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

Part 4:

Measurement of heat release for determination of low levels of combustibility

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

Partie 4: Mesurage du débit calorifique pour la détermination des bas niveaux de combustibilité

Reference number
ISO 5660-4:2008(E)

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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 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 5660-4 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

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 measurement* [Technical Report]

- *Part 4: Measurement of heat release for determination of low levels of combustibility*

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

Part 4:
Measurement of heat release for determination of low levels of combustibility

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

Materials containing volatile organic substances, decomposition products or large amounts of moisture can produce violent releases of combustible gases or water vapour during testing.

1 Scope

This part of ISO 5660 specifies a method for evaluating materials and products that produce low levels of heat release when exposed to high heat flux (i.e. irradiance levels) typical of fully developed fires. It differs from ISO 5660-1 [15] by prescribing items such as specific specimen size, specimen holder, specimen orientation, volumetric flow rate for O_2 analyses and irradiance levels at which testing is conducted.

This test method is intended for use on products and materials that contain only small amounts of combustible elements, e.g. test specimens that yield a total heat release of < 15 MJ m^{-2}.

NOTE The test method for specimens that yield moderate to high total heat release is described in ISO 5660-1.

The information obtained from the test method in this part of ISO 5660 can also be used for fire safety engineering purposes.

2 Normative references

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

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

ISO 13943, *Fire safety — Vocabulary*

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

ISO 14934–2, *Fire tests — Calibration and use of heat flux meters — Part 2: Primary calibration methods*

ISO 14934-3:2006, *Fire tests — Calibration and use of heat flux meters — Part 3: Secondary calibration method*

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** (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 minimized 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

EXAMPLES 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 that is tested together with any substrate or treatment

NOTE For certain types of product, for example products that contain an air gap or joints, it might not be possible to prepare a specimen that is 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

3.12
heat flux
heat passing through a surface per unit area and unit time

NOTE Heat flux is expressed in kilowatt per square metre.

3.13
emissivity
ratio of the radiant heat flux emitted by a surface to the radiant heat flux emitted by a blackbody radiator at the same temperature

NOTE Emissivity is dimensionless.

4 Symbols and units

For the purposes of this document, the following symbols apply.

Symbol	Designations	Unit
A_s	initially exposed surface area of the specimen, 0,020 7 m^2	m^2
C	orifice flow meter calibration constant	$\mathrm{m}^{1/2}\cdot\mathrm{g}^{1/2}\cdot\mathrm{K}^{1/2}$
Δh_c	net heat of combustion	$\mathrm{kJ}\cdot\mathrm{g}^{-1}$
$\Delta h_\mathrm{c,eff}$	effective net heat of combustion	$\mathrm{MJ}\cdot\mathrm{kg}^{-1}$
m	mass of the specimen	g
Δm	total mass change	g
m_f	mass of the specimen at the end of the test	g
m_s	mass of the specimen at sustained flaming	g
$\dot{m}_\mathrm{A,10\text{-}90}$	average mass change rate per unit area between 10 % and 90 % of mass change	$\mathrm{g}\cdot\mathrm{m}^{-2}\cdot\mathrm{s}^{-1}$
m_{10}	mass of the specimen at 10 % of total mass change	g
m_{90}	mass of the specimen at 90 % of total mass change	g
$\dot{m}$	mass change rate of the specimen	$\mathrm{g}\cdot\mathrm{s}^{-1}$
$\dot{m}_\mathrm{e}$	mass flow rate in exhaust duct	$\mathrm{kg}\cdot\mathrm{s}^{-1}$
Δp	orifice meter pressure differential	Pa
$\dot{q}$	heat release rate	kW
$\dot{q}_\mathrm{A}$	heat release rate per unit area	$\mathrm{kW}\cdot\mathrm{m}^{-2}$
$\dot{q}_\mathrm{A,max}$	maximum value of the heat release rate per unit area	$\mathrm{kW}\cdot\mathrm{m}^{-2}$
$\dot{q}_\mathrm{A,180}$	average heat release rate per unit area over the period starting at t_ig and ending 180 s later	$\mathrm{kW}\cdot\mathrm{m}^{-2}$
$\dot{q}_\mathrm{A,300}$	average heat release rate per unit area over the period starting at t_ig and ending 300 s later	$\mathrm{kW}\cdot\mathrm{m}^{-2}$
$Q_\mathrm{A,tot}$	total heat released per unit area during the entire test	$\mathrm{MJ}\cdot\mathrm{m}^{-2}$
r_o	stoichiometric oxygen/fuel mass ratio	1

Symbol	Designations	Unit
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_{O2}	oxygen analyser reading, mole fraction of oxygen	1
$X_{0,O2}$	initial value of oxygen analyser reading	1
$X_{(-1),O2}$	oxygen analyser reading, before delay time correction	1
ε	emissivity	—

5 Principle

The measurement of the heat release rate and total heat release is used to quantify the test specimen's ability to ignite and contribute heat to the fire. It is based on the observation that, generally, the net heat of combustion of a material is directly related to the quantity of oxygen required for its combustion. This relationship is such that approximately $13,1 \times 10^3$ kJ of heat are released per 1,0 kg of oxygen consumed.

