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**Reaction-to-fire tests — Determination of  
fire and thermal parameters of materials,  
products and assemblies using an  
intermediate-scale calorimeter (ICAL)**

*Essais de réaction au feu — Détermination, à l'aide d'un calorimètre à  
échelle intermédiaire (ICAL), des paramètres thermiques et relatifs au  
feu des matériaux, produits et ouvrages*



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## Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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

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.

ISO 14696 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

This first edition cancels and replaces ISO/TR 14696:1999, which has been technically revised.



# Reaction-to-fire tests — Determination of fire and thermal parameters of materials, products and assemblies using an intermediate-scale calorimeter (ICAL)

## 1 Scope

This International Standard provides a method for measuring the response of materials, products and assemblies exposed in vertical orientation to controlled levels of radiant heating with a piloted ignition source.

This test method is used to determine the ignitability, heat release rates, mass loss rates and visible smoke development of materials, products and assemblies under well-ventilated conditions.

The heat release rate is ascertained by measurement of the oxygen consumption as determined by the oxygen concentration and flow in the exhaust product stream as specified in 5.5.8. Smoke development is quantified by measuring the obscuration of light by the combustion product stream.

Specimens are exposed to heating fluxes ranging from 0 kW/m<sup>2</sup> to 50 kW/m<sup>2</sup>. Hot wires are used as the ignition source.

This test method has been developed for material, product or assembly evaluations, mathematical modelling and design purposes. The specimen shall be tested in thicknesses and configurations representative of actual end product or system uses.

The test method in this International Standard is based on the apparatus described in ASTM E1623 [13].

## 2 Normative references

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

ISO 9705, *Fire tests — Full-scale room test for surface products*

ISO 13943: 2000, *Fire safety — Vocabulary*

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

ISO 24473, *Fire tests — Open calorimetry — Measurement of the rate of production of heat and combustion products for fires of up to 40 MW*

### 3 Terms, definitions, symbols and units

#### 3.1 Terms and definitions

For the purposes of this document, the definitions given in ISO 13943 and the following apply.

##### 3.1.1

##### **composite**

combination of materials which are generally recognized in building construction as discrete entities

EXAMPLE Coated or laminated materials.

##### 3.1.2

##### **flashing**

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

##### 3.1.3

##### **heating flux**

incident flux imposed externally from the heater on the specimen at the initiation of the test

##### 3.1.4

##### **heat release rate**

heat evolved from the specimen, per unit of time

##### 3.1.5

##### **ignition**

onset of sustained flaming as defined in 3.1.13

##### 3.1.6

##### **irradiance**

⟨at a point on a surface⟩ the density of radiant flux incident on a surface

##### 3.1.7

##### **material**

single substance or uniformly dispersed mixture, for example metal, stone, timber, concrete, mineral fibre, polymers

##### 3.1.8

##### **orientation**

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

NOTE The orientation of the specimen in this International Standard is vertical and there are no provisions for testing horizontal specimens.

##### 3.1.9

##### **oxygen consumption principle**

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

##### 3.1.10

##### **product**

material, composite or assembly, about which information developed by this test method is required

##### 3.1.11

##### **specimen**

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



**3.1.12****smoke obscuration**

reduction of light transmission by smoke, as measured by light attenuation

**3.1.13****sustained flaming**

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

**3.1.14****transitory flaming**

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

**3.2 Symbols and units**

The symbols and units are the following.

Symbol	Term	Unit
$A$	Cross-sectional area of exhaust duct	$\text{m}^2$
$A_s$	Exposed specimen area	$\text{m}^2$
$E$	Net heat released for complete combustion, per unit of oxygen consumed	13,1 MJ/kg $\text{O}_2$
$E_{\text{CO}}$	Net heat released per unit mass of oxygen consumed for combustion of CO to $\text{CO}_2$	17,6 MJ/kg $\text{O}_2$
$E_{\text{propane}}$	Net heat released for complete combustion of propane, per unit of oxygen consumed	12,78 MJ/kg $\text{O}_2$
$E_{\text{methane}}$	Net heat released for complete combustion of methane, per unit of oxygen consumed	12,51 MJ/kg $\text{O}_2$
$F_{\text{OD}}$	Relative optical density	dimensionless
$f_x$	Yield of gas x	kg/kg
$f_{(\text{Re})}$	Reynolds number correction for bi-directional probe differential pressure measurement	—
$\Delta H_{\text{c,ng}}$	Net heat released per unit mass of natural gas	MJ/kg
$I$	Intensity of transmitted light beam	cd
$I_0$	Intensity of light beam before attenuation	cd
$k$	Smoke extinction coefficient	$\text{m}^{-1}$
$k_c$	Exhaust duct flow velocity profile shape factor	dimensionless
$L_p$	Path length of light	m
$M_a$	Relative molecular mass of incoming air	kg/kmol
$M_{\text{CO}}$	Relative molecular mass of carbon monoxide	28 kg/kmol
$M_{\text{CO}_2}$	Relative molecular mass of carbon dioxide	44 kg/kmol
$M_{\text{dry}}$	Relative molecular mass of dry air	29 kg/kmol
$M_e$	Relative molecular mass of exhaust gases	kg/kmol
$M_{\text{H}_2\text{O}}$	Relative molecular mass of water	18 kg/kmol
$M_{\text{N}_2}$	Relative molecular mass of nitrogen	28 kg/kmol

$M_{O_2}$	Relative molecular mass of oxygen	32 kg/kmol
$m$	Specimen mass	kg
$\dot{m}_e$	Mass flow in exhaust duct	kg/s
$\dot{m}_{ng}$	Mass flow of natural gas to the radiant panel	kg/s
$\Delta p$	Pressure drop across the orifice plate or bi-directional probe	Pa
$\dot{q}$	Heat release rate	kW
$\dot{q}''_{A,60}$	Average heat release rate per unit area of specimen for the first 60 s after ignition	kW/m <sup>2</sup>
$\dot{q}''_{A,180}$	Average heat release rate per unit area of specimen for the first 180 s after ignition	kW/m <sup>2</sup>
$\dot{q}''_{peak}$	Peak heat release rate per unit area of specimen	kW/m <sup>2</sup>
$q''_s$	Total heat released per unit area of specimen	MJ/m <sup>2</sup>
$\dot{q}''_s$	Heat release rate per unit area of specimen	kW/m <sup>2</sup>
$q''_{s,i}$	Heat released per unit area of specimen, in incoming air	MJ/m <sup>2</sup>
$R_{inst}$	Instantaneous rate of production of light-obscuring smoke	m <sup>2</sup> /s
$R_{tot}$	Total amount of smoke	m <sup>2</sup>
$T_e$	Combustion gas temperature at the bi-directional probe or orifice plate	K
$T_s$	Combustion gas temperature near the smoke meter	K
$t$	Time	s
$t_{ig}$	Time to ignition	s
$\dot{V}_e$	Volumetric flow in exhaust duct (at measuring location of mass flow)	m <sup>3</sup> /s
$\dot{V}_s$	Volumetric flow at location of smoke meter (value adjusted for smoke measurement calculations)	m <sup>3</sup> /s
$\Delta t$	Sampling time interval	s
$X_{CO,e}$	Measured mole fraction of CO in exhaust flow	dimensionless
$X_{CO,i}$	Measured mole fraction of CO in incoming air	dimensionless
$X_{CO_2,e}$	Measured mole fraction of CO <sub>2</sub> in exhaust flow	dimensionless
$X_{CO_2,i}$	Measured mole fraction of CO <sub>2</sub> in incoming air	dimensionless
$X_{O_2,e}$	Measured mole fraction of O <sub>2</sub> in exhaust flow	dimensionless
$X_{O_2,i}$	Measured mole fraction of O <sub>2</sub> in incoming air	dimensionless
[x]	Relative mass fraction of gas x	kg/kg
$\alpha$	Combustion expansion factor (an average value of 1,105 is used for mixed fuels or when the exact factor is unknown)	dimensionless
$\rho$	Density of air at the temperature in exhaust duct	kg/m <sup>3</sup>
$\rho_0$	Density of air at 273,15 K: 1,293	kg/m <sup>3</sup>
$\phi$	Oxygen depletion factor	dimensionless

## 4 Principle

**4.1** This test method is designed to measure the heat release rate from a 1 m<sup>2</sup> specimen in a vertical orientation. The specimen is exposed to a uniform and constant heat flux from a gas fired radiant panel up to 50 kW/m<sup>2</sup> and electrically heated wires are used for piloted ignition. Heat release measured using this test method is based on the observation that, generally, the net heat released during combustion is directly related to the amount of oxygen required for combustion <sup>[1], [2]</sup>. The primary measurements are oxygen concentration and exhaust flow rate. Burning may be either with or without ignition wires used at the top and bottom of the specimen.

NOTE The addition of carbon monoxide and carbon dioxide concentration measurements can improve the accuracy of the heat release rate measurement, and can also be used to provide species generation rates of both gases.

**4.2** Additional measurements include the mass of the specimen, which can be used to determine the mass loss rate, the time to sustained flaming and the light intensity of a light beam having traversed the smoky duct, which can be used to determine the smoke-specific extinction area, the relative optical density and the smoke release rate. The apparatus can be used to develop data relative to the other parameters discussed in Annex F.

## 5 Apparatus

### 5.1 General

Dimensions shall have a tolerance of  $\pm 5$  mm on the radiant panel and specimen holder assemblies. An exception to this tolerance is the placement of the screen in front of the ceramic burner which shall be  $\pm 0,5$  mm. The tolerances permitted in the exhaust system of ISO 9705 are permissible.

The apparatus shall consist of the following components.

**5.1.1 Radiant panel assembly**, in a vertical orientation, see Figure 1.

**5.1.2 Radiant panel constant irradiance controller**, capable of being held at a preset level by means of regulating the flow of natural gas to the burners during a test.

**5.1.3 Water-cooled heat shield**, capable of absorbing the thermal energy from the radiant panels.

**5.1.4 Specimen holder**, capable of holding a specimen up to 150 mm thick, see Figure 2.

**5.1.5 Weighing platform**, of a range of 150 kg, capable of weighing the specimen to an accuracy of at least 1 g.

**5.1.6 Exhaust collection system**, consisting of an extraction fan, steel hood, duct, bi-directional probe or orifice plate, thermocouple(s), smoke obscuration measurement system and combustion gas sampling and analysis system.

**5.1.7 Gas flow meter**, capable of measuring gas flow.

**5.1.8 Data acquisition system**, of a category equal to or better than that required in ISO 9705.

A general layout of the whole test apparatus assembly is shown in Figure 17.

### 5.2 Radiant panel

The panel (5.1.1) consists of a support frame, which supports three rows of adjustable, ceramic-faced, natural gas burners and natural gas distribution plumbing (see Figure 1).

