

c) Put layers of ceramic fibre blanket (nominal thickness 13 mm, nominal density 65 kg·m^{-3}) on top until at least one full layer and not more than two layers extend above the rim of the frame.

d) Fit the specimen holder into the frame on top of the ceramic fibre and press down.

e) Secure the retainer frame to the specimen holder and turn the assembly around.

8.3.3 Preparing specimens of materials that require testing under compression

a) Construct the specimen cage holder as shown in Figure 6 by forming a 241 mm × 101 mm sheet of steel mesh, constructed from (1,0 ± 0,1) mm steel wire with (9 ± 1) mm separation around a solid template 10 mm × 100 mm × 100 mm.

b) Remove the template and then pack the test material into the specimen cage such that its density is as in practice.

c) Test the specimen as described in 8.3.1 but do not use the retainer frame.

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

The calibrations described in this clause shall be performed before conducting experiments with a new apparatus or after maintenance, repair, or replacement of the heater assembly or irradiance control system or the weighing device (10.1.1 and 10.1.2).

10.1.1 Irradiance control system response characteristics

This shall be carried out by the supplier or by a laboratory with an exhaust system complying with ISO 5660-1. Turn on power to the cone heater and the exhaust fan prepared in accordance with ISO 5660-1. Set an irradiance of (50 ± 1) $kW{\cdot}m^{-2}$, and an exhaust flow rate of (0,024 ± 0,002) $m^3{\cdot}s^{-1}$. After reaching equilibrium of the heater, record the average heater temperature. Test a specimen of black polymethylmethacrylate (PMMA) without the retainer frame and according to the procedure in Clause 11. The PMMA specimen shall have a thickness of at least 6 mm. The validity of the test shall be checked against the expected average mass loss rate recorded over the first 3 min following ignition for the specific batch of PMMA which should be provided by the supplier. Terminate the test at 5 min after ignition. During the test, record the average heater temperature at 5 s intervals.

10.1.2 Weighing device response time

The cone heater shall not be turned on for this calibration. Place an empty specimen holder with a (500 ± 25) g mass on the load cell. The mass 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 mass of (250 ± 25) g on the holder and record the weighing device output. After equilibrium is reached, gently remove the second mass 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.

10.1.3 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 cone heater and the exhaust fan. Set an irradiance of (50 ± 1) $kW{\cdot}m^{-2}$. After reaching equilibrium of the heater temperature, remove the thermal barrier and place an empty specimen holder plus a (500 ± 25) g mass on the load cell. The mass 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 mass of (250 ± 25) g on the specimen holder. After equilibrium is reached, start recording the weighing device output at a maximum of 5 s intervals for a period of 30 min. Calculate the drift of the weighing device output as the absolute value of the difference of initial and final values.

10.2 Operating calibrations

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.

10.2.1 Weighing device accuracy

At the start of testing each day, the weighing device shall be calibrated with standard masses 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 plus a (500 ± 25) g mass on the weighing device. The mass accounts for the retainer frame, which is not used during this calibration. Measure the load cell output, and mechanically or electronically adjust the value to zero. Gently add a mass between 50 g and 200 g to the holder and measure the load cell output after it reaches a steady value. Repeat this procedure at least four times after adding masses of the same mass range. At the end of the calibration, the total mass of all masses on the holder shall be at least 500 g. The accuracy of the load cell is determined as the maximum difference between the mass of the masses and the load-cell output recorded during the calibration.

10.2.2 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 ±2 % at the sample surface, 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 at a stable set point

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 C or the procedures described in ISO 14934-3. Comparisons shall be made at irradiance levels of (25 and 75) kW·m^{-2}. The readings from the two meters shall agree to within ±2 %. If the operating heat flux meter is found to disagree with the reference meter by more than ±2 %, the operating heat flux meter should be recalibrated and re-checked against the reference meter(s). If the operating heat flux meter cannot be brought to within a ±2 % agreement over the entire range, the operating meter shall be replaced.

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 involved 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 (10kV). 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 Turn on power to the cone heater and the exhaust system in the enclosure in which the instrument is being used. Power to the load cell shall not be turned off on a daily basis.

11.2.2 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 ceramic fibre blanket) in place during warm up and between tests to avoid excessive heat transmission to the weighing device. Adjust the distance between the bottom plate of the cone heater and the specimen surface as specified in 6.6.1 or 7.5.

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 (7.3).