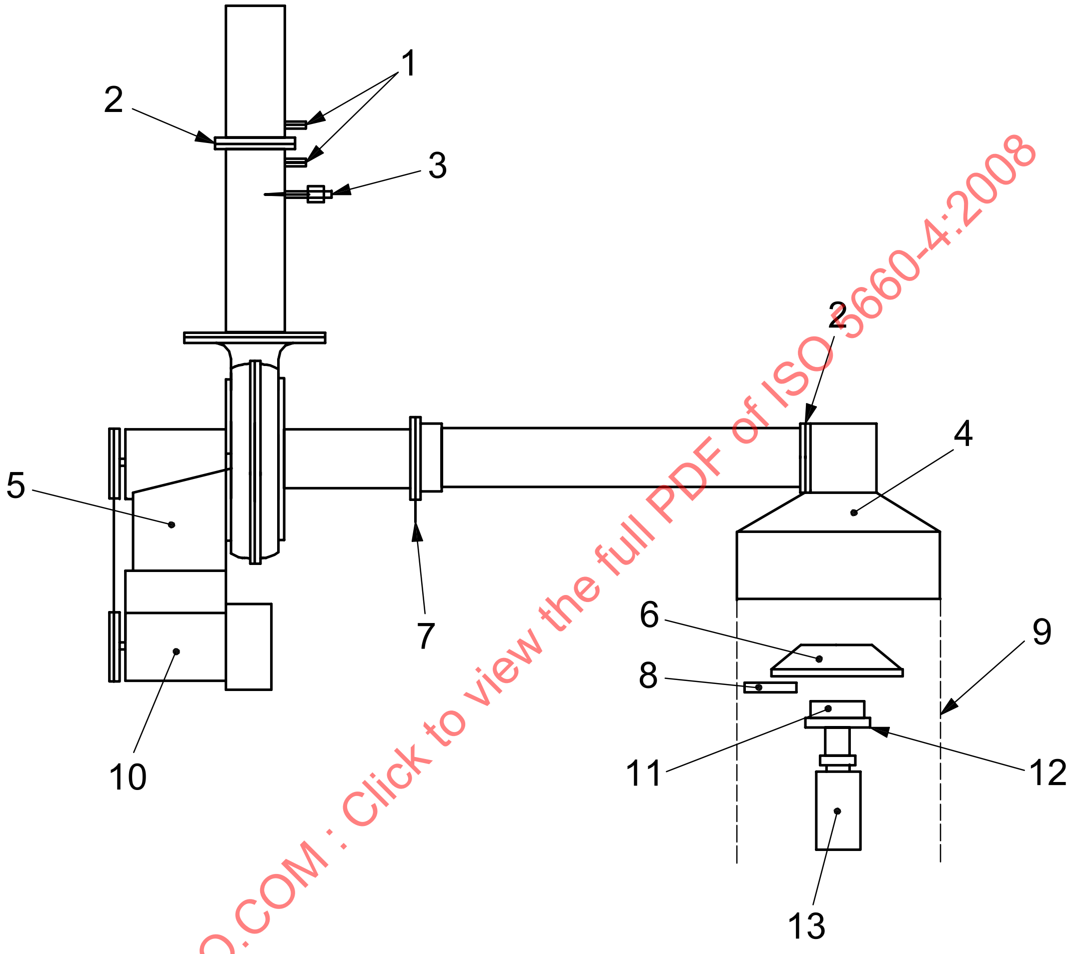
Specimens are exposed in ambient air conditions, while being subjected to an irradiance of 75 kW/m^2 in the presence of a spark ignition source. Alternatively, testing may be conducted at an exposure of 50 kW/m^2 provided there is ignition of the specimen at the 50 kW/m^2 irradiance level, and there is no evidence of continued combustion after the 20 min test duration. The changes in oxygen, O_2, concentration of gases and exhaust gas flow rate are monitored and, from these data, the heat release is calculated. Additionally, the time to sustained flaming is observed and mass-loss rate is measured.

In this part of ISO 5660, the heat release is measured from the moment the specimen is subjected to the radiant thermal exposure of a conical heater and is continued for 20 min. The primary measurements are oxygen concentration and exhaust-gas flow rate. Provision is also made for the time to sustained flaming. This test method is used to evaluate specimens in a horizontal orientation under an external heat flux.

6 Apparatus

6.1 General

The apparatus shall be set up as shown in Figure 1, with the individual components as described in detail in 6.2 to 6.6.



