TECHNICAL
SPECIFICATION

ISO/TS
5660-4

First edition
2016-12-15

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

Part 4:
Measurement of low levels of heat release

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/TS 5660-4:2016(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.

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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

A list of all parts in the ISO 5660 series can be found on the ISO website.

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

Part 4:
Measurement of low levels of heat release

WARNING — The test procedures involve high temperatures and combustion processes. Therefore, hazards can exist for burns, ignition of extraneous objects or clothing, and for 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 or decomposition products, or large amounts of moisture may produce violent releases of combustible gases or water vapour during testing.

Materials with high release rates normally tested in ISO 5660-1 **would give dangerous conditions if tested in this apparatus. To ensure this does not happen, specimens shall first be tested at the smaller 100 × 100 mm** ISO 5660-1 **specimen size to check approximate heat release rates before proceeding.**

1 Scope

This document specifies a method for evaluating materials and products that produce low levels of heat release when exposed to high irradiance levels typical of fully developed fires. It differs from ISO 5660-1 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.

The test method described in this document 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 0,75 MJ/m^2 to 15 MJ/m^2.

NOTE 1 The test method for specimens that yield moderate to high total heat release is described in ISO 5660-1. The information obtained from this test method in this document can also be used for fire safety engineering purposes.

NOTE 2 As in ISO 5660-1, the heat release rates are not measured directly and absolute but are theoretically calculated using the empirically derived constant of proportionality that links heat released and measured oxygen consumed.

2 Normative references

The following documents are referred to in text in such a way that some or all of their content constitutes requirements 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 291, *Plastics — Standard atmospheres for conditioning and testing*

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

ISO 5660-1:2015, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method)*

ISO 5660-3, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 3: Guidance on measurement*

ISO/TR 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 14934-3:2012, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
emissivity
ratio of the radiation emitted by a radiant source to the radiation that would be emitted by a black body radiant source at the same temperature

Note 1 to entry: Emissivity is dimensionless.

[SOURCE: ISO 13943:2008, 4.75]

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 1 to entry: Convective heating is minimized in the horizontal specimen orientation. For this reason, the term “irradiance” is used instead of “heat flux” throughout this document 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

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 1 to entry: 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 and 10 s

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	m$^{1/2}$ g $^{1/2}$ K $^{1/2}$
Δh_c	net heat of combustion	kJ g^{-1}
$\Delta h_{c,eff}$	effective net heat of combustion	MJ·kg^{-1}
m	mass of the specimen	g
Δm	total mass loss	g
m_f	mass of the specimen at the end of the test	g
m_s	mass of the specimen at sustained flaming	g
$\dot{m}_{A,10-90}$	average mass loss rate per unit area between 10 % and 90 % of mass loss	g·m^{-2}·s^{-1}
m_{10}	mass of the specimen at 10 % of total mass loss	G
m_{90}	mass of the specimen at 90 % of total mass loss	G
$\dot{m}$	mass change rate of the specimen	g·s^{-1}
$\dot{m}_e$	mass flow rate in exhaust duct	kg s^{-1}
Δp	orifice meter pressure differential	Pa
$\dot{q}$	heat release rate	kW
$\dot{q}_A$	heat release rate per unit area	kW m^{-2}
$\dot{q}_{A,max}$	maximum value of the heat release rate per unit area	kW m^{-2}
$\dot{q}_{A,180}$	average heat release rate per unit area over the period starting at t_{ig} and ending 180 s later	kW m^{-2}
$\dot{q}_{A,300}$	average heat release rate per unit area over the period starting at t_{ig} and ending 300 s later	kW m^{-2}
$Q_{A,tot}$	total heat released per unit area during the entire test	MJ m^{-2}
r_o	stoichiometric oxygen/fuel mass ratio	1
t	time	s
t_d	delay time of the oxygen analyser	s
t_{ig}	time to ignition (onset of sustained flaming)	s
Δt	sampling time interval	s

Symbol	Designations	Unit
t_{10}	time at 10 % of total mass loss	s
t_{90}	time at 90 % of total mass loss	s
T_e	absolute temperature of gas at the orifice meter	K
X_{O_2}	oxygen analyser reading, mole fraction of oxygen	1
$X_{O_2}^0$	initial value of oxygen analyser reading	1
$X_{O_2}^{-1}$	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 × 10^3 kJ of heat are released per 1,0 kg of oxygen consumed. Optionally, additional measurements of carbon dioxide and carbon monoxide can be made and used in calculation of heat release. The apparatus procedures and calculation methods described in Annex C are then applicable.

Specimens are exposed in ambient air conditions, while being subjected to an irradiance of 50 kW·m^{-2} in the presence of a spark ignition source. Alternatively, testing may be conducted at an exposure of 75 kW·m^{-2}. The changes in oxygen, O_2, concentration of gases and exhaust gas mass 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 document, 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 mass 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 irradiance.