Hollow 50 mm × 50 mm square steel tubing or galvanized 41,3 mm × 41,3 mm × 2,7 mm “C” channel can be used for the support frame application <sup>1)</sup>.

Each row comprises 10 burners 385 mm tall and 172 mm wide, fastened next to each other with a 1 mm to 2 mm air gap between them. Each burner consists of four vertically stacked perforated ceramic elements 12,7 mm deep times 95 mm high times 158 mm wide, encased in a steel sheet metal can forming a plenum space on the back of the ceramic elements. Natural gas is injected at a controlled rate by the burner's control system through a round 51,2 mm diameter opening (injection port) at the bottom of the can. Combustion air is aspirated into the plenum space through the gas and air injection port.

The face of each burner is covered with stainless steel 330 floating screens for higher surface temperature and safety. The screens shall be carefully installed to allow for thermal expansion. This prevents screen deformation and allows the distance between the burners and screens to remain constant when heated. The optimum distance between the surface of the burners and the outer surface of the screen is 20 mm. The rows of gas burners on the panel shall be vertically separated by a distance of 110 mm from each other and also attached to the support frame at the locations indicated in Figure 1. The space between the rows shall be filled with lightweight ceramic boards installed flush with the front burner surface and extending the entire width of the radiant panel. A 110 mm tall lightweight ceramic board shall be installed underneath the entire bottom burner row. This ceramic board shall also be flush with the front surface of the burners and extend the entire width of the panel. A 33 mm wide gap shall be left in the centre of the ceramic board between the top and the middle burner rows to allow the use of an infrared (IR) pyrometer.

Natural gas with a net heating value of at least 48 MJ/kg shall be supplied to the unit through a control system provided with a safety interlock. All gas pipe connections to the burners shall be sealed with a gas pipe compound resistant to liquefied petroleum gases. A drip leg shall be installed in the gas supply line going to the radiant panel to minimize the possibility of any loose scale or dirt within the gas supply line from entering the burner's control system. An approved flexible hose or fixed piping is used to supply natural gas to the radiant panel constant irradiance controller (5.1.2). Fixed piping shall be provided from the controller to individual burners. Each row of the burners is fed by a nominal 25 mm diameter horizontal steel pipe branching from a vertical nominal 32 mm diameter steel pipe located on one side of the back of the radiant panel. At each burner, a nominal 6 mm diameter pipe branches from the horizontal pipe to feed each burner. Each burner-feeding pipe includes a shut-off, a fine regulating needle valve and a nozzle directed into the injection port opening perpendicularly to the plane of the opening. The hose or piping as well as other gas line components should be capable of delivering a quantity of gas corresponding to a heating power of 400 kW.

Ignition of the burners shall be accomplished manually or by an automatic safety system. A recommended safety system designed to prevent accidental release of unburned natural gas is described in E.6.

### **5.3 Radiant panel constant irradiance controller**

The irradiance from the radiant panel assembly (5.1.1) shall be capable of being held at a preset level by means of regulating the flow of natural gas to the burners during a test (see E.2 for more information). The flow of the gas is regulated using an automatic flow controller, a motorized valve and a thermocouple located on the surface of a ceramic burner. The thermocouple shall be attached by ceramic cement on the exposed surface of the burner top ceramic element located in the fourth burner (from either end) of the middle row of the radiant panel. The irradiance is directly proportional to the temperature on the surface of the ceramic burners. Gas flow shall be continuously measured to calculate the heat released from the radiant panel assembly. This value is necessary in computations of the heat release rate from the specimen.

A laminar flow element was found to be suitable for the gas flow measurement. If a laminar element is used the natural gas temperature measurement is necessary at the location of the laminar flow element in order to calculate the flow. In order to calculate accurately the heat released from the radiant panel assembly (5.1.1), it is necessary to account for any variation of the properties of natural gas (net heating value, net heat released per unit mass of oxygen, expansion coefficient and density) by location and over time. It cannot be assumed

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1) A modified MODINE high intensity burner unit, from Modine Manufacturing Company, 1500 DeKoven Avenue, Racine Wisconsin 53403, USA, is an example of a suitable product available commercially. This information is given for the convenience of users of ISO 14696 and does not constitute an endorsement by ISO of this product.

that these values are the same as the values for pure methane. In order to provide a set of appropriate values, it is necessary to determine these properties over time based on the concentrations of the gas constituents and their variability.

## 5.4 Specimen holder assembly components

### 5.4.1 Specimen holder

The specimen holder (5.1.4) assembly is shown in Figure 2 and is capable of holding a specimen up to 150 mm thick. (A thicker specimen holder is necessary to accommodate specimens thicker than 150 mm.) The top portion of the assembly is removable to facilitate specimen insertion. Alternatively, the top portion is not removable, in which case the specimen is inserted from the back. The specimen holder shall be made as closely as possible to that shown in Figure 2 to Figure 16, to prevent bending of the holder due to non-uniform heating. If Figure 2 to Figure 16 are not followed, then the specimen holder shall be designed so that the top of the holder does not move towards or away from the radiant panel for more than 1 cm during a test.

Prior to starting the test, the specimen shall be protected from the radiant panel heat flux exposure by the water-cooled shield (5.1.3). A drip tray, shown in Figure 14, shall be attached to the legs of the specimen holder (5.1.4) directly below the specimen frame to contain limited amounts of materials that melt and drip. Two wire igniters, described in 5.5.2, are attached to the specimen holder. An air-stream-interrupting projection plate shown in Figure 16 is mounted at the bottom of the specimen (see 5.5.3).

### 5.4.2 Weighing platform

The general arrangement of the specimen holder (5.1.4) and the weighing platform (5.1.5) is indicated in Figure 2. The weighing platform shall have a range of 150 kg, shall be capable of weighing the specimen to an accuracy of at least 1 g, and shall have dimensions suitable to fit on the trolley and accommodate the sample holder.

The weighing platform shall be protected from the radiant panel irradiance by an insulation board cover as shown in Figure 2. The insulation board shall have sufficient thickness and adequate thermal properties to protect the weighing platform from the temperature increase of any of its parts by 10 °C or more during a test. A suitable protection of the weighing platform shall be demonstrated by temperature measurement on the inside of the front wall of the platform cover before the apparatus is put in operation, and after any changes of the insulation board cover. The temperature measurement shall be performed by a Type K 0,127 mm bare wire thermocouple attached to the inside of the platform wall facing the radiant panel approximately at the centre of the wall. If a calcium silicate or similar hygroscopic material is used for the insulation board cover, it shall be completely water-vapour-sealed prior to use to prevent weight loss due to water evaporation during a test. The front of the insulation board cover and the top of the specimen holder floor shall be completely covered by aluminium foil additionally to protect the weighing platform from heat radiation. The foil shall be installed with the shiny surface facing outward. The foil shall be replaced prior to a test if it becomes dirty, damaged or covered with melted material so as to no longer provide reflectance of radiant heat.

### 5.4.3 Specimen holder trolley

A trolley, as shown in Figure 17, shall be provided to hold the specimen holder (5.1.4) and weighing platform (5.1.5) so that the specimen can be moved to a predetermined location in front of the radiant panel at the beginning of a test. The trolley shall be placed on rails or guides to facilitate exact specimen placement with respect to the radiant panel. The trolley tracks shall be located perpendicular to the plane of the radiant panel so that the specimen is moved directly toward the radiant panel. The trolley tracks shall be long enough to move the specimen holder to a distance of 6 m from the radiant panel. This distance makes mounting the specimen easier.

## 5.5 Other major components

**5.5.1 Specimen heat shield**, capable of absorbing the thermal energy from the radiant panels prior to testing.

This water-cooled heat shield (5.1.3, Figure 18) can be constructed of standard steel, and shall be designed so that a preset water flow will maintain a shield temperature on the unexposed face below 100 °C. The shield shall be positioned directly in front of the radiant panel assembly (5.1.1) at a distance of 75 mm. The mounting method used shall enable the shield to be removed in less than 2 s.

**5.5.2 Wire igniters**, capable of being used as specimen pilot igniters.

Two 0,81 mm Chromel<sup>2)</sup> wires (from Type K thermocouple wires) are used as specimen pilot igniters. One wire is positioned horizontally, spanning the full width of the specimen, 80 mm above the bottom exposed edge of the specimen and 15 mm from the specimen surface. The other wire is positioned horizontally, spanning the full width of the specimen, 20 mm above the top exposed edge of the specimen and 15 mm from the specimen's vertical plane. A bracket [see Figures 15 a) and 15 b)] shall be attached to each end of each wire to compensate for the wire expansion during the test. It shall remain under tension throughout the test so that the igniter wire remains in position. When used, sufficient power shall be applied to the wires to produce an orange glow. Low voltages, between 30 volts and 35 volts, shall be used for safety reasons. More information about the choice of the wire igniters is given in E.3.

NOTE The upper wire is intended for igniting specimens that release pyrolysis gases at the top only. Examples are sandwich panels and other specimens with a non-combustible protective skin on the exposed face.

**5.5.3 Air-stream-interrupting projection plate.**

A thin steel plate which projects 10 cm out from the specimen surface shall be attached to the specimen holder (5.1.4) perpendicularly to the specimen surface along the lower exposed specimen edge (see Figure 16). Information about the air-stream-interrupting projection plate is given in E.5.

**5.5.4 Heat flux meter**, of the Schmidt-Boelter<sup>3)</sup> (thermopile) type, with a design range of 50 kW/m<sup>2</sup> to 100 kW/m<sup>2</sup>.

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 should be robust, simple to set up and use, and stable in calibration. The instrument shall have an accuracy of within ± 3 % and a repeatability of within ± 0,5 %.

**5.5.5 Heat flux calibration panel**, capable of establishing the heat flux/distance relationship.

The panel shall be constructed from nominally 12,7 mm thick lightweight ceramic fibreboard. It shall be the same size as a specimen (1 000 mm × 1 000 mm) and shall have holes with diameters to accommodate the heat flux meter from 5.5.4. Five rows and columns of holes shall be symmetrically drilled with centres 167 mm apart.

**5.5.6 Digital data collection.**

The data collection system (5.1.8) shall be equal to or better than that required in ISO 9705. Readings shall be made at intervals not exceeding 2 s.

**5.5.7 Exhaust collection system.**

**5.5.7.1** Construct the exhaust collection system (5.1.6) with the following minimum requirements: an extraction fan, steel hood, duct, bi-directional probe (see Figure 25) or orifice plate, thermocouple(s), smoke obscuration measurement system (white light lamp and photocell/detector or laser) and combustion gas

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2) Chromel and Alumel are suitable products available commercially. This information is given for the convenience of users of ISO 14696 and does not constitute an endorsement by ISO of this product.