Key

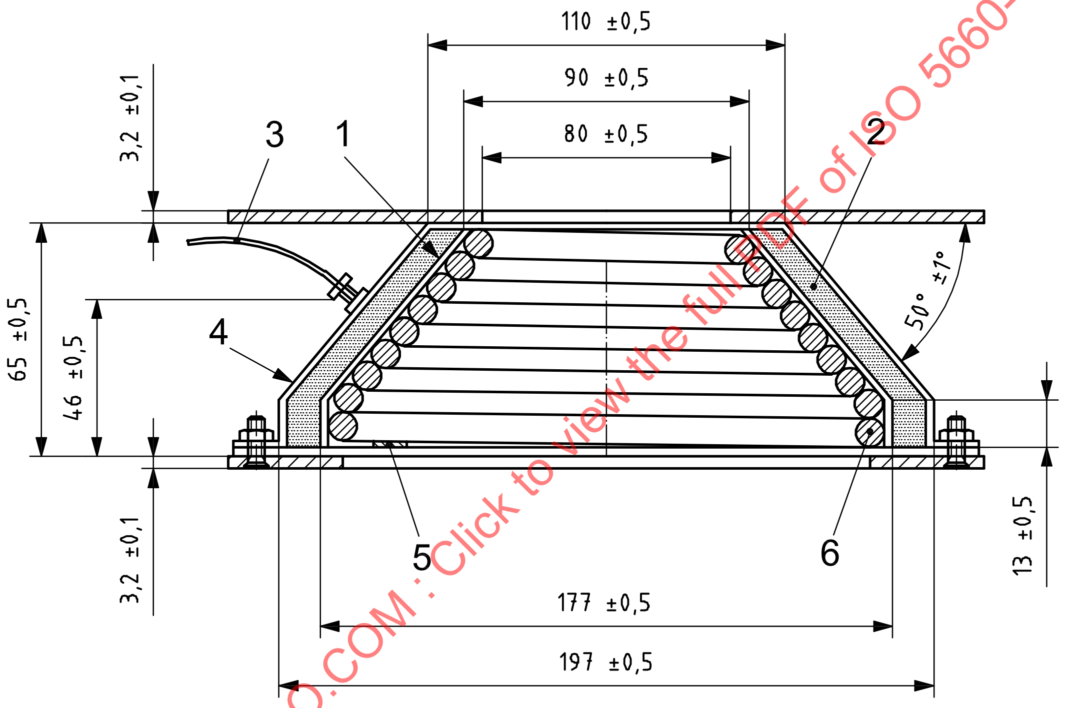
1	pressure ports	8	spark plug
2	orifice plate	9	optional screen
3	thermocouple (located on stack centreline)	10	blower motor
4	hood	11	retainer frame and specimen
5	blower	12	specimen holder
6	heater	13	weighing device
7	gas sampling ring probe		

Figure 1 — 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 refractory 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 a 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 irradiance on the surface of the specimen of up to 100 kW/m^2. The irradiance shall be uniform within the central 50 mm × 50 mm area of the exposed specimen surface, to within ± 2 %.

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

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[1] Inconel is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 5660 and does not constitute an endorsement by ISO of this product.

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:

a) water-cooled and coated with a durable matt black finish of surface emissivity, ε, equal to 0,95 ± 0,05;

b) not water-cooled, 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.

6.4 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 10.2.5 at the preset level to within ± 10 °C.

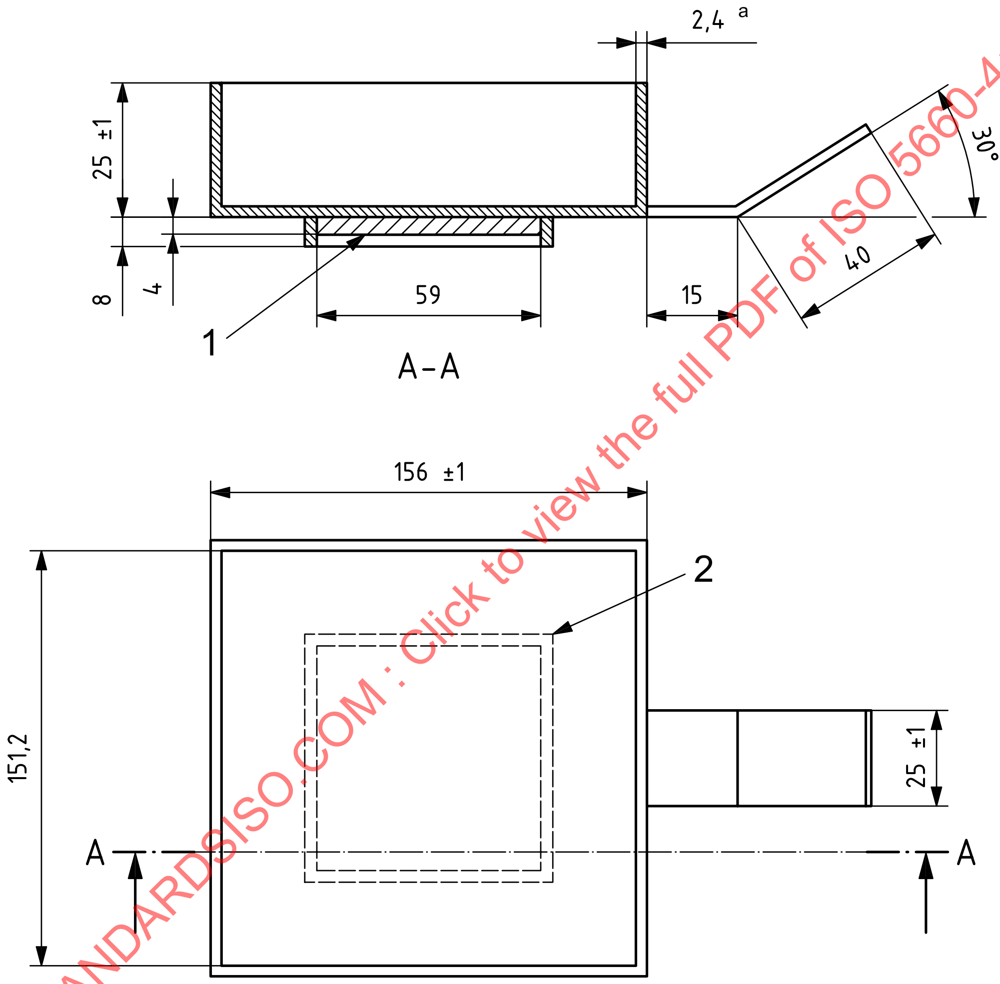
6.5 Weighing device

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 2,0 kg. The weighing device shall have a 10 % to 90 % response time of 4 s or less, as determined according to the calibration described in 10.1.2. 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

The specimen holder shall be as shown in Figure 3. The specimen holder shall have the shape of a square pan with an outside dimension of (156 ± 1) mm × (156 ± 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 of 65 kg/m^3) 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).

