

INTERNATIONAL STANDARD

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Fire tests – Reaction to fire –

Part 1:

Rate of heat release from building products (Cone calorimeter method)

Essais au feu – Réaction au feu –

Partie 1: Débit calorifique des produits du bâtiment (Méthode au calorimètre conique)



Reference number
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Contents

	Page
1 Scope	1
2 Normative references.....	1
3 Definitions	1
4 Symbols	2
5 Principle	2
6 Apparatus	3
7 Suitability of a product for testing	4
8 Specimen construction and preparation	5
9 General	6
10 Calibration	6
11 Test procedure	7
12 Calculations	8
13 Test report	8
 Annexes	
A Commentary and guidance notes for operators	21
B Resolution, precision and bias	23
C Mass loss rate and effective heat of combustion	25
D Summary of test report	26
E Calibration of the working heat flux meter	28
F Calculation of heat release with additional gas analysis	29
G Bibliography	31

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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.

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.

International Standard ISO 5660-1 was prepared by Technical Committee ISO/TC 92, *Fire tests on building materials, components and structures*, Subcommittee SC 1, *Reaction to fire*.

ISO 5660 consists of the following parts, under the general title *Fire tests – Reaction to fire*:

- *Part 1: Rate of heat release from building products (Cone calorimeter method)*
- *Part 2: Dynamic smoke measurement*
- *Part 3: Burning rate of building products*

Annexes A to G of this International Standard are for information only.

Introduction

Fire is a complex phenomenon: its behaviour and its effects depend upon a number of interrelated factors. The behaviour of materials and products depends upon the characteristics of the fire, the method of use of the materials and the environment in which they are exposed. The philosophy of reaction to fire tests is explained in ISO/TR 3814.

A test such as is specified in this International Standard deals only with a simple representation of a particular aspect of the potential fire situation typified by a radiant heat source and a spark. It cannot alone provide any direct guidance on behaviour or safety in fire. A test of this type may, however, be used for comparative purposes or to ensure the existence of a certain quality of performance (in this case heat release rates considered to have a bearing on fire performance generally). It would be wrong to attach any other meaning to performance in this test.

The term heat release rate is defined in ISO/IEC 52 Guide as the calorific energy released per unit time by a material during combustion under specified test conditions. It is one of the fundamental properties of fire and should almost always be taken into account in any assessment of fire hazard since it significantly affects the development of fire in a building.

This test is based on part of the ASTM standard E 1354. However, not all the equipment in E 1354 is the subject of this International Standard. Some of the additional instrumentation, in particular that measuring the properties of smoke and its rate of production, is the subject of discussion in ISO/TC 92.

This test does not rely upon the use of asbestos-based materials.

Fire tests – Reaction to fire –

Part 1:

Rate of heat release from building products (Cone calorimeter method)

1 Scope

This International Standard specifies a method for assessing the heat release rate of essentially flat products exposed to controlled levels of radiant heating with or without an external igniter. The rate of heat release is determined by measurement of the oxygen consumption derived from the oxygen concentration and the flow rate in the combustion product stream. The time to ignition (sustained flaming) is also measured in this test. Products with surface irregularities may be tested according to specific requirements.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5660. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5660 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO/TR 3814:1989, *Tests for measuring "reaction-to-fire" of building materials – Their development and application.*

ISO/IEC Guide 52:1990, *Glossary of fire terms and definitions.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO/IEC Guide 52 and the following definitions apply.

3.1 assembly: Fabrication of materials or composites, for example sandwich panels.

NOTE 1 This may include an air gap.

3.2 composite: Combination of materials which are generally recognized in building construction as discrete entities, for example coated or laminated materials.

3.3 essentially flat surface: Surface whose irregularity from a plane does not exceed ± 1 mm.

3.4 flashing: Existence of flame on or over the surface of the specimen for periods of less than 1 s.

3.5 ignition: Onset of sustained flaming as defined in 3.12.

3.6 irradiance (at a point of a surface): Quotient of the radiant flux incident on an infinitesimal element of surface containing the point, by the area of that element.

NOTE 2 Convective heating is negligible in the horizontal specimen orientation. In the vertical orientation, it is small, but not negligible. Despite this contribution from convective heating, the term "irradiance" is used instead of "heat flux" throughout this International Standard as it best indicates the essentially radiative mode of heat transfer.

3.7 material: Single substance or uniformly dispersed mixture, for example metal, stone, timber, concrete, mineral fibre, polymers.

3.8 orientation: Plane in which the exposed face of the specimen is located during testing, either vertical or horizontally face upwards

3.9 oxygen consumption principle: Proportional relationship between the mass of oxygen consumed during combustion and the heat released.

3.10 product: Material, composite or assembly about which information is required.

3.11 specimen: Representative piece of the product which is to be tested together with any substrate or treatment.

NOTE 3 This may include an air gap.

3.12 sustained flaming: Existence of flame on or over the surface of the specimen for periods of over 4 s.

3.13 transitory flaming: Existence of flame on or over the surface of the specimen for periods of between 1 and 4 s.

4 Symbols

A_s	initially exposed surface area of the specimen, expressed in square metres (m ²)
C	calibration constant for oxygen consumption analysis, expressed in (m·kg·K) ^{1/2}
Δh_c	net heat of combustion, expressed in kilojoules per gram (kJ/g)
$\Delta h_{c,eff}$	effective net heat of combustion, expressed in kilojoules per gram (kJ/g)
m	mass of the specimen, expressed in kilograms (kg)
m_f	mass of the specimen at the end of the test, expressed in kilograms (kg)
m_i	mass of the specimen at sustained flaming, expressed in kilograms (kg)
\dot{m}	mass loss rate of the specimen, expressed in kilograms per second (kg/s)
\dot{m}_e	mass flow rate in exhaust duct, expressed in kilograms per second (kg/s)
Δp	orifice meter pressure differential, expressed in pascals (Pa)

\dot{q}	heat release rate, expressed in kilowatts (kW)
\dot{q}''	heat release rate per unit area, expressed in kilowatts per square metre (kW/m ²)
\dot{q}_{max}''	maximum value of the heat release rate, expressed in kilowatts per square metre (kW/m ²)
\dot{q}_{180}''	the average heat release rate over the period starting at t_{ig} and ending 180 s later, expressed in kilowatts per square metre (kW/m ²)
\dot{q}_{300}''	the average heat release rate over the period starting at t_{ig} and ending 300 s later, expressed in kilowatts per square metre (kW/m ²)
q_{tot}''	the total heat released during the entire test, expressed in megajoules per square metre (MJ/m ²)
r_O	stoichiometric oxygen/fuel mass ratio
t	time, expressed in seconds (s)
t_d	delay time of the oxygen analyser, expressed in seconds (s)
t_{ig}	time to ignition (sustained flaming), expressed in seconds (s)
Δt	sampling time intervals, expressed in seconds (s)
T_θ	absolute temperature of gas at the orifice meter, expressed in kelvin (K)
x_{O_2}	oxygen analyser reading, mole fraction of oxygen
$x_{O_2}^0$	initial value of oxygen analyser reading
$x_{O_2}^1$	oxygen analyser reading, before delay time correction

5 Principle

This test method is based on the observation that, generally, the net heat of combustion is proportional to the amount of oxygen required for combustion. The relationship is that approximately 13,1 × 10³ kJ of heat are released per kilogram of oxygen consumed. Specimens in the test are burned in ambient air conditions, while being subjected to a predetermined external irradiance within the range 0 kW/m² to 100 kW/m² and measurements are made of oxygen concentrations and exhaust gas flow rates.

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

6 Apparatus

6.1 General

The test apparatus shall be constructed as shown in figure 5; a cross-section through the heater is shown in figure 1 and an exploded view of horizontal and vertical orientations are given in figures 7 and 8. Untoleranced dimensions specified as critical shall have a tolerance of ± 1 mm.

All other dimensions are recommended values and should be followed closely.

6.2 Cone-shaped radiant electric heater, capable of horizontal or vertical orientation. The active element of the heater shall consist of an electrical heater rod, rated at 5 000 W at 240 V¹⁾, tightly wound into the shape of a truncated cone (see figure 1). The heater shall be encased on the outside with a double-wall stainless steel cone, packed with a refractory fibre material of approximately 100 kg/m³ density. The irradiance from the heater shall be capable of being held at a preset level by means of a temperature controller and three, type K, stainless steel sheathed thermocouples, symmetrically disposed and in contact with, but not welded to, the heater element (see figure 1). The thermocouples shall be of equal length and wired in parallel to the temperature controller. Either 3 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 may be used. The heater shall be hinged so that it can be swung into either a horizontal or vertical orientation. The heater shall be capable of producing irradiances on the surface of the specimen of up to 100 kW/m². The irradiance should be uniform within the central 50 mm \times 50 mm area of the specimen, to within ± 2 % in the horizontal orientation and to within ± 10 % in the vertical orientation.

6.3 Temperature controller, capable of holding the element temperature steady to within ± 2 °C. A suitable system is a "3-term" controller (proportional, integral and derivative) and a thyristor unit capable of switching currents up to 25 A at 240 V. The controller should have a temperature input range of 0 °C to 1 000 °C, a set scale capable of being read to 2 °C or better and automatic cold junction compensation.

The thyristor unit shall be of the "zero-crossing" and not of the "phase-angle" type.

NOTE 4 A desirable feature is a control which, in the event of an open circuit in the thermocouple line, will either cause the power to fall to near the bottom of its range, or cut the power off.

The heater temperature shall be monitored by a meter capable of being read to ± 2 °C or better; it may either be separated or incorporated into the temperature controller.