3) This is an example of a suitable product available commercially. This information is given for the convenience of users of ISO 14696 and does not constitute an endorsement by ISO of this product.

sampling and analysis system. An example of the exhaust collection system is shown in Figure 19 and explained in Annex A. General rules of ISO 24473 shall be followed if the exhaust system differs from the one shown in Figure 19. However, the flow through the exhaust system shall not be larger than 2,5 m<sup>3</sup>/s to avoid noisy measurements.

**5.5.7.2** Ensure that the system for collecting the combustion has sufficient exhaust capacity and is designed in such a way that all of the combustion products leaving the burning specimen plus the radiant panel burning products are collected. Design the capacity of the evacuation system such that it will exhaust minimally all combustion gases leaving the specimen (see A.1 and A.6).

**5.5.7.3** Place probes for the sampling of combustion gas and for the measurement of flow in accordance with 5.5.8.

**5.5.7.4** Make all measurements of smoke obscuration, gas concentrations and flows at a position in the exhaust duct where the exhaust is uniformly mixed so that there is a nearly uniform velocity across the duct section.

**5.5.7.5** If the length of the straight section before the measurement system is at least eight times the inside diameter of the duct, the exhaust is considered to be uniformly mixed. There should also be a straight section of duct after the measurement section of at least five duct diameters. If the straight section before the measurement section is less than ten times the inside diameter of the duct, or less than five times the inside duct diameter after the measurement section, demonstrate the achievement of equivalent measurement results.

## **5.5.8 Instrumentation in exhaust duct.**

### **5.5.8.1 General**

The following specifications are minimum requirements for exhaust duct instrumentation. Additional information is provided in Annex B.

### **5.5.8.2 Flow**

Measure the flow in the exhaust duct by means of a bi-directional probe (see 5.1.6, 5.5.7.1 and Figure 25) or an equivalent system of measurement with an accuracy of at least  $\pm 5\%$  (see Annex B). The response time to a stepwise change of the duct flow shall not exceed 5 s, to reach 90 % of the final value.

### **5.5.8.3 Combustion gas analysis**

#### **5.5.8.3.1 Sampling line**

Construct the sampling line tubes of a material not influencing the concentration of the combustion gas species to be analysed. The following sequence of the gas train has been shown to be acceptable: sampling probe (see Figure 23, Figure 26, Figure 27 and Figure 28), soot filter, cold trap, gas stream pump, waste vent regulator valve, moisture and carbon dioxide removal columns (if used), flow controller, instrument filter and gas analysers (see Figure 20 and Annex B). Alternative designs of the sampling line shall give equivalent results to those obtained with the above described gas train. The gas train shall also include appropriate spanning and zeroing facilities.

NOTE 1 Granular drierite and granular ascerite<sup>4)</sup> have been found useful for moisture removal and carbon dioxide removal, respectively.

NOTE 2 The use of ascerite to remove carbon dioxide produces moisture and therefore a second dessicant column should be used downstream to remove this additional moisture.

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4) Drierite and ascerite are suitable products available commercially. This information is given for the convenience of users of ISO 14696 and does not constitute an endorsement by ISO of this product.

#### 5.5.8.3.2 Oxygen measurement

The oxygen analyser shall be of the paramagnetic type and capable of measuring at least a range of 16 % to 21 % oxygen with an accuracy of at least  $\pm 0,01$  percent volume fraction<sup>5)</sup> of oxygen, in order to have adequate measurements of heat release rate. The drift of the analyser shall be less than 0,01 % (100 ppm) over a period of 30 min. The time delay of the system, including the time constant of the instrument, shall not exceed 25 s (measured in accordance with Annex B).

#### 5.5.8.3.3 Carbon monoxide and carbon dioxide measurement

The carbon dioxide analyser shall be of the IR type and capable of measuring at least a range of 0 % to 10 % carbon dioxide. The accuracy of the analyser shall be at least  $\pm 1$  % of full scale or 0,1 % (1 000 ppm). The carbon monoxide analyser shall also be of the IR type and capable of measuring at least a range of 0 % to 1 % carbon monoxide. The accuracy of the analyser shall be at least  $\pm 1$  % of full scale or 0,01 % (100 ppm). The time delay of the system, including the time constant of the instrument, shall not exceed 25 s (measured in accordance with Annex B).

#### 5.5.8.4 Smoke obscuration measurement

**5.5.8.4.1** Install an optical system for measurement of light obscuration across the centreline of the exhaust duct. Determine the relative optical density of the smoke by measuring the light transmitted with a photometer system consisting of a white light source and a photocell/detector or a laser system for measurement of light obscuration across the centreline of the exhaust duct.

**5.5.8.4.2** One photometer system found suitable consists of a lamp, lenses, an aperture and a photocell. See Figure 21 and Annex B. Construct the system so that soot deposits on the optics during a test do not reduce the light transmission by more than 5 %.

**5.5.8.4.3** Alternatively, instrumentation can be constructed using a 0,5 mW to 2,0 mW helium-neon laser, instead of a white light system. See Figure 22 and B.4.2. It has been shown that white light and laser systems will give similar results [15].

## 6 Significance and use

**6.1** This test method is used primarily to determine the heat release rate of materials, products and assemblies. Other parameters determined are mass loss rate, the time to ignition, and smoke and gas production. These properties are determined on a specimen which may be an assembly of materials or products that are tested in their end-use thickness. Therefore, the heat release rate of a wall assembly, for instance, can be determined.

**6.2** Representative joints and other characteristics of an assembly shall be included in a specimen when these details are part of the normal design.

**6.3** This test method is applicable to end-use products not having an ideally planar external surface. The radiant flux field shall be adjusted to be that which is desired at the average distance of the surface from the radiant panel.

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5) This is the equivalent of 100 ppm; ppm is a deprecated unit.



## 7 Test specimens

### 7.1 Size and preparation

**7.1.1** The dimensions of test specimens shall be 1 000 mm × 1 000 mm and up to 150 mm in thickness and shall be representative of the construction of the end-use product. Materials and assemblies of normal thickness 150 mm or less shall be tested using their full thickness. If specimens of thickness greater than 150 mm are to be tested, a specimen holder (5.1.4) can be constructed to accommodate the desired specimen thickness up to 500 mm.

**7.1.2** If a product is designed normally to have joints in a field application, then that specimen shall incorporate the joint detail. The joint shall be centred in the specimen's vertical or horizontal centreline as appropriate. The specimen shall also be tested without a joint detail if the design does not include a joint.

**7.1.3** The edges of the specimen shall be covered with 12 mm ceramic wool blanket to eliminate the gap between the holder and the specimen.

### 7.2 Conditioning

Specimens shall be conditioned to moisture equilibrium (constant mass) at an ambient temperature of  $(23 \pm 3)$  °C and a relative humidity of  $(50 \pm 5)$  %.

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

## 8 Calibration of apparatus

### 8.1 General

Calibrate all instruments carefully with standard sources after initial installation. Among the instruments to be calibrated are load cells or weighing platforms, smoke meters, flow or velocity transducers and gas analysers.

### 8.2 Heat flux uniformity

Determine the set temperature of the controller (5.1.2) using the following procedure. Ignite the radiant panel and allow it to reach a burner surface temperature of 850 °C set on the controller. Insert the heat flux meter in the centre hole of the calibration panel so that the sensing face of the heat flux meter extends 15 mm toward the radiant panel from the exposed surface of the calibration panel to minimize the convective heat transfer contribution. Move the calibration panel with the heat flux meter inserted in the centre hole to the predetermined distance of 650 mm from the radiant panel (measure from the ceramic burner surface to the gauge sensing surface). Adjust the burner surface temperature on the controller to a value required for the heat flux meter to read 35 kW/m<sup>2</sup>. Adjust the individual burner outputs by adjusting the needle valves to obtain uniform irradiance over the calibration board. This is accomplished by moving the heat flux meter around all the holes in the calibration panel and adjusting the needle valves of the burners in the corresponding radiant panel area as necessary. Typically, after the final adjustment, the burners in the central area of the calibration board will have the lowest output and the burners on the edges of the radiant panel will have the highest output. The uniformity shall be  $\pm 5$  % of the average heat flux.

### 8.3 Heat flux/distance relationship

**8.3.1** A curve of average flux measurements over the specimen surface versus specimen distance from the radiant panel shall be generated. The calibration panel shall be placed in the position where the projected heat flux meter sensing surface is considered the specimen location. After the calibration panel has come to equilibrium, the flux measurements shall be made with the target face of the flux meter at the following distances away from the radiant panel: 300 mm, 400 mm, 600 mm, 800 mm, 1 000 mm and 2 000 mm.

**8.3.2** No individual flux measurement shall deviate from the average at each of the distances by more than  $\pm 5\%$ .

**8.3.3** The curve generated in 8.3.1 shall be used to determine the distance from the radiant panel for a desired irradiance exposure.

**8.3.4** Calibration shall be performed every three months, or more frequently if any significant changes to equipment are made or if calibration is suspect.

## 8.4 Heat release

**8.4.1** Perform the calibration of the heat release instrumentation in the exhaust duct by burning propane or natural gas and comparing the heat release rates calculated from the metered gas input, and those calculated from the measured oxygen consumption. The value of net heat of combustion for propane is 46,5 MJ/kg. When using natural gas for calibrating the heat release instrumentation, the net heating value, net heat released per unit mass of oxygen, expansion coefficient and density shall be obtained from the natural gas supplier for the actual natural gas used at the time of the calibration. Position the calibration burner in the same location where the specimen is to be placed during a 35 kW/m<sup>2</sup> exposure test. Measure the gas flow at standard atmospheric pressure of (101  $\pm$  5) kPa (measured at the flow gauge) and standard temperature of (20  $\pm$  5) °C.

**8.4.2** The calibration source for the test shall be a gas burner with a nominal 0,3 m  $\times$  0,3 m porous top surface of a refractory material. The top surface of the burner through which the gas is supplied shall be located horizontally, 0,3 m off the floor. The burner shall be supplied with natural grade propane (95 % purity) or natural gas. The gas for the burner flame shall not be premixed with air. The gas flow to the burner shall be measured with an accuracy of at least  $\pm 3\%$ . The heat output to the burner shall be kept constant and controlled within  $\pm 5\%$  of the prescribed value.

A burner may be constructed with a 25 mm thick porous ceramic fibreboard over a 152 mm plenum; or alternatively a minimum 100 mm layer of Ottawa sand can be used to provide the horizontal surface through which the gas is supplied. The sand burner may be preferable for economic reasons. This type of burner is shown in Figure 24.

The burner may be ignited by a pilot burner or a remotely controlled spark igniter. Burner controls shall be provided for automatic shut-off of the gas supply if flameout occurs.