Dimensions in millimetres



Key

1 stainless (mill smooth)
2 spot weld, four corners

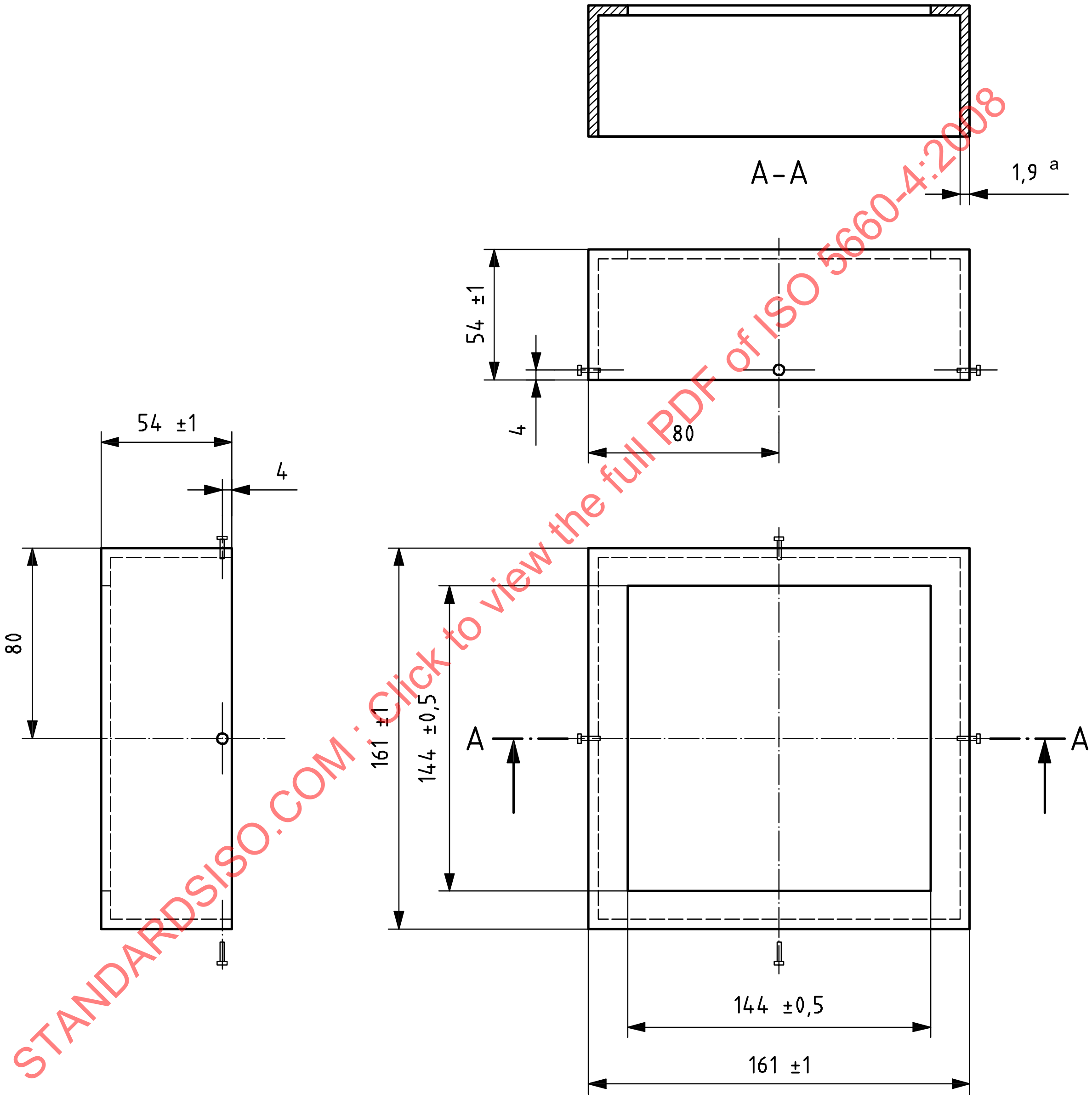
[a] Stainless steel.

Figure 3 — Specimen holder

6.7 Retainer frame

The frame shall be constructed of stainless steel with a thickness of (1,9 ± 0,1) mm, in the shape of a box with an inside dimension of (161 ± 1) mm and a height of (54 ± 1) mm. The opening for the specimen face shall be (144,0 ± 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 the specimen in position.

Dimensions in millimetres



[a] Stainless steel.

Figure 4 — Retainer frame

6.8 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 0,012 m^3/s, under standard conditions of temperature and pressure. The recommended location of the fan is indicated in 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 subclause 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 ± 0,1) mm, to average the stream composition, with the holes facing away from the flow to avoid clogging with soot.