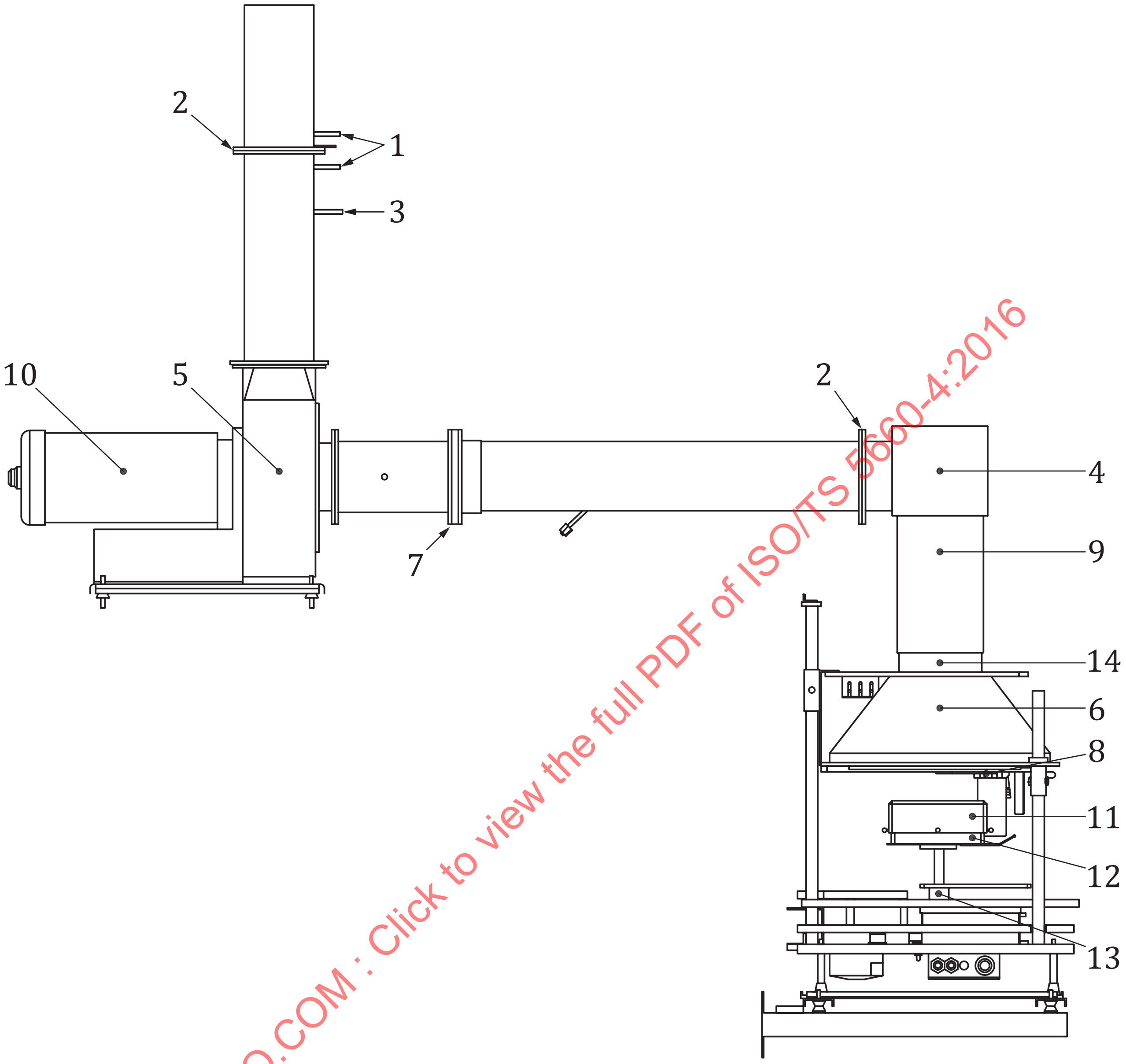
In order to measure heat release of materials or products that produce low levels of heat release when exposed to high irradiances typical of fully developed fires, this part differs from ISO 5660-1 by prescribing a larger specimen size (150 mm × 150 mm), a 50 % lower exhaust gas volumetric flow rate, direct connection between the plenum and heater to ensure collection of all the combustion gases, more stringent specifications for the oxygen analyser to improve accuracy, and a specified irradiance level at which to conduct testing. This document is designed to test samples with peak heat release of <200 kW/m^2 and total heat release of 0,75 MJ/m^2 to 15 MJ/m^2.

PMMA is typically used to check the general operation of a cone calorimeter in ISO 5660-1. PMMA shall not be used with this test method as the heat release rate is too high.

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
2	orifice plates
3	thermocouple (located on stock centreline)
4	plenum with extension piece
5	blower
6	heater
7	gas sampling probe
8	spark plug
9	vertical connection
10	blower motor
11	retainer frame and specimen
12	specimen holder
13	weighing device
14	expansion flange

Figure 1 — Apparatus

6.2 Cone-shaped radiant electrical heater

The specimen shall be irradiated using a heater similar to that used in ISO 5660-1 but larger in dimension and constructed such that it be capable of producing irradiance on the surface of the specimen of up to 80 kW m^2. The irradiance shall be uniform within the central 100 mm × 100 mm area of the exposed specimen surface, to within ±2 %, and to ±3 % over the entire specimen surface.

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 been proven suitable but Inconel or other high-performance materials are also acceptable), symmetrically disposed and in contact with, but not welded to, the heater element, either 3,0 mm outside diameter sheathed thermocouples with exposed hot junction or 1,0 mm to 1,6 mm outside diameter sheathed thermocouples with unexposed hot junction shall be used.

NOTE A heater having a total length of 12 m, lower outer diameter of 350 mm and top outer diameter of 150 mm with a power input of 15 kW has been found suitable.

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, either:

a) water-cooled and coated with a durable matte 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.