6.4 Load cell, for measuring specimen mass loss. The load cell shall have an accuracy of 0,1 g and it should preferably have a measuring range of 500 g and a mechanical tare adjustment range of 3,5 kg.

6.5 Specimen holders, different for the two orientations.

6.5.1 The horizontal specimen holder is shown in figure 2. The bottom of the holder shall be lined with a layer of low density (nominal density 65 kg/m³) refractory fibre blanket with a thickness of at least 13 mm. The distance between the bottom surface of the cone heater and the top of the specimen shall be adjusted to 25 mm by using the sliding cone height adjustment (see figure 1).

6.5.2 The vertical specimen holder is shown in figure 3 and includes a small drip tray to contain a limited amount of molten material. A specimen is installed in the vertical specimen holder by backing it with a layer of refractory fibre blanket (nominal density 65 kg/m³), the thickness of which depends on specimen thickness, but shall be at least 13 mm. A layer of rigid, ceramic fibre millboard shall be placed behind the fibre blanket layer. The millboard thickness shall be such that the entire assembly is rigidly bound together once the retaining spring clip is inserted behind the millboard. In the vertical orientation, the cone heater height is set so the centre lines up with the specimen centre.

6.6 Exhaust gas system, with flow measuring instrumentation.

6.6.1 The exhaust gas system shall consist of a high temperature centrifugal exhaust fan, a hood, intake and exhaust ducts for the fan and an orifice plate flow meter (see figure 4). The exhaust system shall be capable of developing flows from 0,012 m³/s to 0,035 m³/s.

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

6.6.3 A ring sampler shall be located in the fan intake duct for gas sampling, 685 mm from the hood (see figure 5). The ring sampler shall contain 12 small holes to average the stream composition with the holes facing away from the flow to avoid soot clogging.

6.6.4 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 centre-line and 100 mm upstream from the measuring orifice plate.

1) This requires a 30 A supply.

6.6.5 The flow rate shall be determined by measuring the differential pressure across a sharp edge orifice (internal diameter 57 mm) in the exhaust stack, at least 350 mm downstream from the fan, if the latter is located as shown on figure 4.

6.6.6 The geometry of the exhaust system is not so critical. Where necessary, small deviations from the recommended dimensions given in figure 4 are allowed, for example, the inner diameter of the duct and the orifice plates can be slightly different. Also the fan does not need to be at the exact location as indicated on figure 4, but may be located further downstream allowing for a more common type of fan to be used. In this case, undisturbed inflow distance to the gas sampling probe and the measuring orifice shall be sufficient for the flow to be uniformly mixed.

6.7 Gas sampling apparatus

Gas sampling apparatus is shown in figure 6 and 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 oxygen analyser, a further moisture trap and, if CO₂ is not measured, a trap for CO₂ removal.

NOTE 5 If an (optional) CO₂ analyser is used, the equations to calculate the rate of heat release can be different from those for the standard case (see clause 12 and annex F).

6.8 Ignition circuit

External ignition is accomplished by a spark plug powered from a 10 kV transformer. The spark plug shall have a gap of 3 mm. The transformer shall be of a type specifically designed for spark ignition use. The transformer shall have an isolated (unearthed) secondary to minimize interference with the data transmission lines. The electrode length and location of the spark plug shall be such that the spark gap is located 13 mm above the centre of the specimen in the horizontal orientation. In the vertical orientation, the spark plug shall be positioned so that the gap is located in the specimen face plane and 5 mm above the top of the holder.

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

6.10 Oxygen analyser, paramagnetic type, with a range of 0 % oxygen to 25 % oxygen. The analyser shall exhibit a linear response and drift of not more than ± 50 parts per million of oxygen (root-mean-square value) over a period of 30 min. Since oxygen analysers are sensitive to stream pressures, the stream pressure shall be regulated (upstream of the analyser) to allow for flow fluctuations and the readings from the analyser compensated with an absolute pressure regulator to allow for atmospheric pressure variations. The analyser and the absolute pressure regulator shall be located in a constant temperature environment. The oxygen analyser shall have a 10 % to 90 % of full-scale response time of less than 12 s.

6.11 Heat flux meter, of the Gardon (foil) or Schmidt-Boelter (thermopile) type with a design range of about 100 kW/m². The target receiving radiation, and possibly to a small extent convection, shall be flat, circular, of approximately 12,5 mm in diameter and coated with a durable matt black finish. The target shall be water-cooled.

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

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

This meter shall be used to calibrate the heater (figures 7 and 8). It shall be positioned at a location equivalent to the centre of the specimen face in either orientation during this calibration.

6.12 Calibration burner, constructed from a square-section brass tube with a square orifice covered with wire gauze through which the methane diffuses (figure 9). The tube is packed with ceramic fibre to improve uniformity of flow. The calibration burner is suitably connected to a metered supply of methane of at least 99,5 % purity.

6.13 Data collection and analysis system, having facilities for recording the output from the O₂ analyser, the orifice meter, the thermocouples and the load cell. The data collection system should have an accuracy corresponding to at least 50 parts per million of oxygen for the oxygen channel, 0,5 °C for the temperature measuring channels, and 0,01 % of full-scale instrument output for all other instrument channels. The system should be capable of recording data every 5 s for at least 1 h.

7 Suitability of a product for testing

7.1 Surface characteristics

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

- a) an essentially flat exposed surface;
- b) a surface irregularity which is evenly distributed over the exposed surface provided that
 - 1) at least 50 % of the surface of a representative 100 mm square area lies within a depth of 10 mm from a plane taken across the highest points on the exposed surface, or

- 2) for surfaces containing cracks, fissures or holes not exceeding 8 mm in width nor 10 mm in depth, the total area of such cracks, fissures or holes at the surface does not exceed 30 % of a representative 100 mm square area of the exposed surface.

NOTE 6 When an exposed surface does not meet the requirements of either 7.1 a) or 7.1 b), the product may be tested in a modified form complying as nearly as possible with the requirements given in 7.1. The test report should 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 Thin materials

This test method can prove unsuitable for excessively thin materials since insufficient data will be collected for the calculation of heat release rates. For some materials, reducing the data collection interval from 5 s to some shorter value can solve this problem.

7.4 Composite specimens

Composite specimens may be tested, provided they are prepared as specified in 8.3.

7.5 Dimensionally unstable materials

This test method can prove unsuitable for materials that change their dimensions substantially when exposed to the cone radiation, for example materials that intumesce or shrink away from the cone radiator, because the irradiance on the surface of the specimen at the time of ignition can differ significantly from that set initially. The precision of the method can be worse than that indicated in annex B for materials that behave in this way.

8 Specimen construction and preparation

8.1 Specimens

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

8.1.2 The specimens shall be representative of the product and shall be square with sides measuring (100_{-5}^0) mm.

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

8.1.4 For products with normal thickness of greater than 50 mm, the requisite specimens shall be obtained by cutting away the unexposed face to reduce the thickness to (50_{-3}^0) mm.

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

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

NOTE 7 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, then it shall be tested in conjunction with that substrate using the recommended fixing technique, for example, bonded with the appropriate adhesive or mechanically fixed.

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. In the case of specimens of less than 6 mm in thickness and which would be used with an air space adjacent to the unexposed face, the specimens shall be mounted so that there is an air space of at least 12 mm between its unexposed face and the refractory fibre blanket.

NOTE 8 This can be achieved by the use of a metal spacer frame.

8.2 Conditioning of specimens

Before the test, specimens shall be conditioned to constant mass at a temperature of (23 ± 2) °C, and a relative humidity of (50 ± 5) % in accordance with ISO 554.

NOTE 9 Constant mass is considered to be reached when two successive weighing operations, carried out at an interval of 24 h, do not differ by more than 0,1 % of the mass of the test piece or 0,1 g, whichever is the greater.

8.3 Preparation

A conditioned specimen shall be wrapped in a single layer of aluminium foil, of 0,03 mm to 0,05 mm thickness, with the shiny side towards the specimen, covering the unexposed surfaces.

Composite specimens shall be exposed in a manner typical of the end-use condition [for example, if used with an air gap (see 8.1.6), an air gap shall be included behind the specimen and within the aluminium foil]. They shall be

tested with the retainer frame (see figure 10, applicable to horizontal orientation testing only) and also prepared so that the sides are enveloped with the outer layer(s) or otherwise protected. If such methods are used, they shall be specified in the test report.

NOTE 10 The use of the retainer frame is recommended for testing in the horizontal orientation.

Figure 11 shows a wire grid suitable for testing specimens which move towards the heater. The exact mounting and retaining method shall be specified in the test report.

9 Test environment

The apparatus 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.

10 Calibration

10.1 Heater calibration

Adjust the temperature controller so that the conical heater produces the required irradiance, as measured by the heat flux meter, at the start of each test day, when changing to a new irradiance or when the conical heater orientation is changed.

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 and ensure that the controller is within its proportional band before beginning this calibration.

10.2 Oxygen analyser calibration

10.2.1 Preliminary calibrations

10.2.1.1 Determine the delay time of the oxygen analyser by delivering a methane flow rate equivalent to 5 kW to the calibration burner. The heater shall not be turned on for this calibration. Record the output of the analyser as the methane supply, turned on and ignited, reaches a steady value, and then returns to baseline after the supply is removed. In addition monitor the temperature of the exhaust orifice meter.

The turn-on delay is the time difference between the temperature reading reaching 50 % of its ultimate deflection and the oxygen reading reaching 50 % of its ultimate deflection. Calculate the turn-off delay similarly. The delay time t_d is the average of at least three turn-on and turn-off delays. The oxygen concentration at a given time shall be taken as the concentration registered after the time interval t_d .

10.2.1.2 If the oxygen analyser is equipped with an electric response time adjustment, set it so that at turn-off there is just a trace of overshoot when switching rapidly between two different calibration gases.