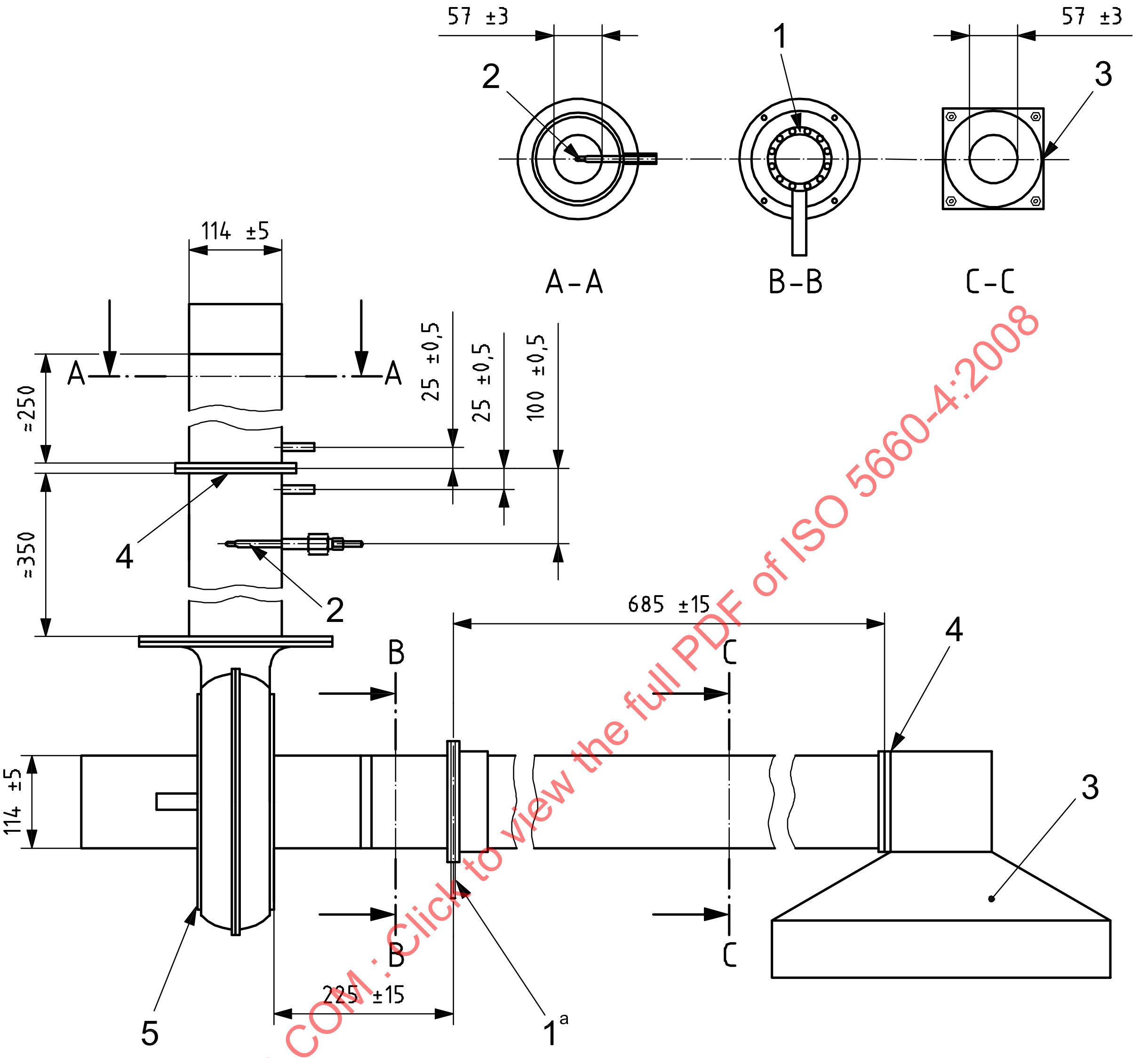
The temperature of the gas stream shall be measured using a 1,0 mm to 1,6 mm outside-diameter, sheathed-junction thermocouple or a 3 mm outside-diameter, exposed-junction thermocouple positioned in the exhaust stack on the centreline and (100 ± 5) mm upstream from the measuring orifice plate.

The flow rate shall be determined by measuring the differential pressure across a sharp edge orifice [internal diameter (57 ± 3) mm, thickness (1,6 ± 0,3) mm] in the exhaust stack, at least 350 mm downstream from the fan, if the latter is located as shown in Figure 5. The fan shall be located as indicated in Figure 5.

As an alternative, the fan may be located further downstream and the measuring orifice located before the fan.

The exhaust system shall be checked for proper operation before testing and shall discharge into a building exhaust system with adequate capacity. Provision shall be made for collecting and venting any combustion products that shall for whatever reason fail to be collected by the normal exhaust system of the apparatus.

Dimensions in millimetres



Key

1 gas-sampling ring probe
2 thermocouple
3 hood
4 orifice plate
5 fan

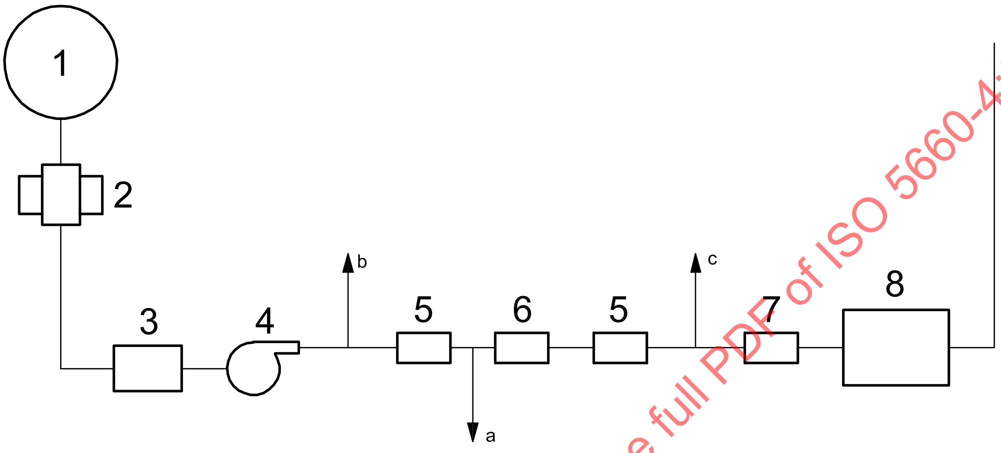
[a] Sample holes face blower.