11.3.2 Insert the radiation shield in position (6.3). Remove the thermal barrier protecting the weighing device. 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 (6.3), remove the shield and start the test. Within 1 s of removing the shield, insert and power the igniter.
- For type b) shields (6.3), remove the shield within 15 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; do not remove the spark until the entire test is completed. Report these events in the test report.

11.3.5 Collect all data until

a) 32 min after the time to sustained flaming,

NOTE The 32 min consists of a 30 min test period and an additional 2 min post-test period to collect data that will be time-shifted. Data are processed to the time to sustained flaming plus 30 min.

b) 30 min have elapsed and the specimen has not ignited, or

c) the mass of the specimen is less than 0.1 g for 60 s.

The end of the test is the beginning of the 60 s period *t*, whichever occurs first. 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 mass loss readings shall be compared for the three specimens. If any of these mean readings differs 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 specimen 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 Average mass loss rate

12.1.1 The average mass loss rate can be calculated for any time interval during the test from the monitoring of mass data. The usage of this data can be tailored to user needs but it is suggested that average values for the first 180 s and 300 s after ignition along with peak values are likely to be found to be the most valuable data.

The average mass loss rate between two times A and B in a test is given by the following formula:

$$\text{average mass loss rate in } g \cdot s^{-1} = \frac{m_\mathrm{A} - m_\mathrm{B}}{t_\mathrm{B} - t_\mathrm{A}} \tag{1}$$

where

m_A is the mass at time A g;

m_B is the mass at time B g;

t_B is the time B s;

t_A is the time A s.

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

For the first scan (i = 0):

$$-\left[\dot{m}\right]_{i=0} = \frac{25m_0 - 48m_1 + 36m_2 - 16m_3 + 3m_4}{12\Delta t} \tag{2}$$

For the second scan (i = 1):

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

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

$$-\left[\dot{m}\right]_{i} = \frac{-m_{i-2} + 8m_{i-1} - 8m_{i+1} + m_{i+2}}{12\Delta t} \tag{4}$$

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

$$-\left[\dot{m}\right]_{i=n-1} = \frac{-3m_n - 10m_{n-1} + 18m_{n-2} - 6m_{n-3} + m_{n-4}}{12\Delta t} \tag{5}$$

For the last scan (i = n):

$$-\left[\dot{m}\right]_{i=n} = \frac{-25m_n + 48m_{n-1} - 36m_{n-2} + 16m_{n-3} - 3m_{n-4}}{12\Delta t} \tag{6}$$

12.1.3 The mass loss rate per unit area which includes the “main” burning period, i.e. from 10 % of ultimate mass loss being lost to 90 %, is given by

$$\dot{m}_{A,10-90} = \frac{m_{10} - m_{90}}{t_{90} - t_{10}} \cdot \frac{1}{A_s} \qquad (7)$$

where

$\Delta m = m_i - m_f$;

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

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

13 Test report

The test report shall be as comprehensive as the given results of all specimens tested. It 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 thickness, expressed in millimetres (mm), and mass, expressed in grams (g). 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 testing, and special mounting procedures that were used;

l) irradiance expressed in kilowatts per square metre ($kW \cdot m^{-2}$) and exhaust system flow rate expressed in cubic metres per second ($m^3 \cdot s^{-1}$);

m) number of replicate specimens tested under the same conditions;

NOTE This shall be a minimum of three, except for exploratory testing.

n) time to sustained flaming, expressed in seconds;

o) test duration, i.e. the time between the start of the test and the end according to 11.3.5, expressed in seconds;

p) the mass remaining after the test, m_f, expressed in grams;

q) values determined in items n), o), q), averaged for all replicates;

r) additional observations, such as transitory flaming or flashing;

s) difficulties encountered in testing, if any;

t) average values for the first 180 s ($\dot{m}_{180}$) and 300 s ($\dot{m}_{300}$) after ignition, or for other appropriate periods and peak ($\dot{m}_{\mathrm{max}}$), values, expressed in grams per second;

u) average rate of specimen mass loss per unit area, m, expressed in grams per square metre second ($\mathrm{g \cdot m^{-2} \cdot s^{-1}}$), computed over the period between 10 % and 90 % of mass loss.

Annex A
(informative)

Commentary and guidance notes for operators

A.1 General

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.

The test method does not prescribe the irradiance levels and whether external ignition is used. 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 the mass loss is calculated.