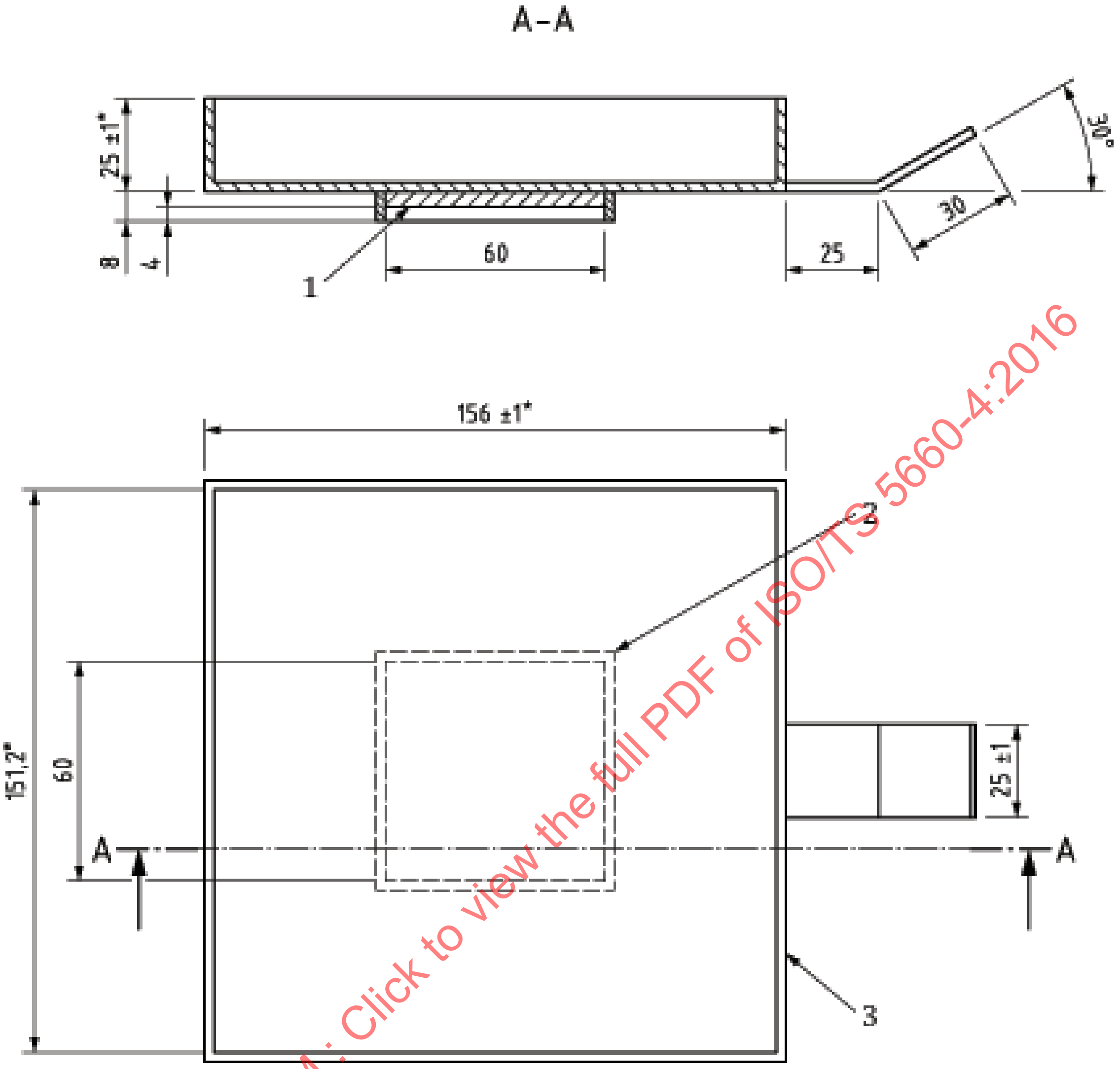
6.5 Weighing device

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 10.1.3. The output of the weighing device shall not drift by more than 1 g over a 30 min period, as determined with the calibration described in 10.1.4.

6.6 Specimen holder

The specimen holder shall be as shown in Figure 2. 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.