Figure 5 — Exhaust system

6.9 Gas sampling apparatus

Gas sampling apparatus incorporates 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 CO_2 removal, all as shown in the schematic view of an example of the gas sampling apparatus 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 10.1.4, and shall not exceed 60 s.

NOTE If an (optional) CO_2 analyser is used, the equations to calculate the heat release rate can be different from those for the standard case (see Clause 13 and Annex A).



Key

1 ring sampler
2 particulate filter
3 cold trap and drain
4 pump
5 moisture trap
6 CO_2 removal trap
7 flow controls
8 oxygen analysers

[a] To optional CO_2 and CO analysers.
[b] Waste.
[c] Alternative position for waste.

Figure 6 — Gas sampling and measurement system

6.10 Ignition circuit

An external ignition source shall be provided by a spark generated by a spark plug powered from a 10 kV transformer or an equivalent 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 for dimensionally unstable materials for which the distance is (48 ± 2) mm (see 7.5).

6.11 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.

6.12 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 µl/l 2) of oxygen over a period of 30 min, and a noise of not more than 50 µl/ of oxygen during this 30 min period, as measured according to 10.1.5. 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 shall be 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 10.1.4.

6.13 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.

The heat-flux meter shall be of the Schmidt-Boelter (thermopile) type with a design range of 0 kW/m^2 to 100 kW/m^2 and a diameter of (12,5 ± 0,2) mm. The sensing surface shall be coated with a durable matt black finish of surface emissivity, ε, equal to 0,95 ± 0,05. The heat-flux meter shall be water-cooled. The water temperature should be kept above the dew point of the ambient air to avoid condensation of water on the sensing surface of the heat flux meter.

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

The working-standard heat-flux meter shall be calibrated according to ISO 14934-3. The secondary-standard heat-flux meter shall be calibrated according to ISO 14934-2.

6.14 Calibration burner

The calibration burner shall be constructed from a tube with a square or circular orifice with an area of (500 ± 100) mm^2 covered with wire gauze through which the methane diffuses. The tube shall be packed with refractory fibre to improve uniformity of flow. The calibration burner shall be connected to a metered supply of methane of at least 99,5 % purity. The accuracy of the flow meter shall be ± 2 % of the readout corresponding to a heat release rate of 1 kW. The accuracy verification shall be performed according to 10.3.3.

6.15 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 µl/l of oxygen for the oxygen channel, 0,5 °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 they can be recovered and used to check the accuracy of the software.

6.16 Optional side screens

For operational or safety reasons, the heater and sample holder may be guarded 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.

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2) “µl/l” are approximately equal to “ppm,” which is a deprecated unit.

WARNING — Attention is drawn to the fact that precautions should be taken to protect the operator if the screens form an enclosure because 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 should 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 that is evenly distributed over the exposed surface provided that

- — at least 50 % of the surface of a representative 225 mm^2 area lies within a depth of 10 mm from a plane taken across the highest points on the exposed surface, or
- — 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 225 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 nearly as possible with the requirements given in 7.1. The test report shall state that the product was tested in a modified form and clearly describe the modification.

7.2 Asymmetrical products

A product submitted for this test can have faces that 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 shall be considered suitable for testing provided 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 a 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 should be noted that the time to ignition and heat-release rates 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 ± 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 in such a way 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, a minimum of 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 $(150_{-2}^{\ 0})$ 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 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, whichever is appropriate.

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 should 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 that is normally 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 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 a constant mass at a temperature of (23 ± 2) °C, and a relative humidity of (50 ± 5) % in accordance with ISO 554.

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

Materials such as polyamides, which require more than one week in conditioning to reach equilibrium, may be tested after conditioning as described in ISO 291 [20]. 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 aluminium 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 being 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 down.

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

c) Put layers of refractory 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 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 following good laboratory and operating procedures shall be practised at all times.

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

b) The oxygen analyser and the pressure transducer shall be located in an isothermal environment to reduce the pressure variations.

c) The CO_2 trap and the moisture traps shall be checked daily and the sorbents shall be replaced daily or as often as necessary.

d) Any accumulated water shall be removed from the cold trap daily before the testing day and subsequently, when necessary.

e) Calibrations of all operating instruments, including calculation of the C constant, shall be performed at least once daily.

10 Calibration

10.1 Initial calibrations

10.1.1 General

The calibrations in this clause, 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, the weighing device (see 10.1.2 and 10.1.3), the oxygen analyser or other major components of the gas analysis system (see 10.1.5). The calibration tests to determine the effect of side screens in 6.16 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 Weighing device response time

The cone heater shall not be turned on for this calibration. Place an empty specimen holder with a (750 ± 50) g non-combustible weight piece on the weighing device.

NOTE The weight piece 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 weight piece with a mass of (750 ± 50) g on the holder and record the weighing device output. After equilibrium is reached, gently remove the second weight piece 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 exhaust fan and cone heater. Set an exhaust flow rate of (0,012 ± 0,002) m^3/s and an irradiance of (50 ± 1) kW/m^2. Once the heater temperature has reached its equilibrium, remove the thermal barrier and place an empty specimen holder with a (750 ± 50) g weight piece on the weighing device.