10.2.1.3 The timing of the scans by the data collection system shall be calibrated with a timer accurate to within 1 s in 1 h. The data output shall show event times correct to 3 s.

10.2.2 Operating calibrations

At the start of testing each day, zero and calibrate the oxygen analyser. For zeroing, feed the analyser with nitrogen gas, with the same flow rate and pressure as for the sample gases. Calibration shall be similarly achieved using dried ambient air and adjusting for a response of 20,95 %. Carefully monitor analyser flow rates and set to be equal to the flow rate used when testing specimens. After each specimen has been tested, ensure that a response level of 20,95 % is obtained using ambient air.

10.3 Heat release rate calibration

10.3.1 Perform heat release rate calibration at the start of testing each day. Introduce methane into the calibration burner at a flow rate corresponding to 5 kW based on the net heat of combustion of methane ($50,0 \times 10^3$ kJ/kg) using a precalibrated flowmeter. The flowmeter used may be a dry test meter, a wet test meter or an electronic mass flow controller. If an electronic mass flow controller is used, it shall be calibrated periodically against a dry test meter or a wet test meter. The test meter shall be equipped with devices to measure the temperature and pressure of the flowing gas, so that appropriate corrections to the reading can be made. If a wet test meter is used, the readings shall also be corrected for the moisture content. The exhaust fan shall be set to the speed to be used for subsequent testing. The required calculations are given in clause 12.

NOTE 11 Calibration may be performed with the cone heater operating or not, but should not be performed during heater warm-up.

10.3.2 At least once a month, perform a further calibration with a flow rate corresponding to 10 kW, using the basic procedure as described in 10.3.1. With the instrument calibrated at 5 kW, the 10 kW calibration shall give a heat release rate value of 10 kW to within ± 5 %.

10.4 Mass measuring system calibration

The load cell shall be calibrated with standard weight-pieces in the range of test specimen mass at least every day, and when the load cell mechanical zero needs to be adjusted. The load cell mechanical zero may have to be adjusted after changing orientation, due to different specimen holder tare masses.

11 Test procedure

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 for burns, ignition of extraneous objects or clothing. The operator shall use protective gloves for insertion and removal of test specimens. Neither the cone heater nor the associated fixtures shall be touched while hot except with the use of protective gloves. Care shall be taken never to touch the spark igniter which carries a substantial potential (10 kV). The exhaust system of the apparatus shall be checked for proper operation before testing and shall discharge into a building exhaust system with adequate capacity. The possibility of the violent ejection of molten hot material or sharp fragments from some kinds of specimens when irradiated cannot totally be discounted and it is therefore essential that eye protection be worn.

11.1 Initial preparation

11.1.1 Check the CO₂ trap and the final moisture trap. Replace the sorbents if necessary. Drain any accumulated water in the cold trap separation chamber. The normal operating temperature of the cold trap shall be between 0 °C and 4 °C.

NOTE 12 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, for example by introducing pure nitrogen, at the same flow-rate and pressure as for the sample gases, from a nitrogen source connected as close as possible to the ring sampler. The oxygen analyser should then read zero.

11.1.2 Turn on power to the cone heater (see annex A.4.1) and the exhaust. Power to the gas analysers, load cell and pressure transducer shall not be turned off on a daily basis.

11.1.3 Set an exhaust flow rate of $(0,024 \pm 0,002 \text{ m}^3/\text{s})$. If a different flow-rate is used, this shall be indicated in the test report.

11.1.4 Perform the required calibration procedures specified in clause 10. In the horizontal orientation, put an empty specimen holder with refractory blanket (6.5.1), in place during warm-up and between tests to avoid excessive heat transmission to the load cell.

NOTES

13 When testing in the vertical orientation, the use of an empty specimen holder is not necessary.

14 A retainer frame (figure 10) and wire grid (figure 11) should be used when testing intumescent specimens in the horizontal

orientation and can also be used to reduce unrepresentative edge burning of composite specimens and for retaining specimens prone to delamination. Other techniques may be utilized if documented in the test report. The wire grid shown in figure 11 is also suitable for the vertical orientation.

11.1.5 If external ignition is used, position the spark plug holder in the location appropriate to the orientation being used (6.5).

11.2 Procedure

11.2.1 When ready to test, if testing in the horizontal orientation, first remove the empty specimen holder (see 11.1.4).

11.2.2 Place the specimen, held in the appropriate holder (6.5), in position and start the data collection (6.13). (The holder shall be at room temperature initially.) The data collection intervals shall be 5 s or less.

11.2.3 Start the ignition timer (6.9) if external ignition is to be used. Move the spark plug into place and turn on the power.

11.2.4 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, re-insert the spark igniter and turn on the spark. If flaming re-occurs, 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.

11.2.5 Collect all data until either:

- a) 2 min after flaming or other signs of combustion cease and the average mass loss over a 1 min period has dropped below 150 g/m²; or
- b) 60 min have elapsed;

whichever occurs first. Observe and record physical changes to the sample such as melting, swelling and cracking.

11.2.6 Remove specimen and specimen holder.

11.2.7 For testing in the horizontal orientation, replace an empty specimen holder.

11.2.8 If the specimen does not ignite in 10 min, remove and discard, unless the specimen is showing signs of heat evolution.

11.2.9 Three specimens shall be tested and the results reported as described in clause 13. The 180 s mean heat release rate readings shall be compared for the three specimens. If any of these mean readings differ by more

than 10 % from the arithmetic mean of the three readings, then a further set of three specimens shall be tested. In such cases, the arithmetic mean of the set of six readings shall be reported.

NOTE 15 The test data have limited validity if the specimen melts sufficiently to overflow the drip trough when testing in the vertical orientation or the sample holder when testing in horizontal orientation, if explosive spalling occurs, or if the specimen swells excessively and touches the spark igniter or the heater base plate.

12 Calculations

NOTE 16 The equations in this clause assume only O₂ is measured as indicated on the gas analysis system on figure 6. Appropriate equations for cases where additional gas analysis equipment (CO₂, CO and possibly H₂O) is used and CO₂ is not removed from the O₂ sampling lines can be found in annex F. In any case where CO₂ is removed from the O₂ sampling line (even when CO₂ is measured), equations (1) to (5) should be used.

12.1 Calibration constant for oxygen consumption analysis

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

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

where 10,0 corresponds to 10,0 kW methane supplied, 12,54 × 10³ is Δ*h_c* / *r_O*, for methane and 1,10 is the ratio of the molecular weights of oxygen and air.

12.2 Heat release rate

12.2.1 Prior to performing other calculations, calculate the oxygen analyser time shift, *t_d*, using the following equation:

$$x_{O_2}(t) = x_{O_2}^1(t + t_d) \quad \dots (2)$$

12.2.2 Calculate the heat release rate, *q̇(t)*, from

$$\dot{q}(t) = (\Delta h_c / r_O)(1,10) C \sqrt{\frac{\Delta p}{T_e} \frac{x_{O_2}^0 - x_{O_2}}{1,105 - 1,5x_{O_2}}} \quad \dots (3)$$

where Δ*h_c* / *r_O* for the specimen is taken as 13,1 × 10³ kJ/kg unless a more accurate value is known.

12.2.3 Heat release rate per unit area can then be obtained from

$$\dot{q}''(t) = \dot{q}(t) / A_s \quad \dots (4)$$

where *A_s* is the initially exposed area of the sample, for example, 0,008 8 m² in the vertical orientation and in the horizontal orientation when the retainer frame is used.

12.3 Exhaust duct flow rate

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

$$\dot{m}_e = C \sqrt{\frac{\Delta p}{T_e}} \quad \dots (5)$$

NOTE 17 Equations for specimen mass loss rate *ṁ* and effective heat of combustion Δ*h_{c,eff}* are given in annex C.

13 Test report

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

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

- name and address of test laboratory;
- name and address of sponsor;
- name and address of manufacturer/supplier;
- date of the test;
- operator;
- tradename and specimen identification code or number;
- composition or generic identification;
- specimen thickness²⁾, expressed in millimetres, and mass²⁾, expressed in grams. With composites and assemblies a nominal thickness and density of each of the components shall be given, together with the apparent (overall) density of the whole;
- colour of the specimens;
- details of specimen preparation by the testing laboratory;

2) Report these items for each specimen.

k) test orientation, specimen mounting, face tested and whether the retainer frame, the wire grid, or other special mounting procedures were used;

l) irradiance³⁾, expressed in kilowatts per square metre, and exhaust system flow rate³⁾, expressed in cubic metres per second;

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

NOTE 18 This should be a minimum of three, except for exploratory testing.

n) time to sustained flaming³⁾, expressed in seconds;

o) test duration³⁾, i.e. the time between the start of the test and the end according to 11.2.5, expressed in seconds;

p) heat release rate (per unit area), expressed in kilowatts per square metre, represented as a curve³⁾, recorded for the entire test;

q) average \dot{q}'' value³⁾ for the first 180 s (\dot{q}''_{180}) and 300 s (\dot{q}''_{300}) after ignition, or for other appropriate periods and peak³⁾ \dot{q}'' (\dot{q}''_{\max}), values, expressed in kilowatts per square metre.

For specimens which do not show sustained flaming, report the above quantities tabulated for periods beginning with the next reading after the last negative rate of heat release reading at the beginning of the test.

Certain specimens do not show visible, sustained flaming, but do indicate non-zero rate of heat release values. There will be negative readings, in general, since before the specimen starts burning the output is $0 \pm$ noise. Average rate of heat release values have to be calculated using the trapezium rule for integration.