**8.4.3** Another calibration burner is a pipe, with an inner diameter of (100  $\pm$  1,5) mm, supplied with gas from beneath as described in ISO 9705. The gas for the burner flame shall not be premixed with air.

**8.4.4** Obtain a minimum of two calibration points. Obtain a lower heat release rate value of 350 kW and a higher heat release rate of 600 kW. Approximate propane flows for any required heat release rate value are estimated using the following constant: 1 485 kW min/l, determined at standard atmospheric pressure of (101  $\pm$  5) kPa (measured at the flow gauge) and standard temperature of (20  $\pm$  5) °C.

**8.4.5** Take measurements at least once every 2 s and start 1 min prior to ignition of the burner. Determine the average heat release rate over a period of at least 1 min by

- the oxygen consumption method, and
- calculating the heat release rate from the gas mass flow and the net heat of combustion.

Correct factors for the heat released per oxygen consumed and the combustion expansion factor for the calibration fuel gas shall be used in the oxygen consumption method [Equation (D.4)]. Note that for propane the heat released per oxygen consumed is  $E_{\text{propane}} = 12,78$  (MJ/kg O<sub>2</sub>) and the combustion expansion factor is  $\alpha_{\text{propane}} = 1\,040$ . The difference between the measured heat release rate, comparing time average values over 1 min, shall not be more than 5 % of the actual heat output from the burner. The two values shall not exceed 5 %.

**8.4.6** Calibration shall be performed every three months, or more frequently if any significant changes to equipment are made or if calibration is suspect.

**8.4.7** When calibrating a new system, or when modifications are introduced, check the response time of the measurement system using the following test sequence:

Time	Burner output
0–5 min	0 kW
5–10 min	350 kW
10–20 min	600 kW
20–25 min	0 kW

The response of the system to a stepwise change of the heat output from the burner shall be a maximum of 15 s to 90 % of final value.

**8.4.8** Perform the calibration in 8.4.7 at a duct air flow of  $2 \text{ m}^3/\text{s} \pm 5 \%$  if the exhaust system (5.1.6) shown in Figure 19 is used. A minimal duct flow predetermined to be sufficient to collect all the combustion gases of the largest expected fire (about 1 000 kW) shall be used for the exhaust systems different to that shown in Figure 19. However, the flow shall not be larger than  $2,5 \text{ m}^3/\text{s}$  to avoid noisy measurements.

**8.4.9** The change in measured heat release rate for each step, comparing time average values over 1 min, shall not be more than 5 % of the change in actual heat output for that step.

## 8.5 Mass loss

If required by the type of scale used, perform the calibration by loading the weighing platform (5.1.5) with known masses corresponding to the measuring range of interest, to ensure that accuracy conforms to 5.4.2. Calibrate the weighing platform daily, prior to testing.

## 8.6 Smoke obscuration

Calibrate the smoke meter initially to read correctly for two neutral density filters at 0,5 and 1,0 values of relative optical density, and also at 100 % transmission. Once this calibration is set, it is only necessary to set the zero value of extinction coefficient (100 % transmission) each day, prior to testing. Investigate any excessive departure from the zero line at the end of a test, and correct it.

## 8.7 Gas analysis

**8.7.1** Calibrate gas analysers daily, prior to testing (see ASTM E800 <sup>[14]</sup> for further guidance).

**8.7.2** The analyser delay times shall be determined by arranging for a propane flow rate equivalent to 350 kW to the calibration burner. The radiant panel shall not be turned on for this calibration. Record the output of the analyser as the propane supply, turned on and ignited, reaches a steady value, and then returns to baseline after the supply is cut off. Record the temperature for the exhaust flow probe at the same time. Determine the turn-on delay as the time difference between the time when the temperature reading reaches 50 % of its ultimate deflection and the time when the gas reading reaches 50 % of its ultimate deflection. Determine the turn-off delay similarly at turn-off. Take the delay time as the average of the turn-on delay and turn-off delay. Use these values for the individual analysers subsequently to time-shift all the gas concentration readings.

## 8.8 Heat flux meter

The heat flux meter shall be calibrated in accordance with ISO 14934-3.

## 9 Test methods

### 9.1 Preparation

9.1.1 Position the specimen holder (5.1.4) assembly remote to the test location.

9.1.2 Place the water-cooled shield (5.1.3) in front of the radiant panel assembly (5.1.1) and adjust the water flow sufficiently high so that water exiting the shield does not exceed 50 °C.

9.1.3 Insert the specimen into the specimen holder. Position the specimen by removing the top specimen holder cap section, inserting the specimen and replacing the top cap or by inserting the specimen from the back of the specimen holder against the specimen holder lip and arranging for the specimen to stay in place during the test.

9.1.4 Establish a duct flow of 2,0 m<sup>3</sup>/s if the exhaust system (5.1.6) described in Figure 19 is used. Other suitable duct flows shall be used for different exhaust systems. However, the duct flow shall not be more than 2,5 m<sup>3</sup> to avoid excessive measurement noise.

9.1.5 Turn on the flow of gas to the radiant panel and ignite the burners.

9.1.6 Operate the burners for 30 min prior to testing.

9.1.7 Turn on all sampling and recording devices and calibrate the analysers.

9.1.8 Switch on the wire igniters.

### 9.2 Procedure

9.2.1 Move the specimen trolley to the location necessary for the desired flux exposure.

9.2.2 Collect baseline data for 1 min after the signal from the weighing platform (6.1.5) settles down to equilibrium.

9.2.3 Remove the water-cooled specimen shield (5.1.3) in not more than 2 s and start the timer, marking the beginning of the test.

9.2.4 Record the time when flashing or transitory flaming occurs; when sustained flaming occurs, record the time and turn the igniters off. If the flame becomes extinguished, turn the igniters on again.

The igniters are turned off to prevent them from overheating and breaking. The igniters can be left on during entire tests if the possibility of them breaking is not a concern.

9.2.5 If the duct flow is not sufficient to collect all the fire gases, then the duct flow shall be gradually increased.

9.2.6 Record all significant events during the test, such as cracking, melting, collapse of all or part of the specimen, deformations and intumescing.

9.2.7 Collect data for 2 min after sustained flaming occurs on the unexposed side of the specimen or 32 min if the specimen does not burn through.

9.2.8 Unless otherwise specified in the material or performance standard, make three determinations and report as specified in Clause 11.

## 10 Calculations

**10.1** The specimen heat release rate is calculated by subtracting the radiant panel assembly heat release rate from the total heat release rate. The radiant panel heat release rate contribution measured by the natural gas flow rate shall be multiplied by a factor of 1,05 to use the correct factor of heat released per oxygen consumed for natural gas ( $E_{\text{methane}} = 12,51$ ).

NOTE The factor of 1,05 and  $E_{\text{methane}}$  given above are for methane. Correct factor and  $E$  factor should be calculated for the actual natural gas used based on its composition.

**10.2** Considerations for heat release measurements are presented in Annex C. Calculate heat release data using the equations in D.1 and D.2. Use one of the equations in D.1 to calculate heat release, based on the gas analysers installed.

**10.3** Calculate mass loss rate using the procedures in D.4.

**10.4** Calculate smoke production data using the equations in D.3.

**10.5** The exposed surface area of the specimen is 0,84 m<sup>2</sup>. Use 0,84 m<sup>2</sup> to calculate parameters per unit surface area.

## 11 Test report

The following information shall be included in the test report.

### 11.1 Descriptive information

The test report shall include the following information:

- a) name and address of the testing laboratory;
- b) specimen identification code or number;
- c) date and identification number of the report;
- d) name and address of the test sponsor;
- e) name of product manufacturer or supplier, if known;
- f) composition or generic identification;
- g) density, or mass per unit surface area, total mass, thickness of the main components in the specimen, thickness of the specimen, moisture content of hygroscopic materials and mass of combustible portion of specimen, if known;
- h) description of the specimen, if different from the product;
- i) details of specimen preparation by the testing laboratory;
- j) details of special mounting methods used;
- k) heating flux and exhaust system flow;
- l) number of replicates tested under the same conditions. (This shall be a minimum of three except for exploratory testing.);
- m) conditioning of the specimens;
- n) date of test;

- o) test number and any special remarks;
- p) test results (see Annex F).

### **11.2 Table of numerical results**

The table of numerical results shall include the following:

- a) time to sustained flaming(s);
- b) peak heat release rate (kW), and the time at which it occurred/occurs;
- c) average heat release rate values for the first 60 s, 180 s and 300 s after ignition, or for other appropriate periods (kW);
- d) total heat released (MJ);
- e) peak instantaneous rate of production of light-obscuring smoke ( $\text{m}^2/\text{s}$ ), and the time at which it occurred;
- f) average instantaneous rate of production of light-obscuring smoke values for the first 60 s, 180 s and 300 s after ignition, or for other appropriate periods ( $\text{m}^2/\text{s}$ );
- g) total amount of smoke ( $\text{m}^2$ );
- h) total mass loss (kg);
- i) total percentage of mass loss (%);
- j) equation used to calculate heat release rate;
- k) average yield of carbon monoxide (kg CO/kg fuel) (optional).

### **11.3 Graphical results**

The graphical results shall include the following:

- a) plot of heat release rate vs. time;
- b) plot of instantaneous rate of production of light-obscuring smoke vs. time;
- c) plot of mass loss vs. time;
- d) plot of mass loss rate vs. time;
- e) plots of the duct temperature vs. time;
- f) plot of mass flow in the exhaust duct vs. time;
- g) plot of the heat release rate vs. time from the radiant panel (baseline).

### **11.4 Descriptive results**

The descriptive results shall include the following:

- a) photographs or videotape of the fire development;
- b) all available information requested in 9.2.6.

## 12 Test limitations

The test data may have limited validity if any of the following occur:

- a) the specimen melts sufficiently to overflow the drip tray;
- b) explosive spalling occurs.

## 13 Hazards

The test procedures involve high temperatures and combustion processes. Therefore, hazards may exist for burns, ignition of extraneous objects or clothing and for inhalation of combustion products. The operator shall use protective gloves and clothing while removing the specimen shield (5.1.3) and while moving the specimen trolley toward or away from the radiant panels. The construction of a viewing wall with windows is recommended for laboratories with small spaces where the operator and viewers cannot move far enough away from the area of the radiant panel.

The water-cooled shield (5.1.3) placed in front of the radiant panel assembly (5.1.1) dramatically lowers the heating of the laboratory space. Additionally, it lowers the potential for harm to operators working in the area.

## 14 Precision and bias

Information regarding the precision and bias of the test method is given in Annex G.