NOTE The weight piece 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 weight piece with a mass of (1 500 ± 500) 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.

10.1.4 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 (0,012 ± 0,002) m^3/s. Determine the delay time of the oxygen analyser by delivering a methane flow rate equivalent to 1 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 determined by 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, shall be determined using 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 shall be 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, it is not necessary to control the methane flow rate accurately, because the delay and response time is not sensitive to the oxygen level.

10.1.5 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 (0,012 ± 0,002) m^3/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 ± 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 establishes the short-term drift. Determine the noise by computing the root-mean-square deviation, x_{rms}, around the linear trend line according to Equation (1):

$$x_{\text{rms}} = \sqrt{\frac{\sum_{i=1}^{n} x_i^2}{n}} \qquad (1)$$

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

Record this root-mean-square noise value in terms of microlitres per litre of oxygen.

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 weight pieces 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 (750 ± 50) g weight piece on the weighing device. The retainer frame is not used during this calibration. Measure the weighing device output and mechanically or electronically adjust the value to zero. Gently add a calibrated weight piece with a mass between 500 g and 600 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 calibrated weight pieces of the same mass. At the end of the calibration, the total mass of all weight pieces on the holder shall not exceed 2 500 g. The accuracy of the weighing device shall be determined as the maximum difference between the mass of the weight pieces 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 (0,012 ± 0,002) m^3/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 ± 0,01) %. Calibration shall be similarly achieved using dried ambient air and adjusting for a response of (20,95 ± 0,01) %. Carefully monitor the analyser flow rates and set them equal to the flow rates used when testing specimens. After each specimen has been tested, ensure that a response level of (20,95 ± 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 shall 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 (0,012 ± 0,002) m^3/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, $\dot{q}_b$, corresponding to (1 ± 0,1) kW based on the net heat of combustion of methane of ($50{,}0 \times 10^3$) kJ/ kg. After the output from each instrument reaches equilibrium, collect data at 5 s intervals over a 3 min period. The orifice constant, C, shall be calculated according to Equation (2), using averages over the 3 min period of the measured values of $\dot{q}_b$, T_e, ΔP, and X_{O2}. $X_{0,O2}$ is determined as the average of the oxygen analyser output measured during the 1 min baseline measurements.

An alternate 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 ± 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 a set point and ensure that the controller is within its proportional band before beginning this calibration.

10.3 Less frequent calibrations

10.3.1 Working-standard heat flux meter calibration

At maximum intervals of 100 working hours, check the working-standard heat flux meter against the secondary-standard heat flux meter according to ISO 14934-3. The operating heat flux meter shall be calibrated in accordance with ISO 14934-3:2006, Annex D.

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 10.2.4, perform a further calibration with a flow rate corresponding to 1 kW ± 10 % and 3 kW ± 10 %, using the basic procedure as described in 10.2.4. With the value for C from the 1 kW calibration, the measured heat release rate at 2 kW and 3 kW shall be within ± 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 six months or when the calibration factor determined according to 10.2.2 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 ± 3 %. If the difference between the two measurements exceeds ± 3 %, the operating flow meter shall be re-calibrated 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 types of specimens when irradiated cannot be discounted and it is, therefore, essential that eye protection be worn.

11.2 Initial preparation

11.2.1 Check the CO_2 trap and the final moisture trap. Replace the solvent 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 line have been opened during the check, the gas sampling 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 shall 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 6.6 or 7.5.

11.2.3 Turn on power to the cone heater 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 (0,012 ± 0,002) m^3/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.

11.2.6 Set an irradiance level of 75 kW/m^2. Alternatively, testing may be conducted at an exposure of 50 kW/m^2 provided that there is ignition of the specimen at the 50 kW/m^3 irradiance level, and there is no evidence of continued combustion after the 20 min test duration.

11.3 Test procedure

11.3.1 Start data collection. Collect at least 3 min of baseline data prior to starting a test. The scan interval shall be equal to or less than 2 s.

11.3.2 Insert the radiation shield in position. Remove the thermal barrier protecting the weighing device.

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

11.3.3 Place the specimen, held in the specimen holder, onto the sample mount assembly. The holder shall be at room temperature initially.

11.3.4 Remove the radiation shield. Start the ignition timer, move the spark plug into place and turn on spark power.

11.3.5 Record the times when flashing or transitory flaming occur. When sustained flaming occurs, record the time, turn off the spark, and remove the spark igniter. If the flame extinguishes in less than 60 s after turning off the spark, reinsert the spark igniter and turn on the spark. If flaming recurs, stop the test, discard the test data and repeat the test without removing the spark until the entire test is completed. Report these events in the test report. Continue the test until a total of 20 min have elapsed since the start of the test.

Sustained flaming shall be reported as the time at which flaming was initially observed, not when the 10 s period elapsed.

11.3.6 At the end of the time period specified in 11.3.5, remove the sample from the holder and wait until all combustion products are cleared from the apparatus. Record the end of test value for the oxygen analyser. The value should be within 100 µl/l from the value recorded at the beginning of the test. If the value is not within the 100 µl/l value, the test shall be run again.

The test may be stopped if explosive spalling or excessive swelling occur. The specimen holder and weighing device may become very hot during operation. Care should be taken by the operator when removing the specimen.