For example, with a 5 s data collection interval, \dot{q}''_{180} is obtained as follows:

1) sum the rate of heat release values for the 2nd scan to the 36th scan after ignition, or to the last negative value. If the test is completed before the 180 s period has elapsed, use the test average instead;

2) add half of the rate of heat release measured at the first scan and at the 37th scan after ignition or after the last negative value;

3) multiply the sum obtained in step 2 by the scan interval and divide it by 180;

r) total heat released by the specimen³⁾, expressed in megajoules per square metre. The total heat shall be computed beginning at the next reading after the last negative rate of heat release reading occurred at the beginning of the test, and continuing until the final reading recorded for the test.

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

s) average $\Delta h_{c,eff}$ for entire test³⁾, expressed in megajoules per kilogram;

t) curve of $\Delta h_{c,eff}$ (optional)³⁾;

u) mass remaining after test³⁾, expressed in grams, and percentage of the total mass pyrolysed³⁾;

v) sample mass loss³⁾, expressed in kilograms per square metre, and the average rate of specimen mass loss per unit area, expressed in grams per square metre second ($g/m^2 \cdot s$), computed over the period between ignition and the end of the test;

w) values determined in items n), o), s), q), r) and v), averaged for all replicates;

x) additional observations, such as transitory flaming or flashing³⁾;

y) difficulties encountered in testing, if any³⁾;

NOTE 19 A suggested summary test report is given in annex D.

3) Report these items for each specimen.

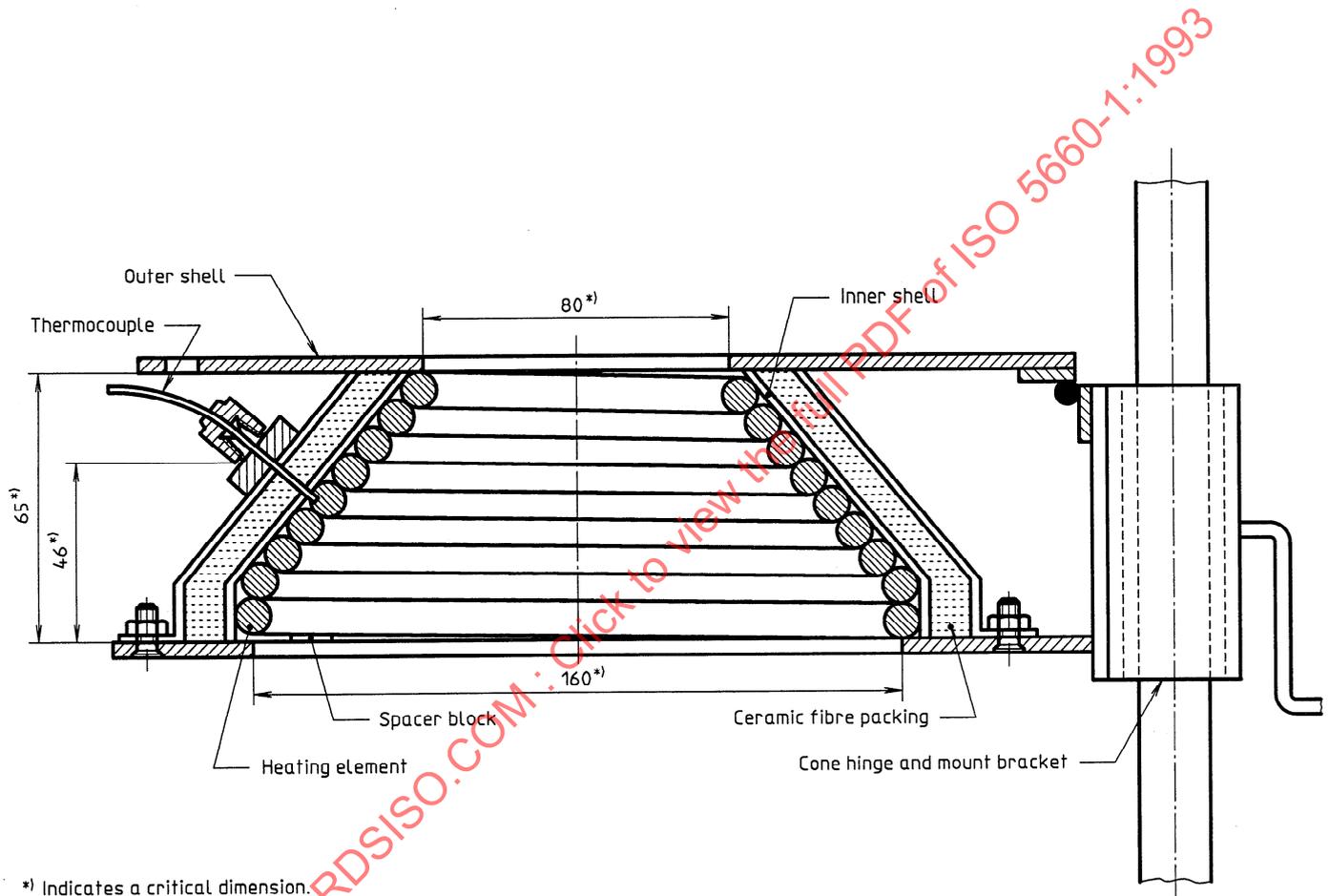
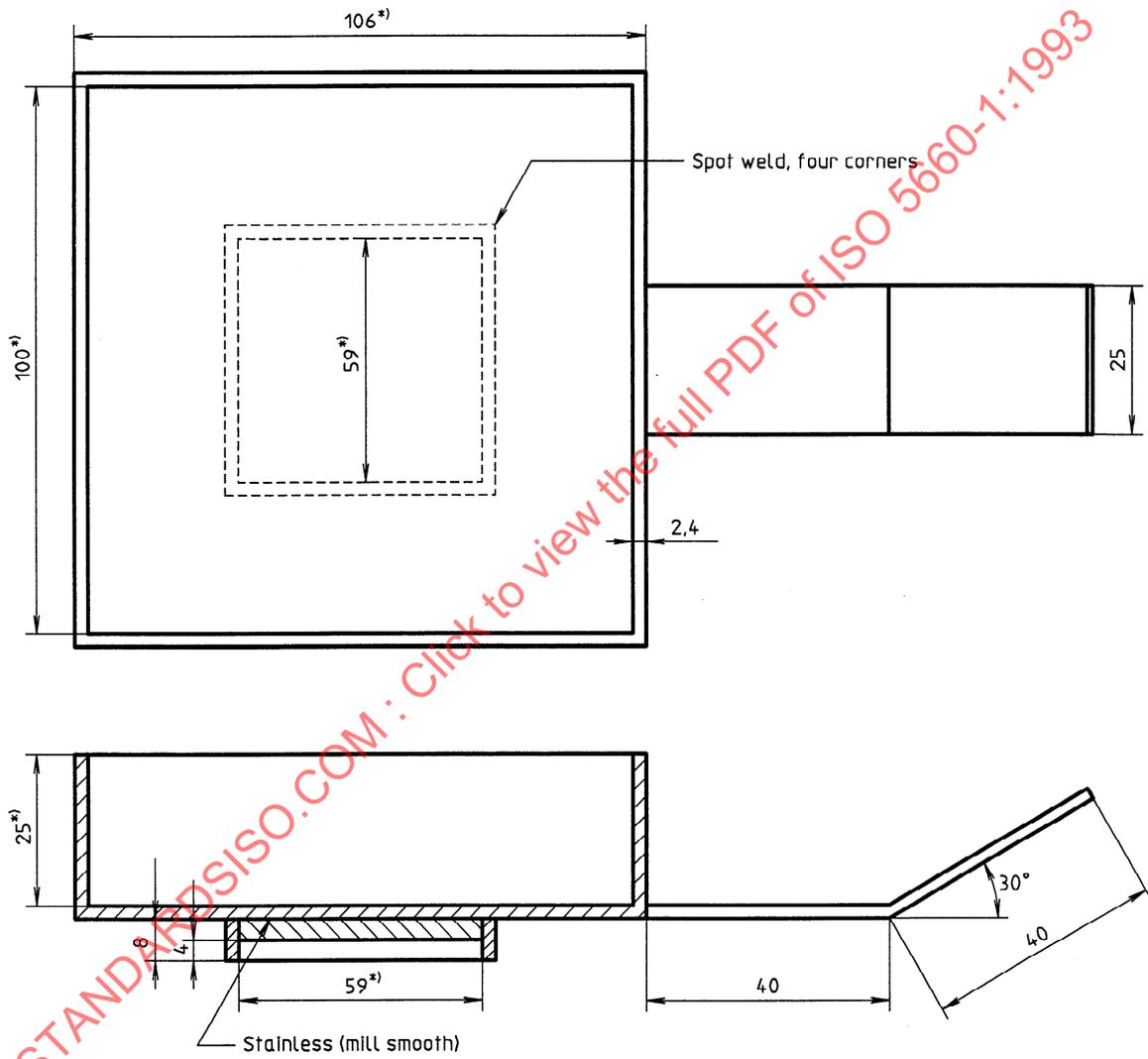
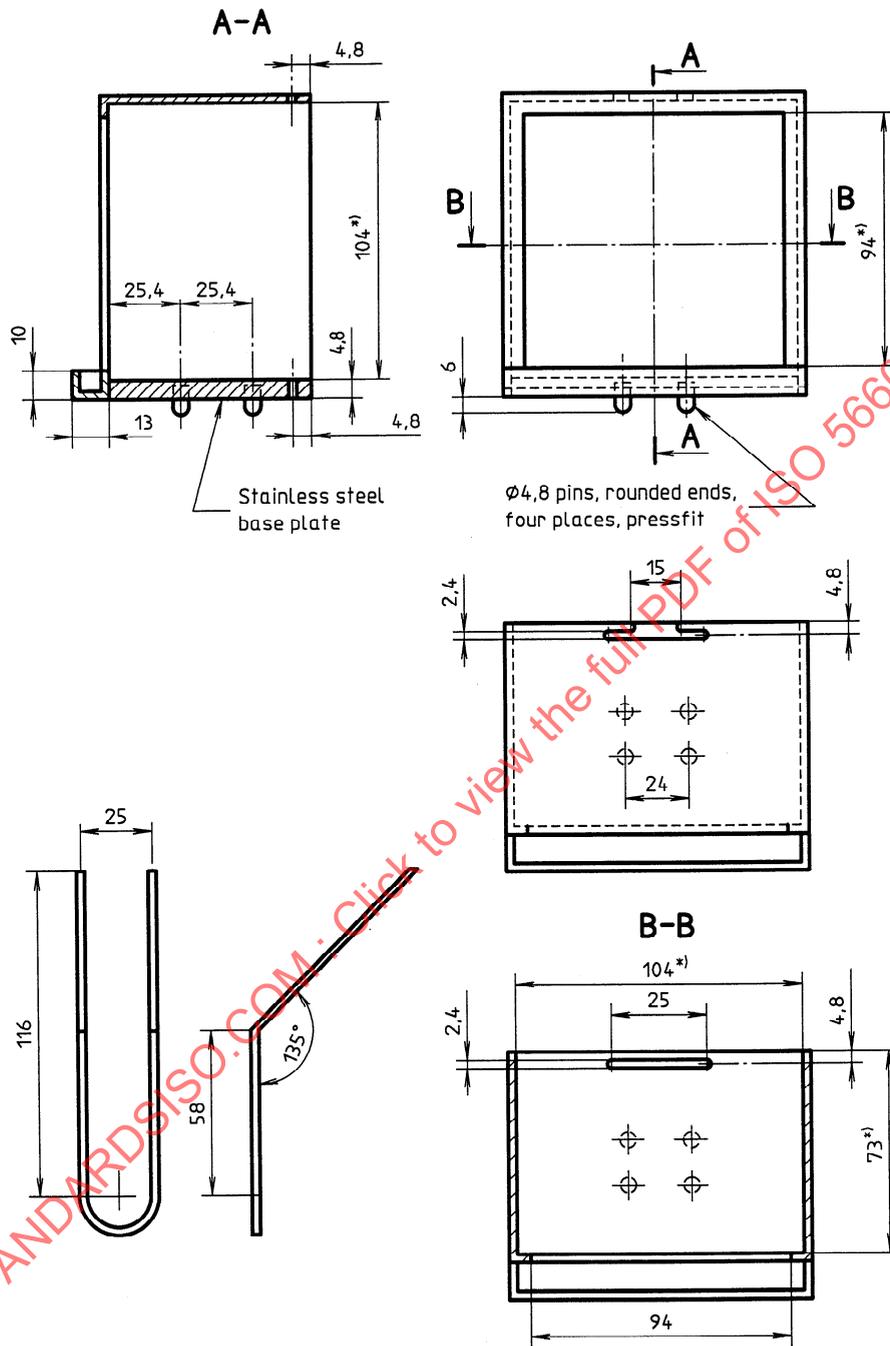