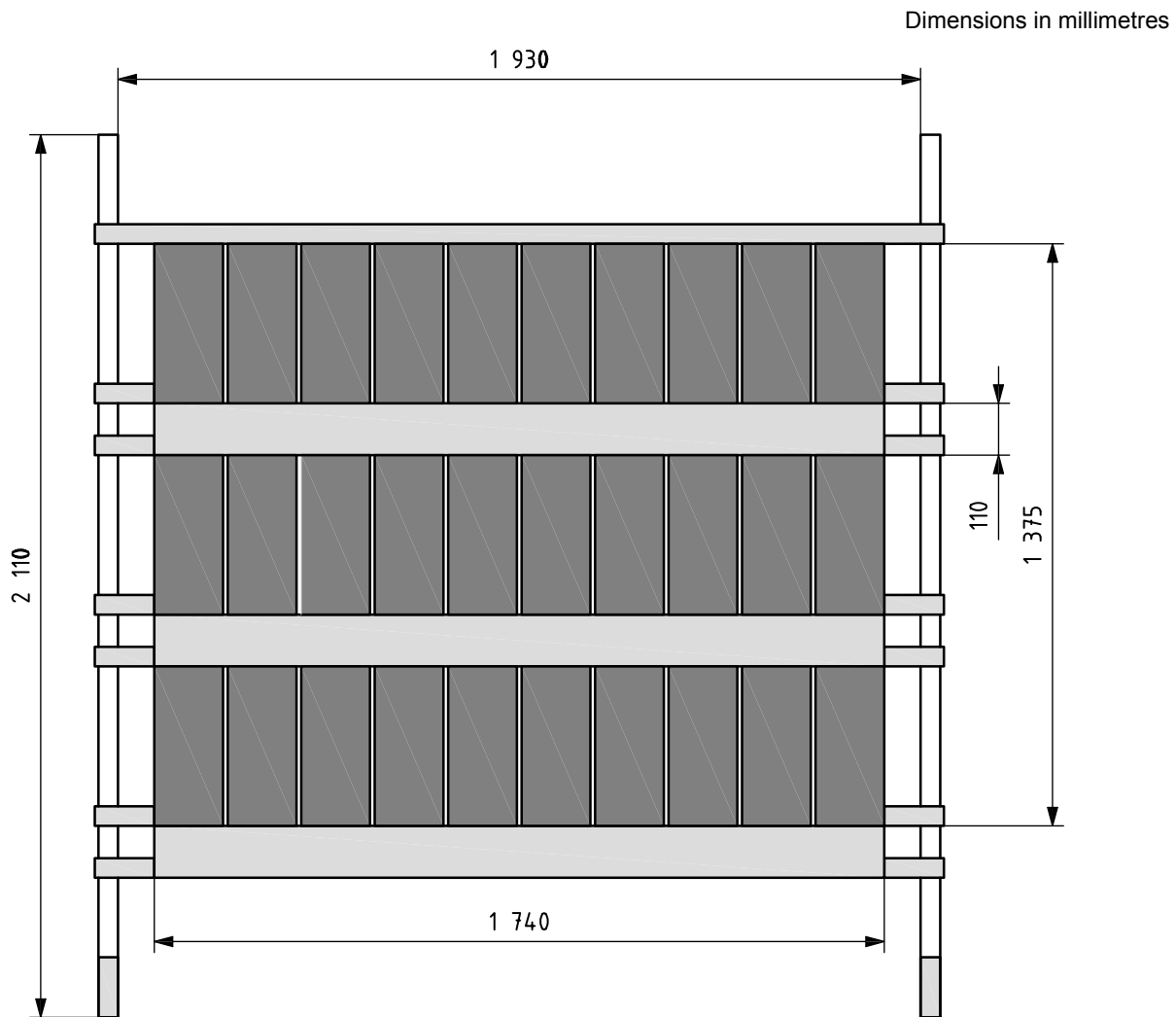
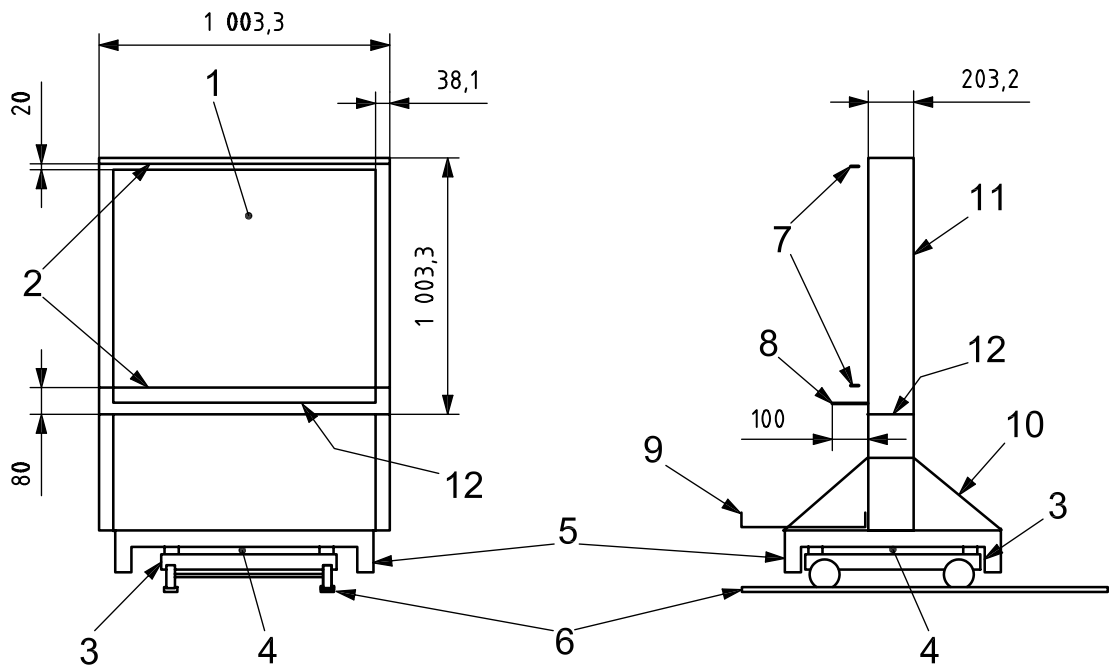


Figure 1 — Radiant panel assembly



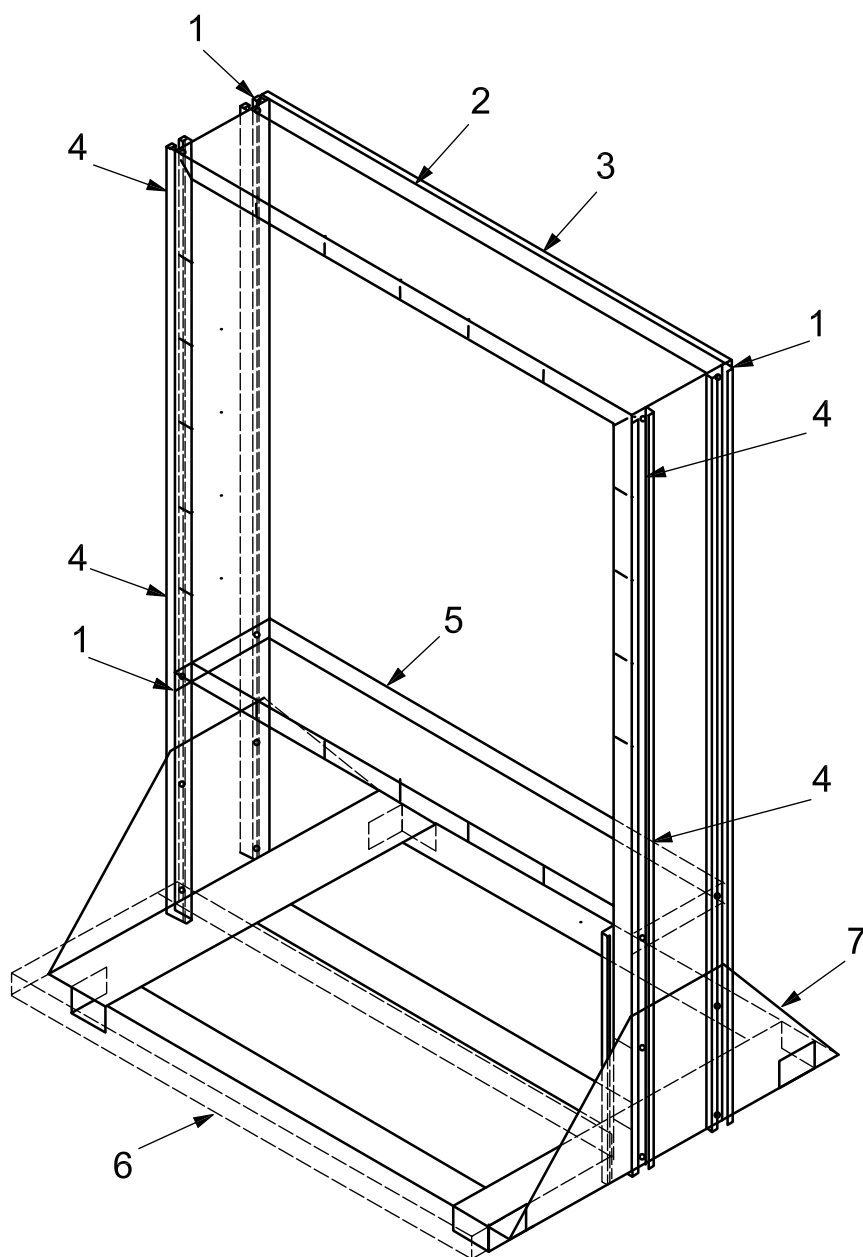
Dimensions in millimetres



**Key**

- |   |                                    |    |  |
|---|------------------------------------|----|--|
| 1 | exposed surface                    | 7  | wire igniters                            |
| 2 | wire igniters                      | 8  | air-stream-interrupting projection plate |
| 3 | scale                              | 9  | drip tray                                |
| 4 | air space                          | 10 | specimen holder base                     |
| 5 | ceramic fibreboard heat shield     | 11 | specimen holder piece 1                  |
| 6 | specimen holder trolley and tracks | 12 | specimen holder piece 2                  |