All dimensions in mm



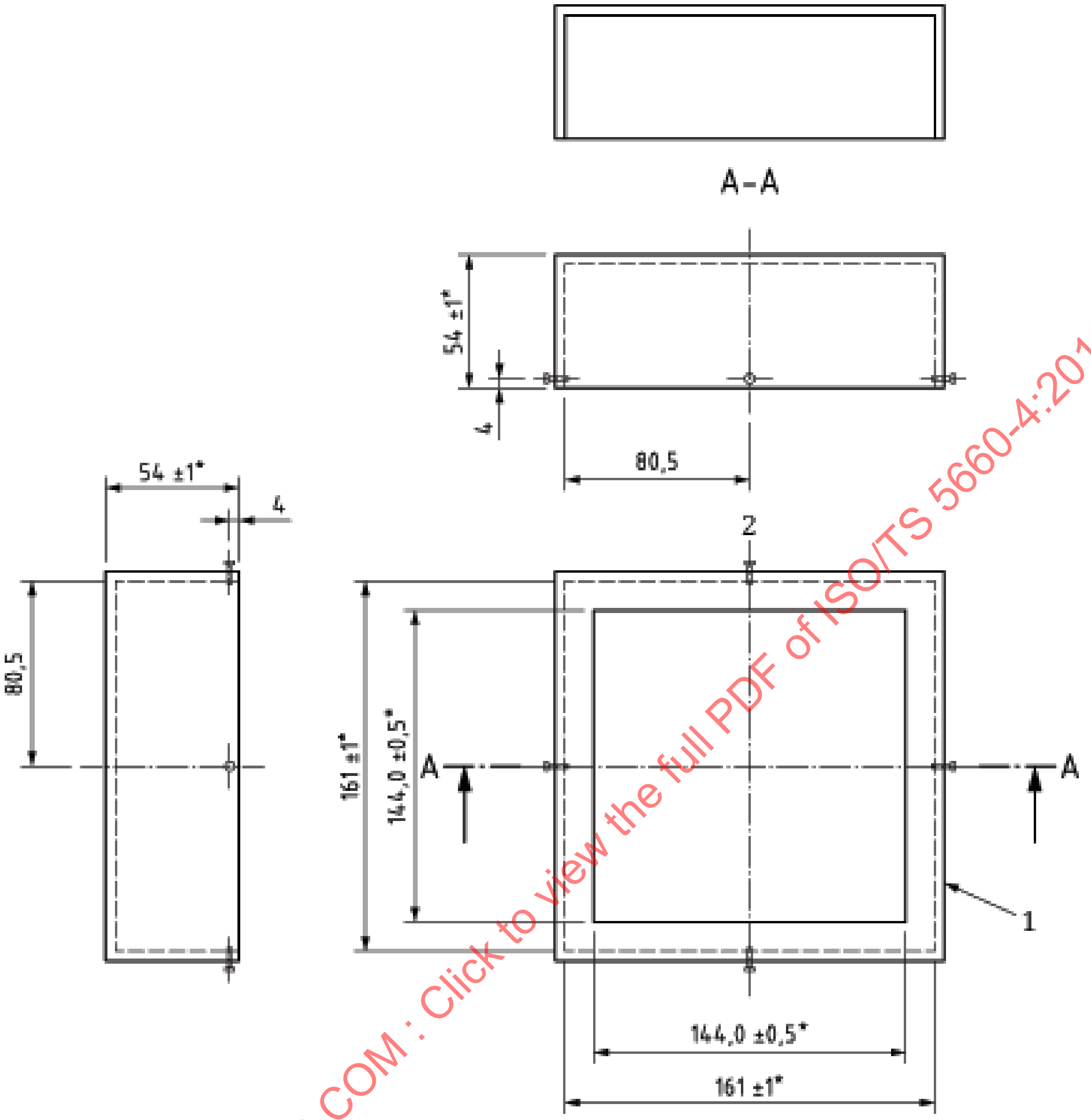
Key

1 stainless (mill smooth)
2 spot weld, four corners
3 2,4 ± 0,15mm stainless steel

Figure 2 — 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 3. The retainer frame shall have an appropriate means to secure it to the specimen holder with the specimen in position.



Key

1 1,9 ± 0,1 mm stainless steel

2 M4 screws (4 pcs)

Figure 3 — 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, intake and exhaust ducts for the fan, and an orifice plate flow meter (see Figure 1). The exhaust system shall be capable of developing flows up to 0,018 m^3/s, under standard conditions of temperature and pressure. The recommended location of the fan is indicated on Figure 4. As an alternative, it is acceptable to locate the fan further downstream and to have the measuring orifice before the fan, provided that the requirements described in the remainder of this clause are fulfilled.

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

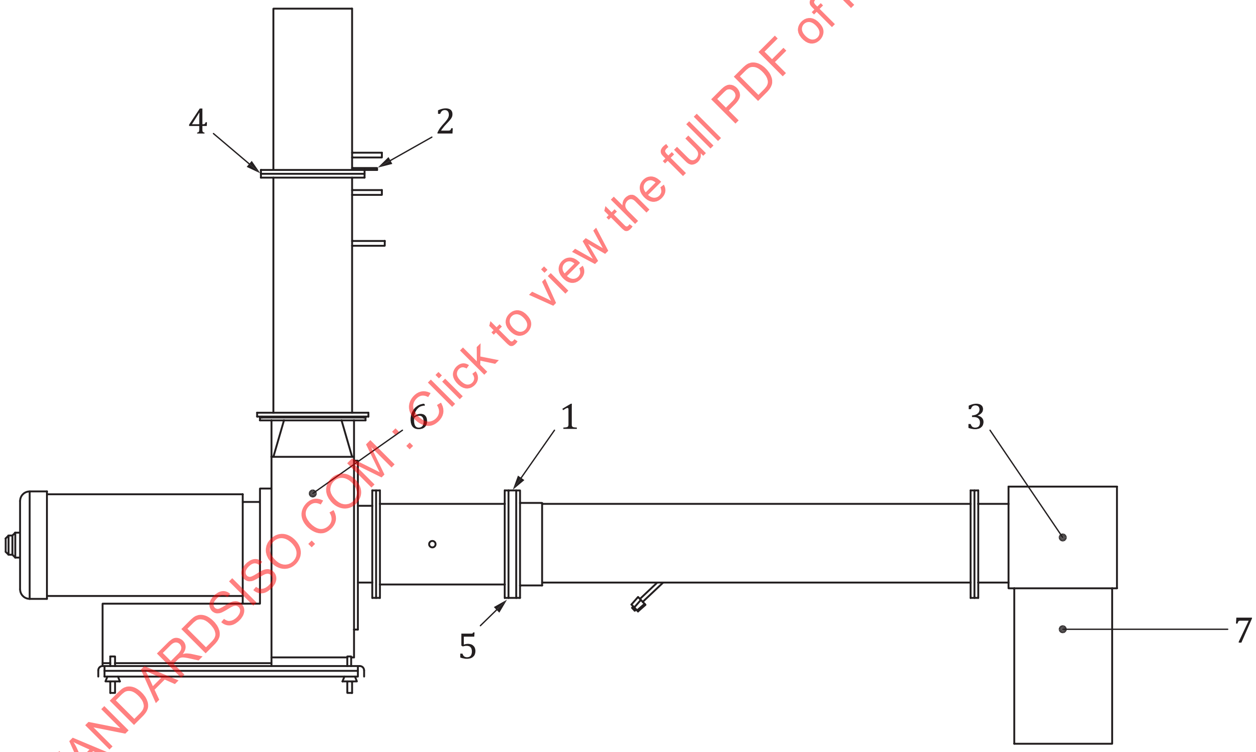
A gas sampling ring probe (sample holes face blower) sampler shall be located in the fan intake duct for gas sampling, (685 ± 15) mm from the plenum (see Figure 4). The gas sampling ring probe (sample holes face blower) shall contain 12 small holes with a diameter of (2,2 ± 0,1) mm, to average the stream 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 mass 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 on Figure 4. The fan shall be located as indicated in Figure 5.