Figure 1 – Cross-sectional view through the heater



* Indicates a critical dimension.

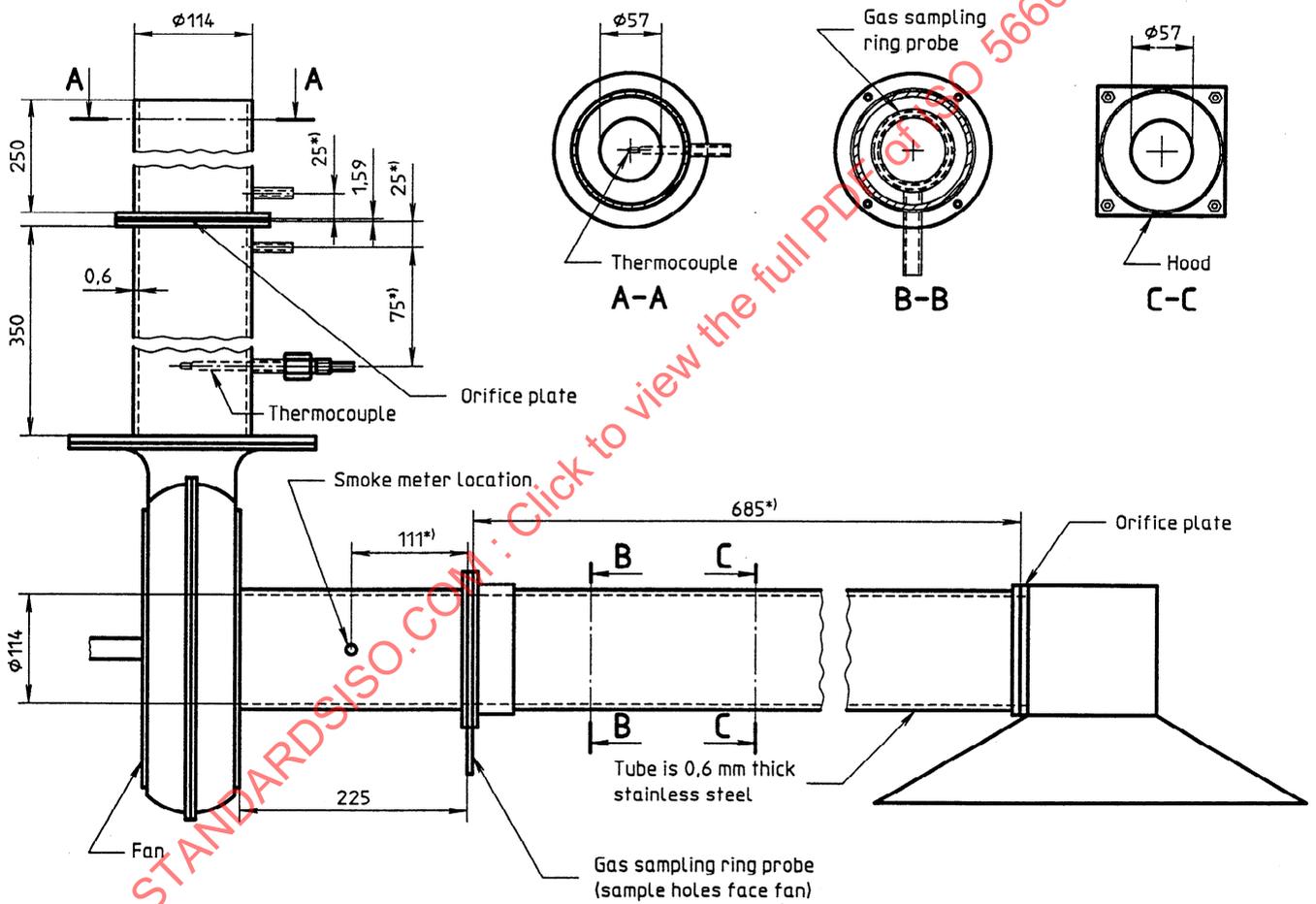
Figure 2 – Horizontal specimen holder



*) Indicates a critical dimension.

NOTE – Material: 1,59 mm thick stainless steel (except base plate).