11.3.7 Insert the thermal barrier to protect the weighing device from irradiance.

11.3.8 Collect data at least 3 min after the specimen removal.

11.3.9 Make three determinations and report as specified in Clause 14.

12 Test data limitations

The test data shall not be valid if

a) explosive spalling occurs, or

b) the specimen swells sufficiently prior to ignition to touch the spark plug or swells up to the plane of the heater base during combustion.

13 Calculations

13.1 General

The equations in this clause assume only O_2 is measured using 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 ISO 5660-1:2002, Annex F. If CO_2 is removed from the O_2 sampling lines (even when CO_2 is measured separately), then the Equations (2) to (4) shall be used.

13.2 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. The calibration constant, C, is calculated using Equation (2):

$$C = \frac{\dot{q}_\text{b}}{(12{,}54 \times 10^3)(1{,}10)} \sqrt{\frac{T_\text{e}}{\Delta p}} \cdot \frac{1{,}105 - 1{,}5 X_{\text{O2}}}{X_{0,\text{O2}} - X_{\text{O2}}} \qquad (2)$$

where

$\dot{q}_\text{b}$	corresponds to the rate of heat release, expressed in kilowatts, of the methane supplied (see 10.2.4);
$(12{,}54 \times 10^3)$ kJ/kg	is the $\Delta h_\text{c}/r_\text{o}$ ratio for methane;
1,10	is the ratio of the molecular weights of oxygen and air.

NOTE A calibration more than 5 % different from the previous one is not normal and suggests instrument malfunction.

13.3 Heat release rate

13.3.1 Prior to performing other calculations, calculate the oxygen analyser reading from the recorded analyser data and the delay time, t_d, using Equation (3):

$$X_\mathrm{O2}(t) = X_\mathrm{1,O2}(t + t_\mathrm{d}) \tag{3}$$

13.3.2 Calculate the heat release rate, $\dot{q}(t)$, from Equation (4):

$$\dot{q}(t) = (\Delta h_\mathrm{c} / r_\mathrm{o})(1{,}10)C\sqrt{\frac{\Delta p}{T_\mathrm{e}}} \cdot \frac{X_\mathrm{0,O2} - X_\mathrm{O2}}{1{,}105 - 1{,}5 X_\mathrm{O2}} \tag{4}$$

where $\Delta h_\mathrm{c}/r_\mathrm{o}$ for the specimen is taken as $(13{,}1 \times 10^3)$ kJ/kg, unless a more accurate value is known, and $X_\mathrm{0,O2}$ is determined as the average of the oxygen analyser output measured during the 1 min baseline measurements.

13.3.3 Heat release per unit area can be obtained from Equation (5):

$$\dot{q}_\mathrm{A}(t) = \dot{q}(t)/A_\mathrm{s} \tag{5}$$

where A_s is the initially exposed area of the sample.

13.4 Exhaust-duct flow rate

Calculate the mass flow rate in the exhaust duct, $\dot{m}_\mathrm{e}$, expressed in grams per second, from Equation (6):

$$\dot{m}_\mathrm{e} = C\sqrt{\frac{\Delta p}{T_\mathrm{e}}} \tag{6}$$

13.5 Mass loss rate

13.5.1 The mass loss rate, $-\dot{m}$, at each time interval may be calculated using the five-point numerical differentiation equations as given in Equations (7) to (11):

For the first scan, $i = 0$:

$$-[\dot{m}]_{i=0} = \frac{25m_0 - 48m_1 + 36m_2 - 16m_3 + 3m_4}{12\Delta t} \tag{7}$$

For the second scan, $i = 1$:

$$-[\dot{m}]_{i=0} = \frac{3m_0 + 10m_1 - 18m_2 + 6m_3 - m_4}{12\Delta t} \tag{8}$$

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

$$-[\dot{m}]_i = \frac{-m_{i-2} + 8m_{i-1} - 8m_{i+1} + m_{i+2}}{12\Delta t} \tag{9}$$

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

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

For the last scan, $i = n$:

$$-\left[\dot{m}\right]_{i=n} = \frac{-25m + 48m_{n-1} - 36m_{n-2} + 16m_{n-3} - 3m_{n-4}}{12\Delta t} \tag{11}$$

The Savitzky-Golay algorithm described in Reference [16] with $\eta_L = \eta_R = 5$ and $r = 2$ may be used as an alternative method to calculate mass change rate.

13.5.2 Calculate the mass change rate which includes the “main” burning period, i.e. from 10 % of ultimate mass loss being lost to 90 %, as given by Equation (12):

$$\dot{m}_{A,10-90} = \frac{m_{10} - m_{90}}{t_{90} - t_{10}} \times \frac{1}{A_s} \tag{12}$$

where

$\Delta m = m_s - m_1$

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

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

NOTE Equations for the effective heat of combustion, $\Delta h_{c,eff}$, are given in ISO 5660-1:2002, Annex C.

14 Test report

The test report shall contain the following information for each specimen:

a) name of manufacturer (and submitter where applicable);

b) date of test;

c) composition or generic identification;

d) specimen mass, expressed in grams;

e) specimen thickness, expressed in millimetres;

f) details of specimen preparation;

g) specimen mounting or other special mounting procedures used;

h) time to sustained flaming, expressed in seconds;

i) heat release rate curve;

j) peak heat-release rate, expressed in kilowatts per square metre;

NOTE It is possible that certain specimens might not show any visible signs of combustion or thermal degradation but can release heat.