NOTE As an alternative, the fan can 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.



Key

1	gas sampling ring probe
2	thermocouple
3	plenum with extension piece
4	orifice plate
5	gas sampling ring probe (sample holes face blower)
6	fan
7	vertical connection

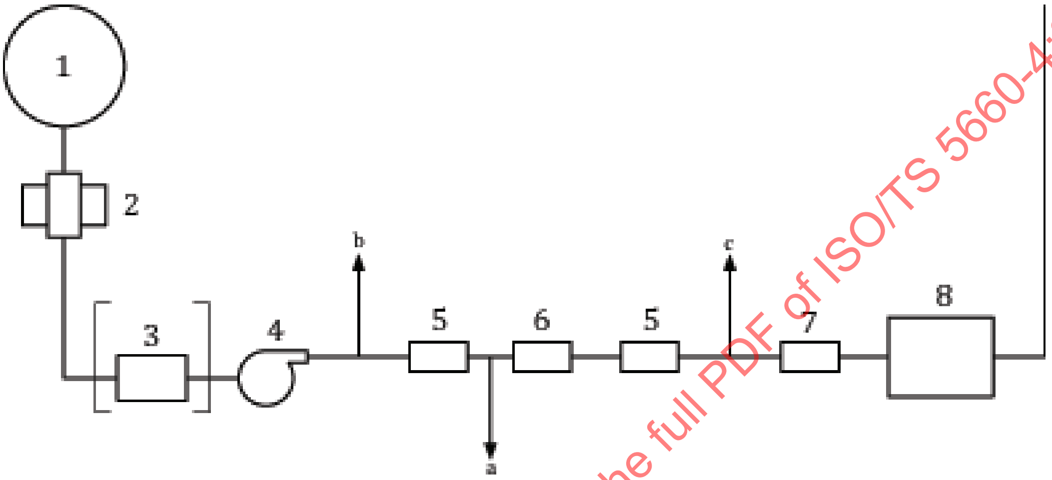
Figure 4 — Exhaust system

6.9 Gas sampling apparatus

Gas sampling apparatus incorporates a pump, filters to prevent entry of soot, facilities for removing 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.

A schematic view of an example of the gas sampling apparatus is shown in Figure 5. Other arrangements which satisfy the requirements may be used. The transport delay time of the oxygen analyser, t_d, shall be determined according to 10.1.5, and shall not exceed 60 s.

NOTE If an (optional) 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 C).



Key

1 gas sampling ring probe (sample holes face blower)
2 particulate filter
3 cold trap and drain (optional)
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 5 — 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.

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 30 µl/l drift of oxygen over a period of 30 min, and a noise of not more than 30 µl/l 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 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 10.2.4). 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 (100 ± 0) kW·m^{-2} and a diameter of approximately 12,5 mm. The sensing surface shall be coated with a durable matte 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 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 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

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 document, in particular for experiments in 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.

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.

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;

NOTE An essentially flat surface is considered to be a surface whose irregularity from a plane does not exceed ±1 mm.

b) a surface irregularity that is evenly distributed over the exposed surface, provided that

   1) 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

   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 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 has been tested in a modified form, and clearly describe the modification.

7.2 Asymmetrical products

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

7.3 Materials of short burning time

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

7.4 Composite specimens

Composite specimens 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 Products with unknown burning behaviour

This document is designed to test samples with peak heat release of < 200kW/m^2 and total heat release of 0,75 MJ/m^2 to 15 MJ/m^2. If there is no previous experience in testing the product and it is not known what the peak and total heat release rate is at the given test conditions, the product shall first be tested in accordance with ISO 5660-1.

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 $150^{\ 0}_{-2}$ 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 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 which would normally be attached to a well-defined substrate, it shall be tested in conjunction with that substrate using the recommended fixing technique, for example bonded with the appropriate adhesive or mechanically fixed. In the absence of a unique or well-defined substrate, an appropriate substrate for testing shall be selected in accordance with ISO/TR 14697.

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

8.2 Conditioning of specimens

Before the test, specimens shall be conditioned to constant mass at a temperature of (23 ± 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 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 in accordance with ISO 291. 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 to be tested may be used to preshape 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

It is imperative that the following good laboratory and operating procedures are practised at all times.

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

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

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

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

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

10 Calibration

10.1 Initial calibrations

10.1.1 General

The calibrations in this subclause shall be performed, when first commissioning a cone calorimeter; or after maintenance, repair or replacement of the heater assembly the weighing device (10.1.3 and 10.1.4), the oxygen analyser or other major components of the gas analysis system (10.1.5). 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 volumetric flow rate of (0,012 ± 0,002) m^3/s and an irradiance of (50 ± 1) $kW{\cdot}m^{-2}$. 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 volumetric flow rate of (0,012 ± 0,002) m^3/s. Determine the delay time of the oxygen analyser by delivering a methane mass flow rate equivalent to 1 kW to the calibration burner. Light the burner outside the hood and allow 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 1 For the purpose of measurement of the oxygen analyser delay and response time, the methane mass flow rate need not be controlled accurately, because the delay and response time is not sensitive to the oxygen level.