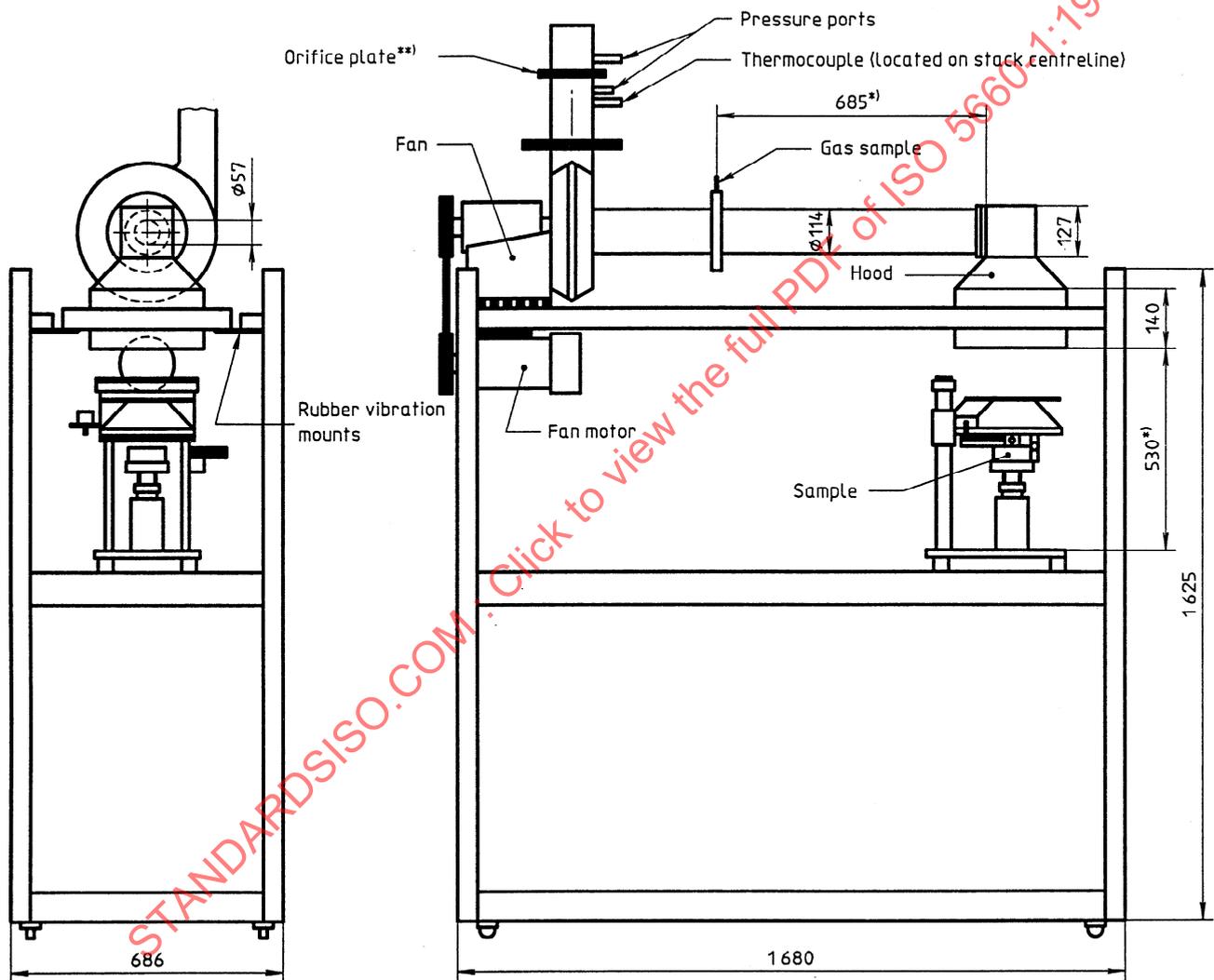
Figure 3 – Vertical specimen holder



*³) Indicates a critical dimension.

NOTE – Use an alignment rod when welding tubes to duct to ensure perfect alignment.

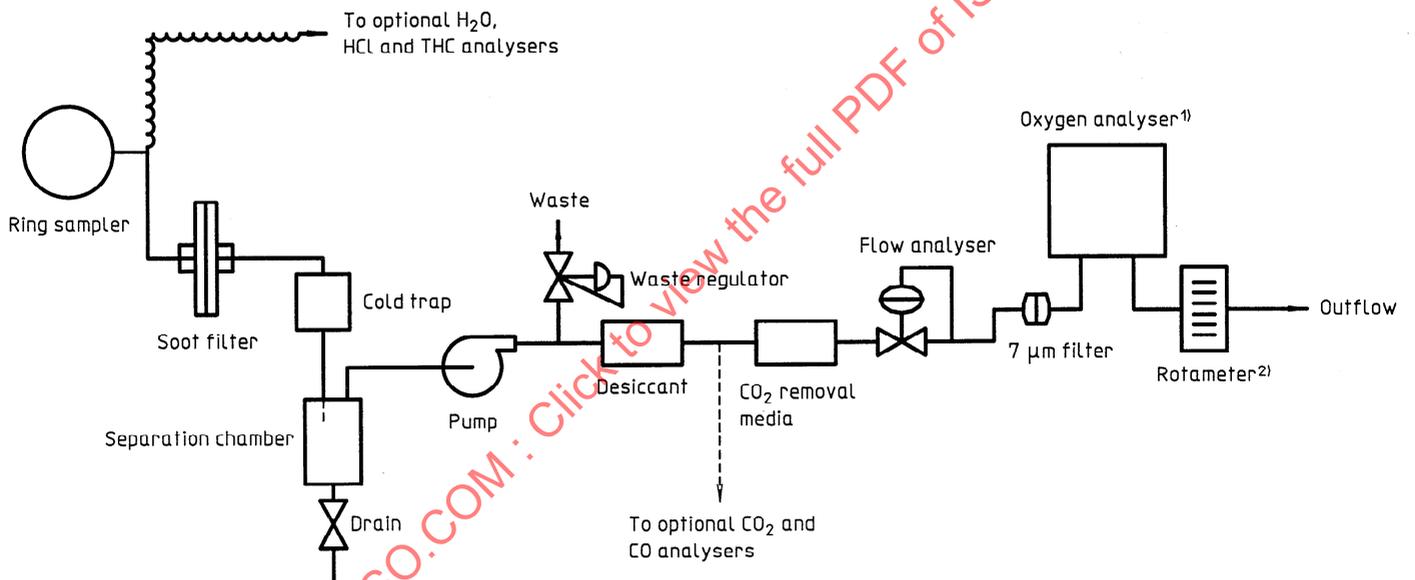
Figure 4 – Exhaust system (not to scale)



*) Indicates a critical dimension.

***) Orifice size is half the internal diameter of the stack.

Figure 5 – Overall view of apparatus



¹⁾ To include absolute pressure transducer.

²⁾ Rotameter is attached to outlet of oxygen analyser.