For exploratory testing, it is recommended to use the spark igniter and an irradiance value of 35 $\text{kW}\cdot\text{m}^{-2}$ initially; in the absence of further specifications from the sponsor, tests at 25 $\text{kW}\cdot\text{m}^{-2}$, 35 $\text{kW}\cdot\text{m}^{-2}$, and 50 $\text{kW}\cdot\text{m}^{-2}$ 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 $\text{kW}\cdot\text{m}^{-2}$) higher than the minimum irradiance level needed for sustained flaming to occur for that specimen.

A.2 Use for factory production control

Both the heat release rate and mass loss rate as a function of time are “finger prints” of fire behaviour of a material/product. If an article in production is of constant composition, then the heat release and mass loss curves also remain constant. Because changes in heat release rates also alter mass loss rate measurements, then a check on constant quality can be determined by monitoring only mass loss rates and ignition performance to be constant. A suggested procedure for the usage of this apparatus in this capacity is described in Table A.1, which also suggests some heat flux levels needed for both the complete test and the factory production control.

Table A.1 — Recommendations for factory production control testing

Function	Complete test levels of heat flux	Factory production control levels of heat flux
Time to ignition	3	1
Burning rate (= mass loss rate)	2	1
Rate of heat release as function of time	2	—

Annex B
(informative)

Calculation of Effective Critical Heat Flux for Ignition

B.1 General

Critical heat flux is the minimum heat flux needed to sustain ignition. An effective critical heat flux value can be determined empirically using the cone and igniter assembly section of the apparatus (or of the ISO 5660-1 apparatus) by exposing specimens to different heat fluxes and measuring the ignition times for sustained flaming, initially observed. It is determined by trial and error with repeated tests in search of the heat flux for no ignition occurring within 15 min duration.

B.2 Procedure

The specimens shall be prepared for a heat release test as in Clause 8 but tested without a retainer frame.

Expose new specimens in each successive experiment to a different heat flux exposure and measure and record the time to sustained ignition of the vapours for each. The minimum heat flux is determined by trial and error. It shall first be determined to a coarse resolution of 5 kW·m^{-2}, and then more finely to 1 kW·m^{-2}.

Determine what is the lowest value of heat flux at which sustained ignition is achieved, and the highest value at which ignition is not achieved. The minimum heat flux for ignition is defined as the average between the lowest heat flux at which there is ignition, and the highest heat flux at which there is no ignition for 15 min. For example, if the specimen ignites at 30 kW·m^{-2}within 15 min, then repeat the same procedure at 25 kW·m^{-2}, 20 kW·m^{-2}, 15 kW·m^{-2}, and 10 kW·m^{-2} (in that order) until there is no ignition for 15 min.

If test results show a crossover, in that the lowest heat flux at which ignition occurs is lower than the highest heat flux at which no ignition was found, it will be necessary to carry out triplicate tests for each determination and average the results. The average of the (averaged) highest flux for non-ignition in 15 min and the averaged lowest heat fluxes for ignition within 15 min is the reported value for the minimum heat flux for ignition.

Annex C
(informative)

Calibration of the working heat flux meter

The inter-comparison of working and reference standard heat flux meters specified in 6.9 may be made using the conical heater (6.2), with each heat flux meter mounted in turn in the calibration position, care being taken to allow the whole apparatus to attain thermal equilibrium. Alternatively, a specially built comparison apparatus may be used (for example, that specified in BS 6809).

The use of two reference standards, rather than one, provides a greater safeguard against change in sensitivity of the reference instruments.

Bibliography

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

[2] ISO/TS 3814:—, *Standard tests for measuring reaction-to-fire of products and materials — Their development and application*

[3] BS 6809: 1987, *Method for calibration of radiometers for use in fire testing*

[4] ASTM E1354-97, *Standard test method for heat and visible smoke release rates for materials and products using an oxygen consumption calorimeter*

[5] TWILLEY. W.H., and BABRAUSKAS, V., User's guide for the cone calorimeter, NBS Special Publication SP 745. National Bureau of Standards, U.S., 1988

[6] JANSSENS. M.L., *Measuring rate of heat release by oxygen consumption*. Fire Technol. 1991, **27** pp. 234–249

[7] BABRAUSKAS. V., and GRAYSON, S.J., eds., Heat release in fires, Interscience Communications, London, ISBN 978-0-9556548-4-8

[8] BABRAUSKAS. V., *Development of the Cone calorimeter — A bench-scale heat release rate apparatus based on oxygen consumption.* Fire Mater. 1984, **8** pp. 81–95

ICS 13.220.50; 13.220.40

Price based on 21 pages