NOTE 2 This procedure should be repeated if the test is repeated at (0,018 ± 0,002) m^3/s

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 volumetric 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 volumetric 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 and at 30 min establishes the short-term drift. Determine the noise by computing the root-mean-square deviation around the linear trend line according to Formula (1):

$$\mathrm{rms} = \sqrt{\frac{\sum_{i=1}^{n} x_i^{\;2}}{n}} \tag{1}$$

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

Record this rms noise value in terms of parts per million 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 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 to (600 ± 10) 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 volumetric flow rate of (0,012 ± 0,002) m^3/s. For zeroing, feed the analyser with oxygen-free nitrogen gas, with the same volumetric 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 analyser volumetric flow rate and set to be equal to the volumetric flow rate 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 volumetric 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 mass flow rate corresponding to q_b = (1 ± 0,1) kW based on the net heat of combustion of methane (50,0 × 103 kJ/ kg). Allow 3 min for the methane flow to reach a constant rate then collect data at 5 s intervals over a 3 min period.

The orifice constant C shall be calculated according to Formula (2), using averages over the 3 min period of the measured values of $\dot{q}_b$, T_e, ΔP, and X_{O_2}. $X_{O_2}^0$ is determined as the average of the oxygen analyser output measured during the 1 min baseline measurements.

An 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. Allow 10 min for the cone heater to reach thermal stability at the set point.

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 14934-3:2012, Annex D.

10.3.2 Linearity of heat release rate measurements

At maximum intervals of 100 working hours, with the instrument calibrated at 1 kW according to 10.2.4, perform a further calibration with a mass flow rate corresponding to 2 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 6 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 both moisture traps. Replace the chemical 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 volumetric flow rate and pressure as for the sample gases, from a nitrogen source connected as close as possible to the gas sampling ring probe (sample holes face blower). 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 6.5 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 volumetric 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 the irradiance level of 50 $kW \cdot m^{-2}$. Alternatively, testing may be conducted at an exposure of 75 $kW \cdot m^{-2}$ provided that there is no ignition of the specimen at the 50 $kW \cdot m^{-2}$ irradiance level, and there is evidence of continued combustion after the 20 min test duration.

11.3 Test procedure

Materials with high heat release rates normally tested in ISO 5660-1 would give dangerous conditions if tested in this apparatus. To ensure this does not happen, a preliminary test should be undertaken following this procedure, but using the smaller size (100 mm × 100 mm) ISO 5660-1 specimen to check approximate heat release rates before proceeding.

11.3.1 Start data collection. Collect at least 60 s of baseline data prior to starting a test. The scan interval shall be as per 7.3.

11.3.2 Insert the radiation shield (which must be cooler than 100 °C) in position. Remove the thermal barrier protecting the weighing device.

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 test and ignition timer. Move the spark plug into place, and turn on spark power.

11.3.5 Record the times when flashing or transitory flaming occurs. When sustained flaming occurs (reported as the time at which flaming was initially observed, not when the 10 s period elapsed) 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.

If smoke is being lost through the cone heater, the test should be repeated at 18 l/s, with a note explaining that this will reduce the resolution.

11.3.6 At the end of the time period specified in 11.3.5, put back the radiation shield position, remove the specimen holder with specimen from the test position and remove the sample from the holder and wait until all combustion products are gone. Record the end of test value for the oxygen analyser. The value should be within 100 ppm from the value recorded at the beginning of the test. If the value is not within the 100 ppm value, the test shall be run again.

The test may be stopped if explosive spalling or excessive swelling occurs. 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 expands sufficiently prior to ignition to touch the spark plug or expands up to the plane of the heater base during combustion.

13 Calculations

13.1 General

The formulae in this subclause assume that only O_2 is measured using the gas analysis system in Figure 5. Appropriate formulae 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 5660–1:2015, Annex F. If CO_2 is removed from the O_2 sampling lines (even when CO_2 is separately measured), then Formulae (2) to (4) shall be used.

13.2 Calibration constant for oxygen consumption analysis

The heat release rate 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 Formula (2):

$$C = \frac{\dot{q}_\mathrm{b}}{(12,54 \times 10^3)(1,10)} \sqrt{\frac{T_\mathrm{e}}{\Delta p}} \cdot \frac{1,105 - 1,5X_{\mathrm{O}_2}}{X^0_{\mathrm{O}_2} - X_{\mathrm{O}_2}} \qquad (2)$$

where

$\dot{q}_\mathrm{b}$ corresponds to the rate of heat release (in kW) of the methane supplied (see 10.2.4);

$(12{,}54 \times 10^3)$ is $\Delta h_c/r_o$ for methane, in kJ/kg;

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 Formula (3):

$$X_{\mathrm{O}_2}(t) = X^1_{\mathrm{O}_2}(t + t_\mathrm{d}) \qquad (3)$$

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

$$\dot{q}(t) = \Delta h_\mathrm{c} / r_\mathrm{o})(1,10)C\sqrt{\frac{\Delta p}{T_\mathrm{e}}} \cdot \frac{X^0_{\mathrm{O}_2} - X_{\mathrm{O}_2}}{1,105 - 1,5X_{\mathrm{O}_2}} \qquad (4)$$

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 $X^0_{\mathrm{O}_2}$ is determined as the average of the oxygen analyser output measured during the last 1 min of the baseline measurements.

NOTE The value of $\Delta h_\mathrm{c}/r_\mathrm{o}$ = 13,1 MJ/kg O_2 is given within 5 % according to ISO 5660-3. However, this value is adapted to the carbonaceous fuel and a consistent combustion. For the purpose of this standard, materials and products with very low levels of heat release are tested. This means that the value of $\Delta h_\mathrm{c}/r_\mathrm{o}$ can significantly deviate from the theoretical value, well beyond the 5 %. Also, if a value of $\Delta h_\mathrm{c}/r_\mathrm{o}$ is not known for the tested fuel, heat release rate measurements should be considered as conventional.