Figure 6 – Gas analyser instrumentation

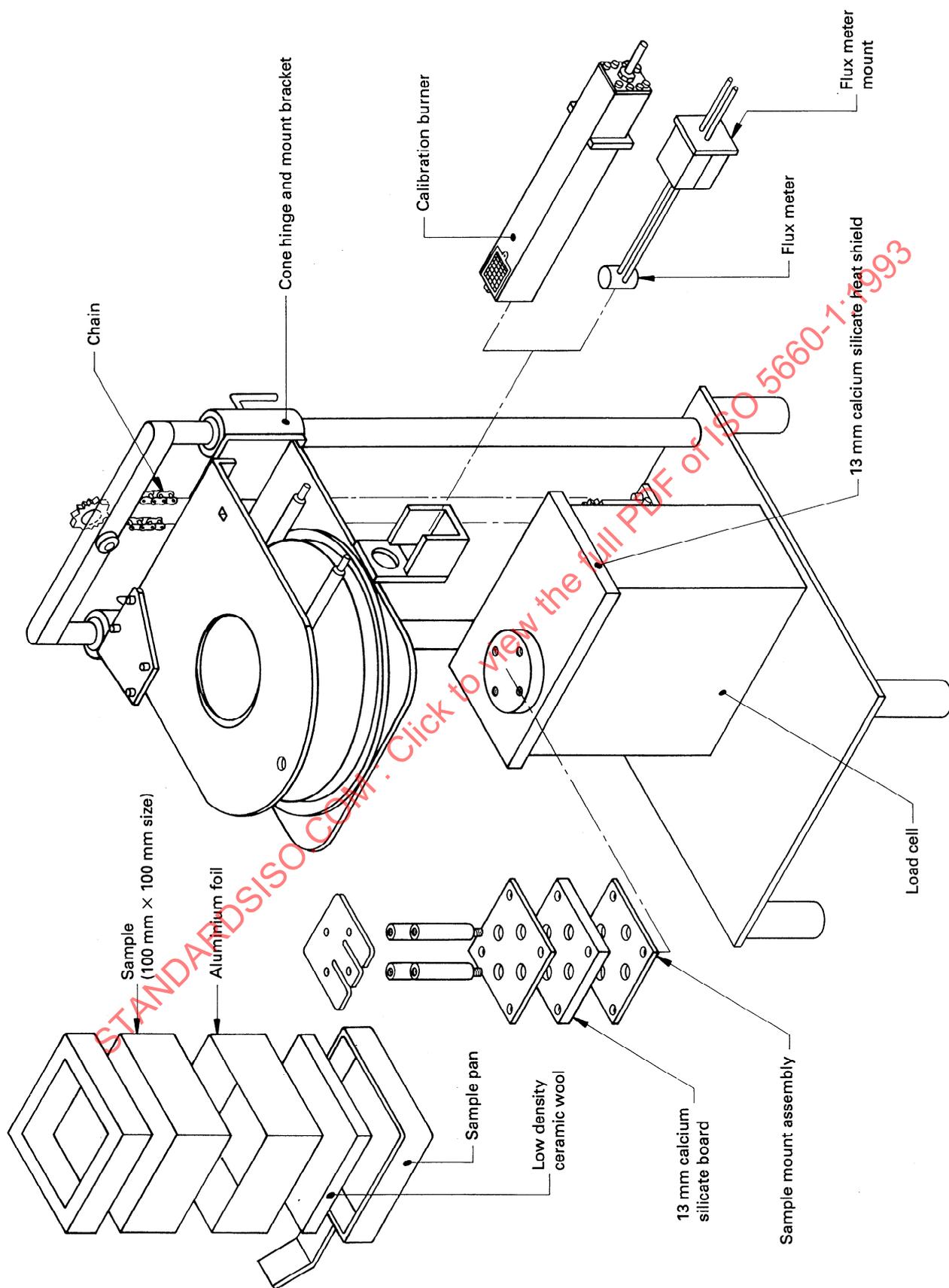


Figure 7 – Exploded view, horizontal orientation

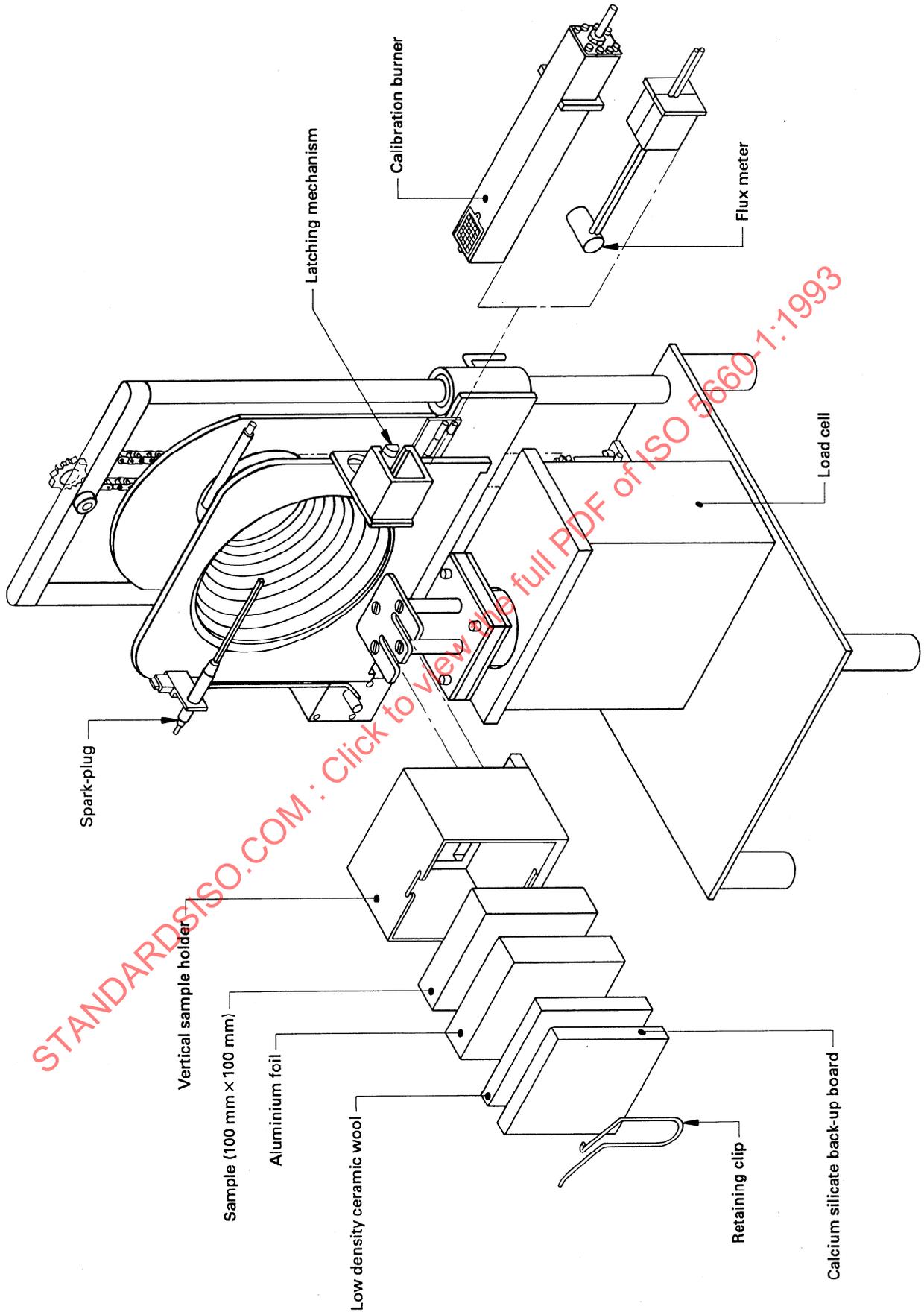


Figure 8 – Exploded view, vertical orientation

Dimensions in millimetres (unless otherwise specified)

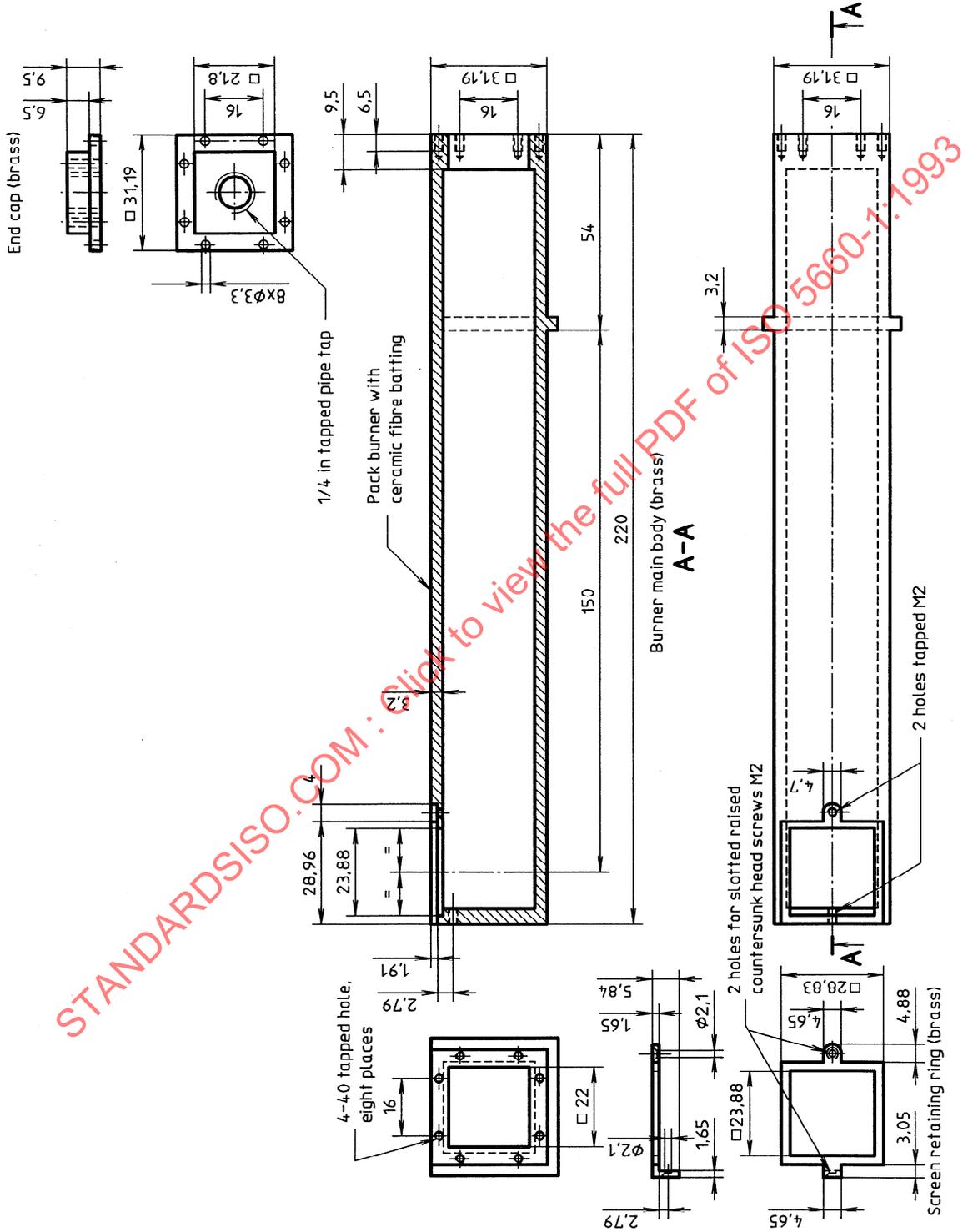
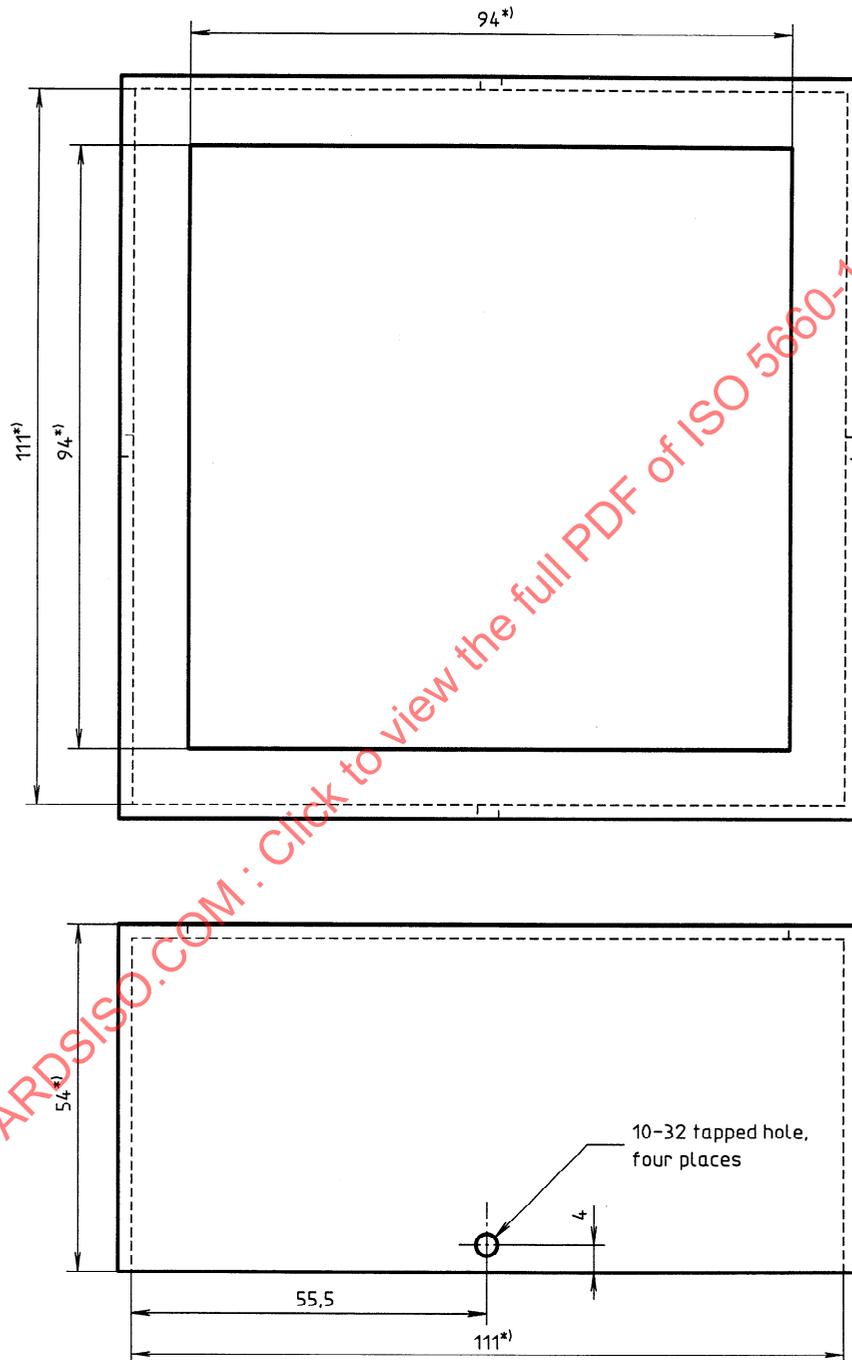