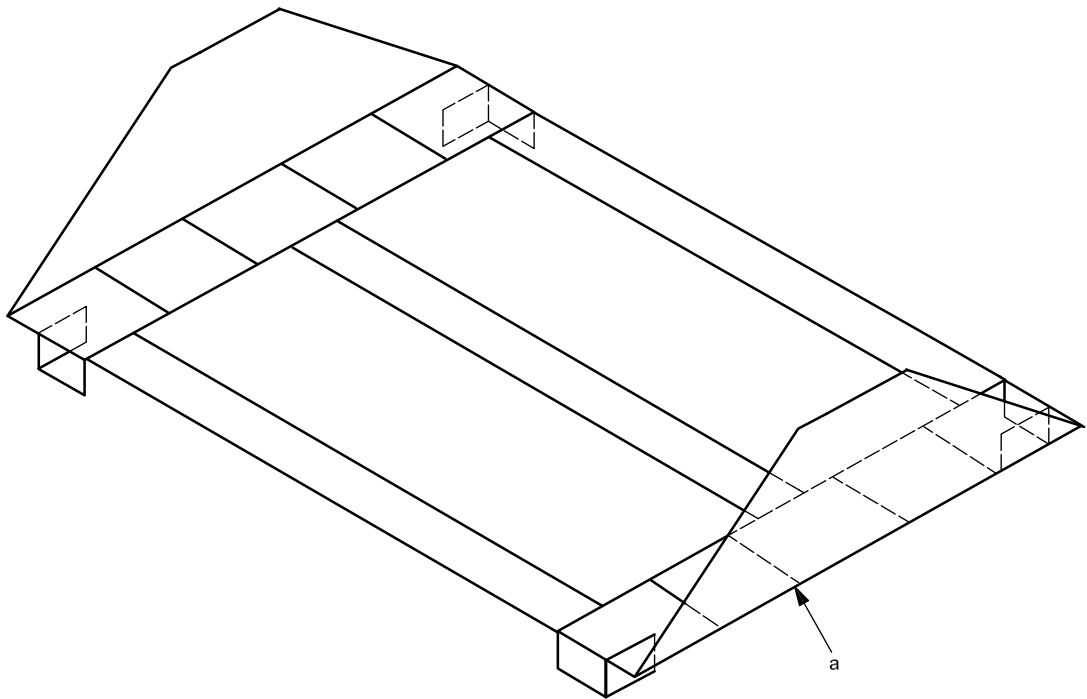
**Figure 2 — Specimen holder with scale, heat shield, trolley and tracks, front and side views**



**Key**

- 1 power-strut channel, 7 places
- 2 specimen holder piece 1
- 3 single channel across the top, fastened only at the ends to the vertical channels
- 4 wire igniter bracket location, 4 places
- 5 specimen holder piece 2
- 6 drip tray
- 7 specimen holder base

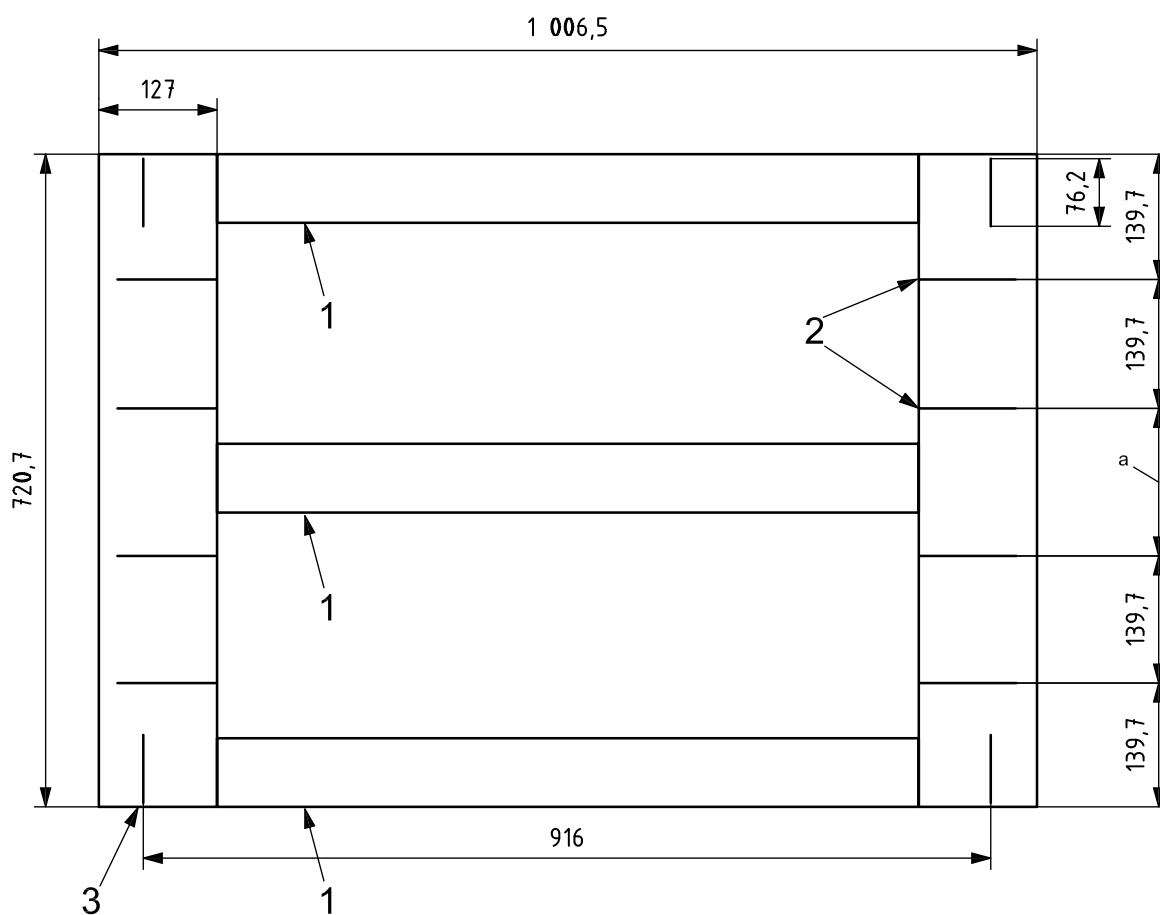
**Figure 3 — Assembled specimen holder, isometric view**



a Cuts end 12,7 mm from bend.

**Figure 4 — Specimen holder base, isometric view**

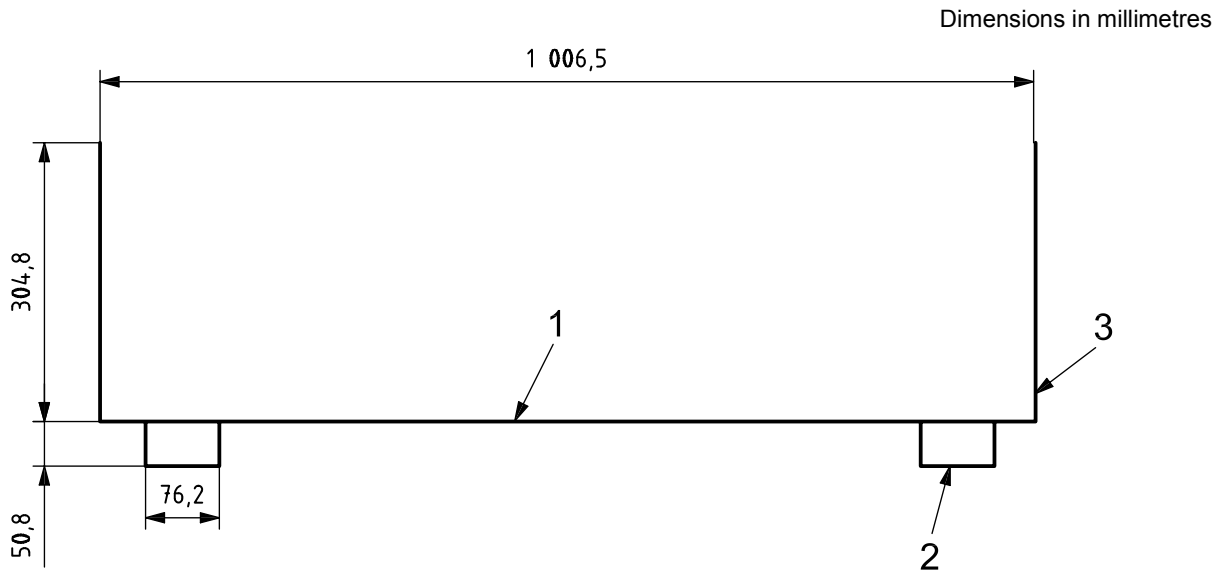
Dimensions in millimetres



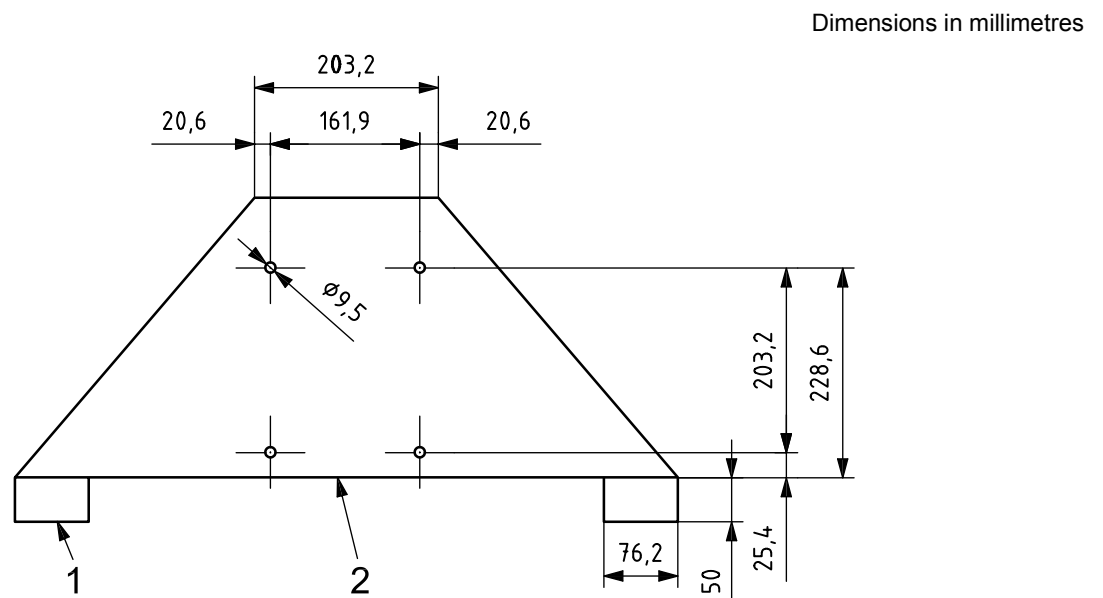
**Key**

- 1 3,4 mm × 76,2 mm × 752,5 mm mild steel, 3 places
- 2 1,5 mm wide cut ending 12,7 mm from bend, 8 places
- 3 2,1 mm mild steel, 2 places
- a Remainder.