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

$$\dot{q}_A(t) = \dot{q}(t) / A_s \qquad (5)$$

where A_s is the initially exposed area of the sample.

13.4 Exhaust duct mass flow rate

Calculate the mass flow rate in the exhaust duct, in kg/s, from Formula (6):

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

13.5 Mass loss rate

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

For the first scan (i = 0):

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

For the second scan (i = 1):

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

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

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

For the next to last scan (i = n–1):

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

For the last scan (i = n):

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

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 %, from Formula (12):

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

where

$\Delta m = m_s - m_f$;

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

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

NOTE Equations for the effective heat of combustion, $\Delta h_{c,eff}$, are given in A.1.8 and ISO 5660–1:2015, 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) name of operator

c) date of test;

d) composition or generic identification;

e) specimen mass, in kg;

f) specimen thickness, in mm;

g) details of specimen preparation;

h) specimen mounting or other special mounting procedures used;

i) irradiance, in kW·m^{-2} and exhaust flow rate, in m^3/s;

j) time to sustained flaming, in s;

k) heat release rate curve;

l) peak heat release rate, in kW·m^{-2};

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

m) total heat released by specimen, in MJ/m^2;

n) mass remaining after test, in kg;

o) values determined in H, J and K above, averaged for all specimens;

p) additional observations, if any;

q) difficulties encountered in testing, if any.

Annex A

(informative)

Overview

A.1 Background

A.1.1 Overview

A brief history to give insight into the development of the method described in this document and to provide a rationale for the design of the various features of the apparatus is discussed in Reference [14]. More recent work on the development of the larger conical heater to be used in this document can be found in in Reference [17].

A.1.2 Heat release rate measurements

A.1.2.1 The rate of heat release is one of the most important variables, in many cases the single most important variable, in determining the hazard from a fire. This rate of heat release is the total rate, as a function of time and irradiance. With many items, composed of many surfaces, contributing to a fire, the evaluation of rate of heat release is quite complex. For each separate surface, it should first be determined when, if at all, it will become ignited. The size of the fire from any already burning items should be known, since that constitutes the external irradiance to nearby items. Next, the flame spread over the surface in question should be evaluated. The rate of heat release from the whole surface can then be evaluated knowing the rate of heat release per unit area for a given irradiance, as a function of time. This last quantity is the only one that can be measured in a bench-scale test.

A.1.2.2 The total heat release from a fire then involves a summation of the heat release per unit area over the surface. Also to be considered is the fact that some elements may burn out and then no longer contribute to the fire. This procedure is conceptually straightforward but can be very cumbersome to compute.

A.1.2.3 Many common combustibles do not have the geometrically simple surfaces that are required to make computations of this kind. Other complications, such as melting, dripping, or collapsing can also preclude a detailed mathematical analysis. In such cases a simpler, more empirical model is appropriate. An example of the use of bench-scale heat release rate measurements in deriving a fire hazard assessment is available.[1] A number of apparatus have been developed over the years for measuring rate of heat release; most of these have been reviewed in detail.[2] Traditionally, the simplest measurement scheme is a direct measurement of flow enthalpy from a chamber thermally lagged to present an adiabatic environment. A truly adiabatic apparatus, with the use of guard heaters, would be possible but would also be prohibitively expensive and has not been implemented. A combustion chamber that is insulated in a simpler manner leads to a significant under measurement of the heat release, so that only an empirical calibration is possible. An example of such an insulated chamber method is ASTM Test Method E906. Furthermore, that calibration may be sensitive to the radiant fraction (or sootiness) of the combustible.[3][4] A more advanced scheme is an isothermal instrument, rather than an adiabatic one, with the heat release rate taken to be that which should be supplied by a substitution burner to maintain isothermal conditions.[5] This scheme gives better results, since only second-order heat loss error terms remain; however, its practical implementation is complex and costly.

A.1.2.4 It can be concluded that it is difficult to measure heat directly without losing some of it. However, it is simple to capture all combustion products without losing any and to measure the oxygen levels in that stream. Heat release can be computed from such measurements with the availability of the oxygen consumption principle.[6] This principle states that for most common combustibles an amount of heat equal to 13,1 kJ × 10^3 kJ is released for each kilogram of oxygen consumed from the air stream. This constant varies only about ±5 % for most common combustibles; certain exceptions are given in.[6] The method remains useful even if a significant fraction of the products become CO or soot, rather than CO_2; in these cases, correction terms are known[6][7] and can be applied.

A.1.3 Variability of rate of heat release results

The rate of heat release depends on many factors, some of which cannot be controlled. Samples that produce a surface char, a layer of adherent ash, or those that are composites or laminates, may not attain a steady-state release rate. Thermally thin specimens, that is, specimens whose unexposed surface changes temperature during the period of test will also not attain a steady-state release rate. Therefore, release rates for a given material will depend, for example, on how the material is used, its thickness, and the method of mounting.

A.1.4 Heater design

A.1.4.1 Experience with various rate-of-heat-release measurement techniques suggests that for minimal errors in irradiance, the specimen should see only

a) a thermostatically controlled heater,

b) a water-cooled plate, or

c) open air.

Nearby solid surfaces, if they are not temperature-controlled, can rise in temperature due to specimen flame heating and then act as further sources of radiation back to the specimen. Further, when oxygen consumption is used as the measurement principle, a gas-fired heater is not desirable because it can contribute a noisy baseline to the oxygen readings, even though it can be subtracted out in steady-state.