Figure 9 – Calibration burner

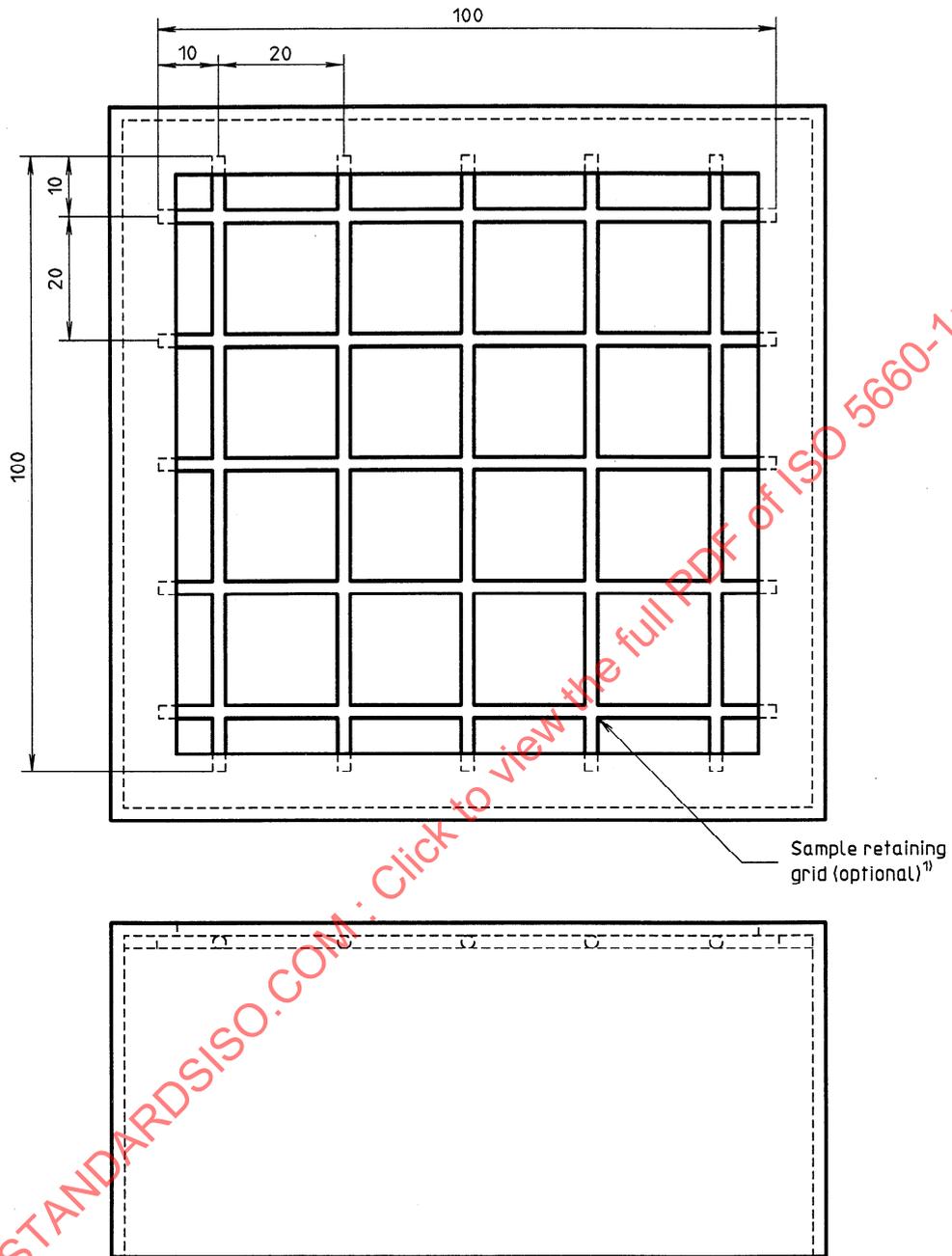
Dimensions in millimetres



*) Indicates a critical dimension.

NOTE – Material: 1,9 mm thick stainless steel.

Figure 10 – Optional retainer frame



¹⁾ For use with samples that are expected to intumesce.
Material: $\varnothing 2$ stainless steel rod. Weld all intersections.

NOTE – Material: 1,9 mm thick stainless steel, unless otherwise specified.

Figure 11 – Optional retaining grid

Annex A (informative)

Commentary and guidance notes for operators

A.1 Introduction

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

A.2 Rate of heat release measurements

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

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

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

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

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

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

A.3 Choice of operating principle

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

A.3.2 It is difficult to measure heat directly without some loss. However, it is simple to contain the total products of combustion without loss and to measure the oxygen concentration in that stream. Heat release can be computed from such measurements using the oxygen consumption principle. This principle states that for most common combustibles an amount of heat equal to 13,1 × 10³ kJ is released for each kilogram of oxygen consumed from the air stream. This quantity varies by about ± 5 % for most common combustibles. This principle forms the basis of the test method detailed in this International Standard. The method remains useful even if a significant fraction of the products become CO or soot rather than CO₂; in these cases, correction factors can be applied.

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

A.4 Heater design

A.4.1 Experience with various rate-of-heat-release measurement techniques suggests that for minimal errors in irradiance, the specimen should see only either a

thermostatically controlled heater, a water-cooled plate or open air. Nearby solid surfaces, if they are not temperature-controlled, can rise in temperature due to specimen flame heating and then act as further sources of radiation back to the specimen which can lead to errors. Furthermore, when oxygen consumption is used as the measurement principle, a gas-fired heater is not desirable because it can contribute a noisy baseline to oxygen readings, even though allowance can be made for its oxygen consumption.

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

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

A.5 Pilot ignition

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

A.6 Back face conditions

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

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Annex B (informative)

Resolution, precision and bias

B.1 Resolution

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

B.2 Speed of response

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

B.3 Precision

A set of inter-laboratory trials have been conducted by ISO/TC 92/SC 1/WG 5. The protocol used was functionally the same as described in this International Standard. The materials tested in these trials were 25 mm black PMMA ($\rho = 1\,180\text{ kg/m}^3$), 30 mm rigid polyurethane foam ($\rho = 33\text{ kg/m}^3$), 13 mm particle board ($\rho = 640\text{ kg/m}^3$), 3 mm hardboard ($\rho = 1\,010\text{ kg/m}^3$), 10 mm gypsum board ($\rho = 1\,110\text{ kg/m}^3$) and 10 mm fire retardant treated particle board ($\rho = 750\text{ kg/m}^3$). Three replicates of each material were tested in two orientations (horizontal and vertical) and at two irradiance levels (25 kW/m² and 50 kW/m²) by six to eight laboratories.

Data from the ISO trials were supplemented by data developed during an analogous set of trials conducted by ASTM E05 SC21 TG 60, again using functionally the same protocol and identical irradiance levels, orientations and number of replicates. Since the findings for repeatability, r

and reproducibility R in the ASTM trials showed generally similar trends, the data were analysed as a combined data set. ASTM data were excluded in the one instance (i.e., for \dot{q}_{180}'') where instructions to the laboratories differed. Six laboratories tested the following materials: 6 mm fire-retardant treated ABS ($\rho = 325\text{ kg/m}^3$), 12 mm particle board⁴⁾ ($\rho = 640\text{ kg/m}^3$), 6 mm black PMMA⁵⁾ ($\rho = 1\,180\text{ kg/m}^3$), 6 mm polyethylene ($\rho = 800\text{ kg/m}^3$), 6 mm PVC ($\rho = 1\,340\text{ kg/m}^3$), and 25 mm rigid polyisocyanurate foam ($\rho = 280\text{ kg/m}^3$).

Values for repeatability r and reproducibility R at the 95 % confidence level were calculated for the complete data set in accordance with ISO 5725 for five variables. Such values for r and R are equal to 2,8 times the appropriate standard deviation and include results identified as "stragglers". The variables were chosen as being representative for the test results: t_{ig} , \dot{q}_{max}'' , \dot{q}_{180}'' , q_{tot} and $\Delta h_{c,eff}$. A linear regression model [equation II in ISO 5725:1986, 15.2 b)] was used to describe r and R as a function of the mean over all replicates and over all laboratories of each of the five aforementioned variables. The regression equations are given below. The range of mean values over which the fit was obtained is also indicated.

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

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

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

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

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

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

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

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

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

4) This was the same material as tested by the ISO group.

5) This was the same material as tested by the ISO group but in a different thickness.

The results for q_{tot}'' in the range of 5 MJ/m² to 720 MJ/m² were

$$r = 7,4 + 0,068 q_{\text{tot}}'' \quad \dots \text{ (B.7)}$$

$$R = 11,8 + 0,088 q_{\text{tot}}'' \quad \dots \text{ (B.8)}$$

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

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

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

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

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

Then the probability is 95 % that the result of the second test will fall between 83 s and 117 s. Suppose now that the

same material is tested by a different laboratory. The value of R is evaluated as

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

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

B.4 Bias

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

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