**Figure 5 — Specimen holder base, top view**

**Key**

- 1 3,4 mm × 76,2 mm × 752,5 mm mild steel, 3 places
- 2 1,7 mm × 50,8 mm × 76,2 mm mild steel, 8 places, tack welded 90° to base only
- 3 2,1 mm mild steel, 2 places

**Figure 6 — Specimen holder base, front view****Key**

- 1 1,7 mm × 50,8 mm × 76,2 mm mild steel, 8 places, tack welded 90° to base only
- 2 2,1 mm mild steel, 2 places

**Figure 7 — Specimen holder base, side view**

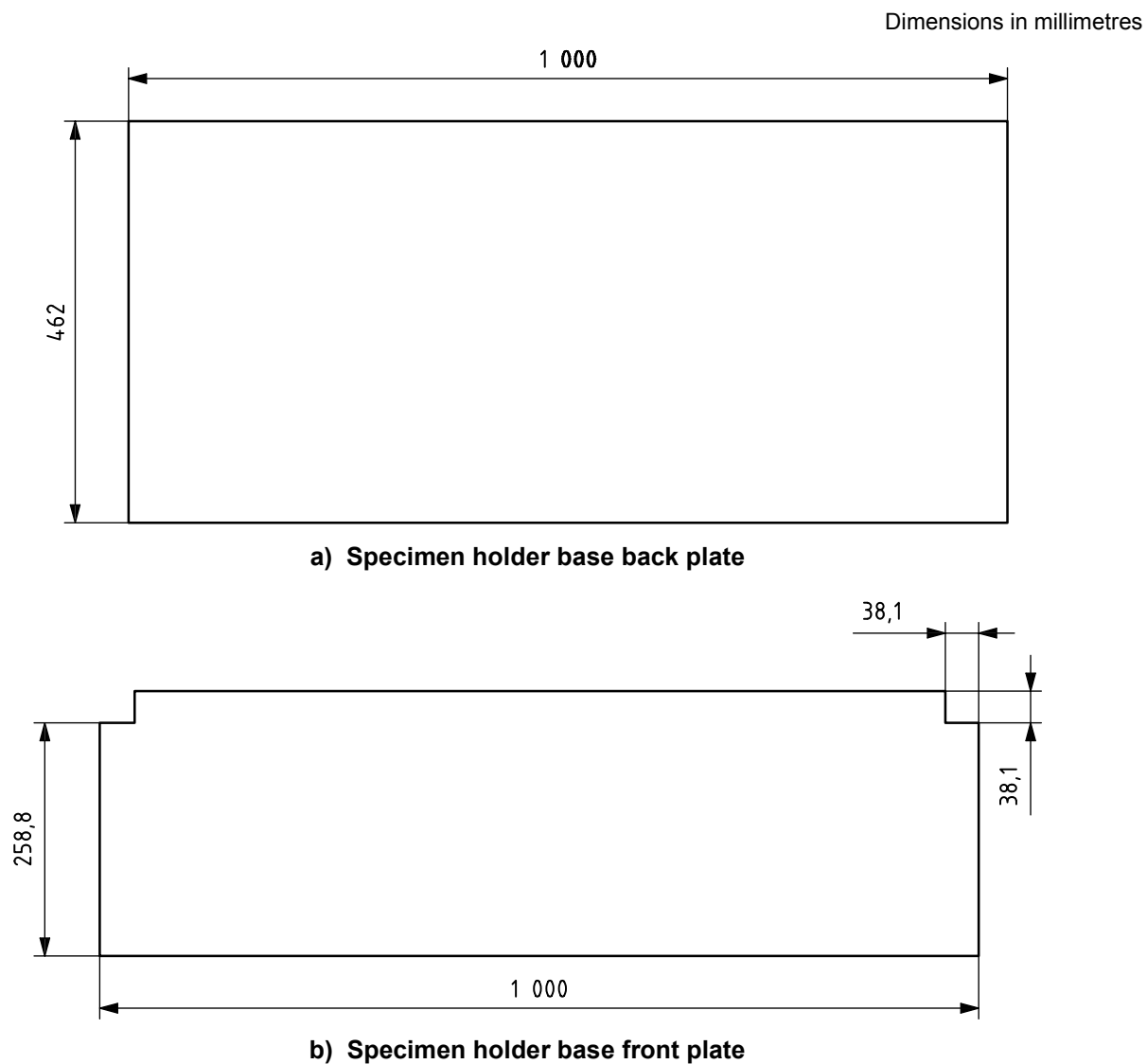


Figure 8 — Specimen holder base plates made of 1,7 mm mild steel

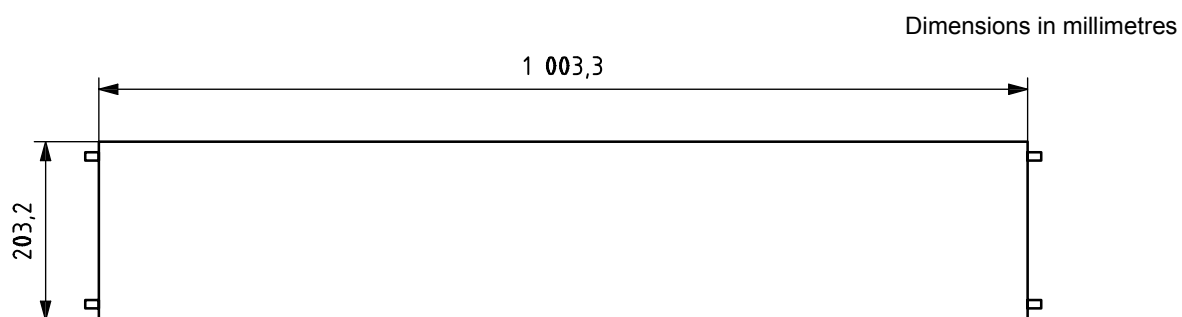
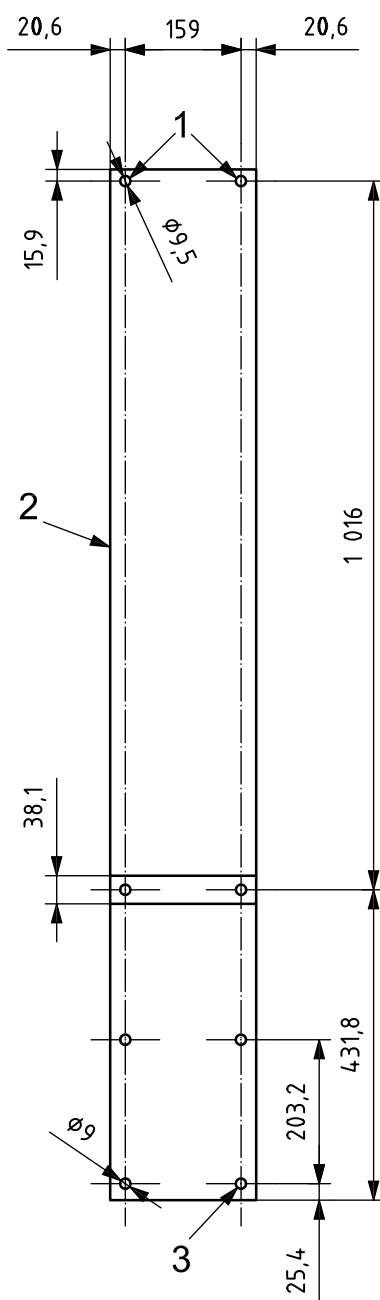


Figure 9 — Top view of specimen holder piece 1 made of 2,1 mm mild steel



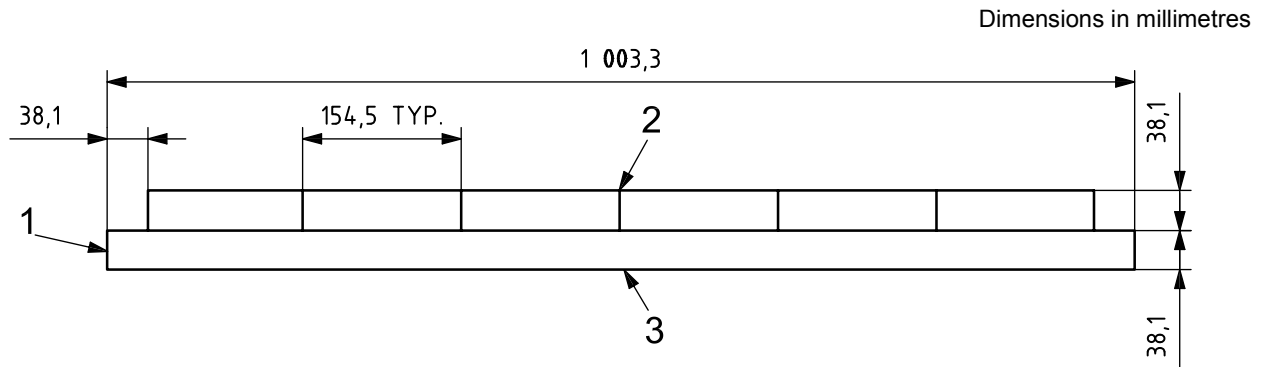


**Key**

- 1 9,5 mm N.C. welded studs, 4 places
- 2 specimen holder piece 1, 2,1 mm mild steel
- 3 9,5 mm diameter holes, 16 places

**Figure 11 — Specimen holder, side view**

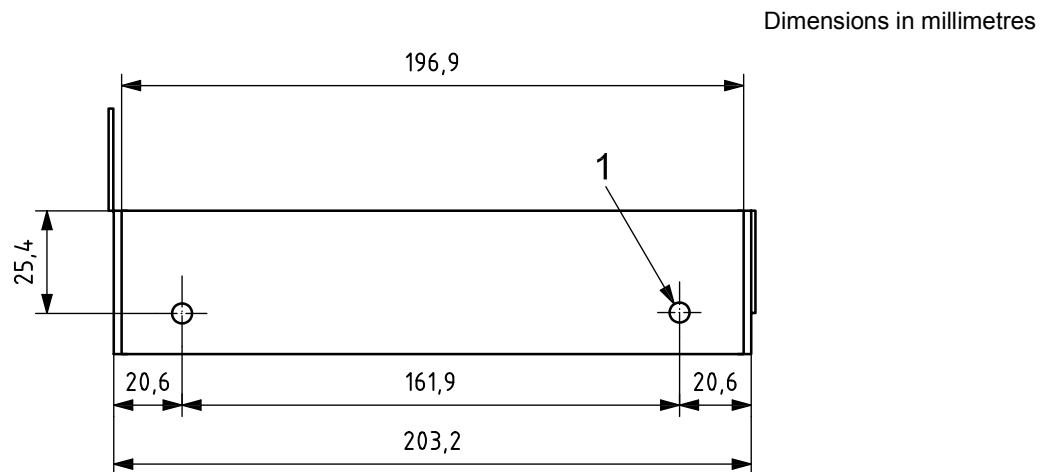




**Key**

- 1 9,5 mm diameter holes, 4 places
- 2 1,5 mm wide cut ending 12,7 mm from bend, 5 places
- 3 2,1 mm mild steel