A.1.4.2 A heater in the shape of a truncated cone was first explored for use in an ignitability apparatus by ISO (see ISO 5657). The heater adopted in the present method is similar, but not identical to the device described in ISO 5657. The main differences include higher irradiances, temperature control, and more rugged design details. In the horizontal orientation, the conical shape approximately follows the fire plume contours while the central hole allows the stream to emerge without impacting on the heater. A thin layer of cool air is pulled along and the flames do not make contact with the sides of the cone. The central hole has a further function; in its absence the middle of the specimen would receive a higher irradiance than the edges. With the hole, the irradiance is uniform to within ±2 %.

A.1.5 Pilot ignition

Ignition of test specimens in many apparatus is achieved by a gas pilot. This tends to have numerous difficulties such as sooting, deterioration of orifices and contribution to heat release rate. It is difficult to design a pilot that can be centrally located over the specimen, is resistant to blowout, and yet does not apply an additional irradiance to the specimen. A point of elevated heating on the specimen makes it difficult to analyse mathematically the response of the specimen. An electric spark is free of most of these difficulties, requiring only an occasional cleaning and adjustment of the electrodes. For these reasons, an electric spark ignition was adopted.

A.1.6 Back face conditions

The heat losses through the specimen back face can have an influence on the burning rate near the end of its burning time. For reproducible measurements, the losses through the back face should be standardized. The simplest theoretical boundary conditions, an adiabatic boundary, or an isothermal one at ambient temperature, are not achievable. However, a reasonable approximation to the former can be made by using a layer of an insulating material. This is easier to do for the horizontal orientation case, in which case a very low density refractory blanket is used.

A.1.7 Oxygen analyser

The analyser should be of the paramagnetic type, with baseline noise and short-term drift of approximately ±25 ppm oxygen. Other types of analysers (for example, electrochemical and catalytic) generally cannot meet this requirement. Paramagnetic analysers also exhibit an intrinsically linear response. The linearity is normally better than can be determined with ±0,1 % oxygen gas mixtures. Since an oxygen analyser is sensitive to stream pressures, either the readings have to be compensated with an absolute pressure transducer, connected to the analyser, or the pressure has to be mechanically regulated both against flow fluctuations and atmospheric pressure variations. The analyser and the pressure regulating or measuring devices should be located in a constant temperature environment to avoid flow errors.

NOTE For additional information pertaining to the oxygen analyser delay and response time as well as noise and drift, it is suggested that 10.1.4 and 10.1.5 be performed for one day, thereby providing a better understanding of the performance of the O_2 analyser.

A.1.8 Effective heat of combustion

The effective heat of combustion is a constant during combustion of homogeneous specimens having only a single mode of degradation and is less than the value of the theoretical net heat of combustion. Examples of a material with a single mode of degradation and, therefore, a constant effective heat of combustion include most organic liquids. Cellulosic products, by contrast, typically show more than one mode of degradation and a varying effective heat of combustion. For materials having more than one mode of degradation, or for composites or non-homogeneous materials, the effective heat of combustion is not necessarily constant.

A.1.9 Specimen mounting methods

The test method presented in this document is a general method, suitable for testing different types of products and materials. These are not the only specimen mounting methods available to the testing laboratory. Reference [9] suggests some additional procedures. For more unusual specimen types, the testing laboratory will have to devise appropriate mounting methods. Since different mounting methods could give different test results, the method used should be documented in the test report. Since test results are inevitably affected by such mounting devices, they should not be used unless prior testing indicates that they are necessary to alleviate anomalous burning conditions.

Annex B

(informative)

Calibration of the working heat flux meter

The inter-comparison of working and reference standard heat flux meters specified in 6.13, may be made using the conical heater (6.2), with each heat flux meter mounted in turn in the calibration position. Care should be taken to allow the whole apparatus to attain thermal equilibrium. Alternatively, a specially built comparison apparatus may be used (for example, devices specified in BS 6809:1987).

The use of two, instead of just one reference standard, provides a greater safeguard against change in sensitivity of the reference instruments.

Annex C
(informative)

Calculation of heat release with additional gas analysis

C.1 General

The formulae in Clause 13 used to calculate heat release rate assume CO_2/CO are removed from the gas sample in a chemical scrubber before O_2 is measured, as indicated in Figure 5. Some laboratories are equipped to measure CO_2/CO in which case it is not necessary to remove the CO_2/CO from the O_2 line giving the advantage that the use of chemical scrubbing agent, which is costly and requires careful handling, can be avoided.

If formulae in this annex are used to obtain the heat release rate values, the response time(s) of the additional gas analyser(s) used must closely match the response time of the oxygen analyser. If this requirement cannot be met, this annex shall not be used for obtaining the heat release rate. Silica gel/Drierite shall not be used as the drying agent if a CO_2/CO analyser is used in the system.

In this annex, formulae are given which are to be used when CO_2/CO is measured but not scrubbed out of the sampling lines. Two cases are considered:

- in the first case, part of the dried and filtered sample stream is diverted into infrared CO_2 and CO analysers (see option in Figure 5);
- in the second case, a water vapour analyser is also added.

To avoid condensation, the measuring of H_2O concentration in the flow of combustion products requires a separate sampling system with heated filters, heated sampling lines, and heated analyser.

C.2 Symbols

The following new symbols are used in this annex:

Symbol	Designation	Unit
E	heat of combustion per unit mass of oxygen consumed (= $\Delta h_c/r_0$)	MJ/kg
E_{CO}	heat of combustion per unit mass of oxygen consumed for CO	MJ/kg
H	relative air humidity	%
M_a	molecular mass of air	kg/kmol
M_e	molecular mass of the combustion products	kg/kmol
M_{O_2}	molecular mass of the oxygen	kg/kmol
p	ambient pressure	Pa
T_a	ambient temperature	K
t_d^1	delay time of the CO_2 analyser	s
t_d^2	delay time of the CO analyser	s
t_d^3	delay time of the H_2O analyser	s