**Figure 12 — Specimen holder piece 2, front view**



**Key**

- 1 9,5 mm diameter holes, 4 places

**Figure 13 — Specimen holder piece 2, side view**

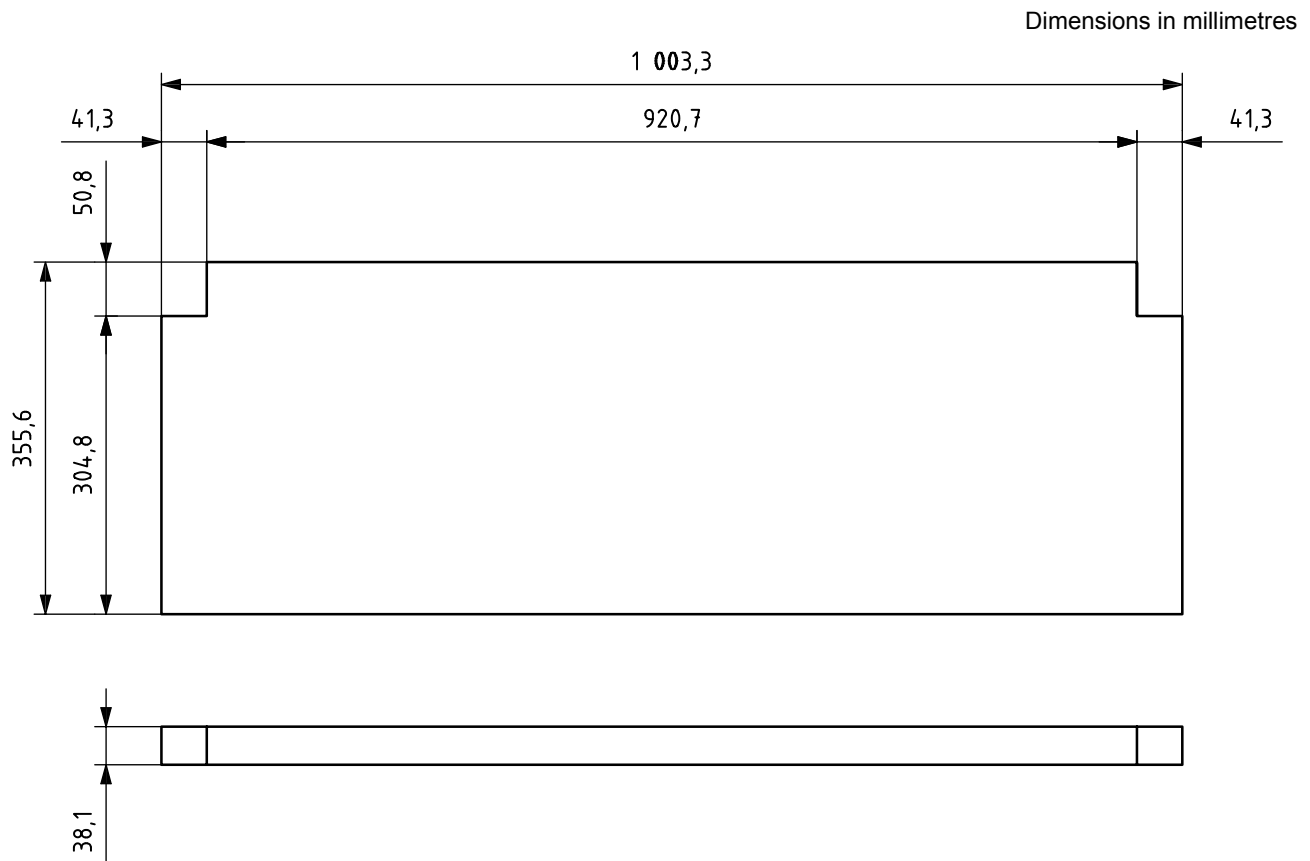
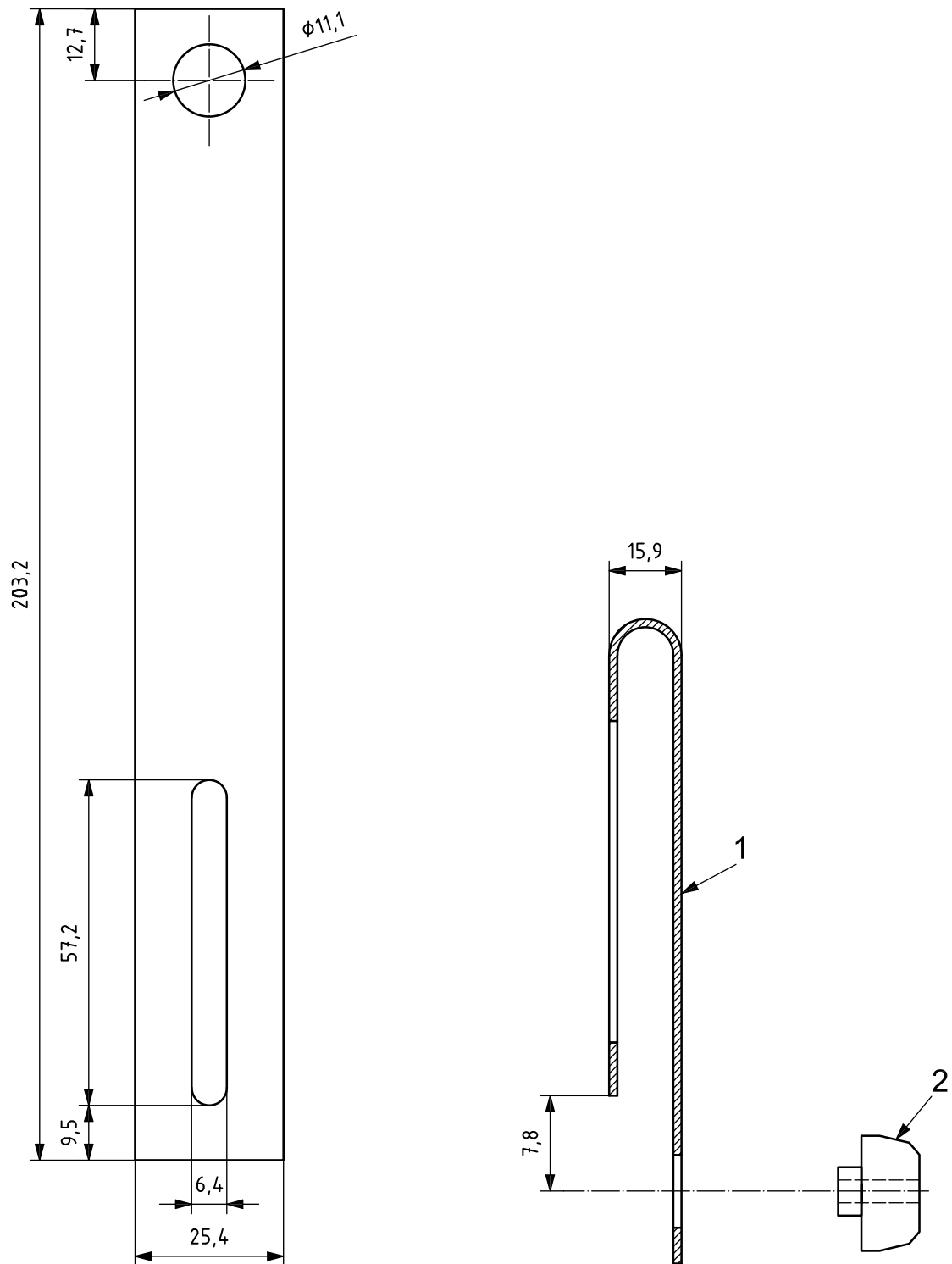


Figure 14 — Drip tray made of 1,7 mm thick mild steel

Dimensions in millimetres



a) Wire igniter bracket made of 1,7 mm mild steel

b) Wire igniter bracket

**Key**

- 1 bracket made of 1,7 mm stainless steel
- 2 ceramic insulator

**Figure 15 — Wire igniter bracket**

Dimensions in millimetres

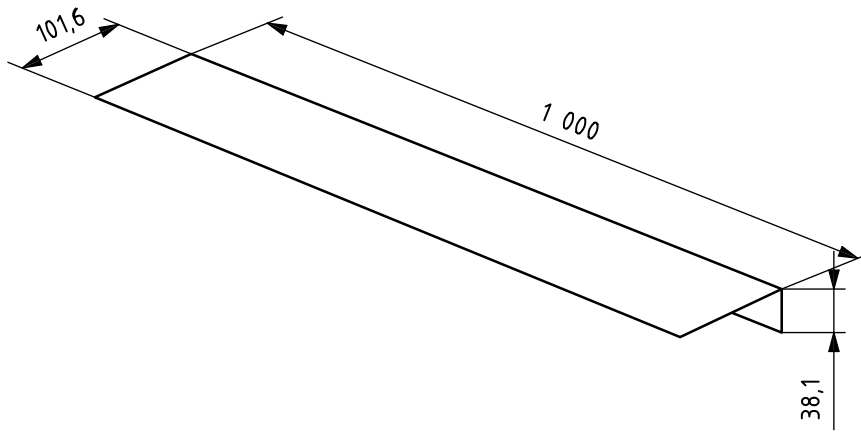
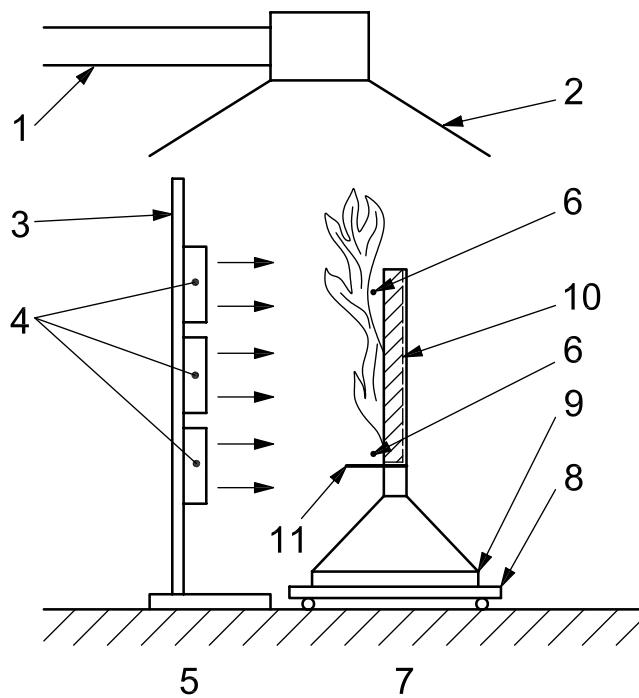


Figure 16 — Air-stream-interrupting projection plate made of 0,5 mm thick mild steel

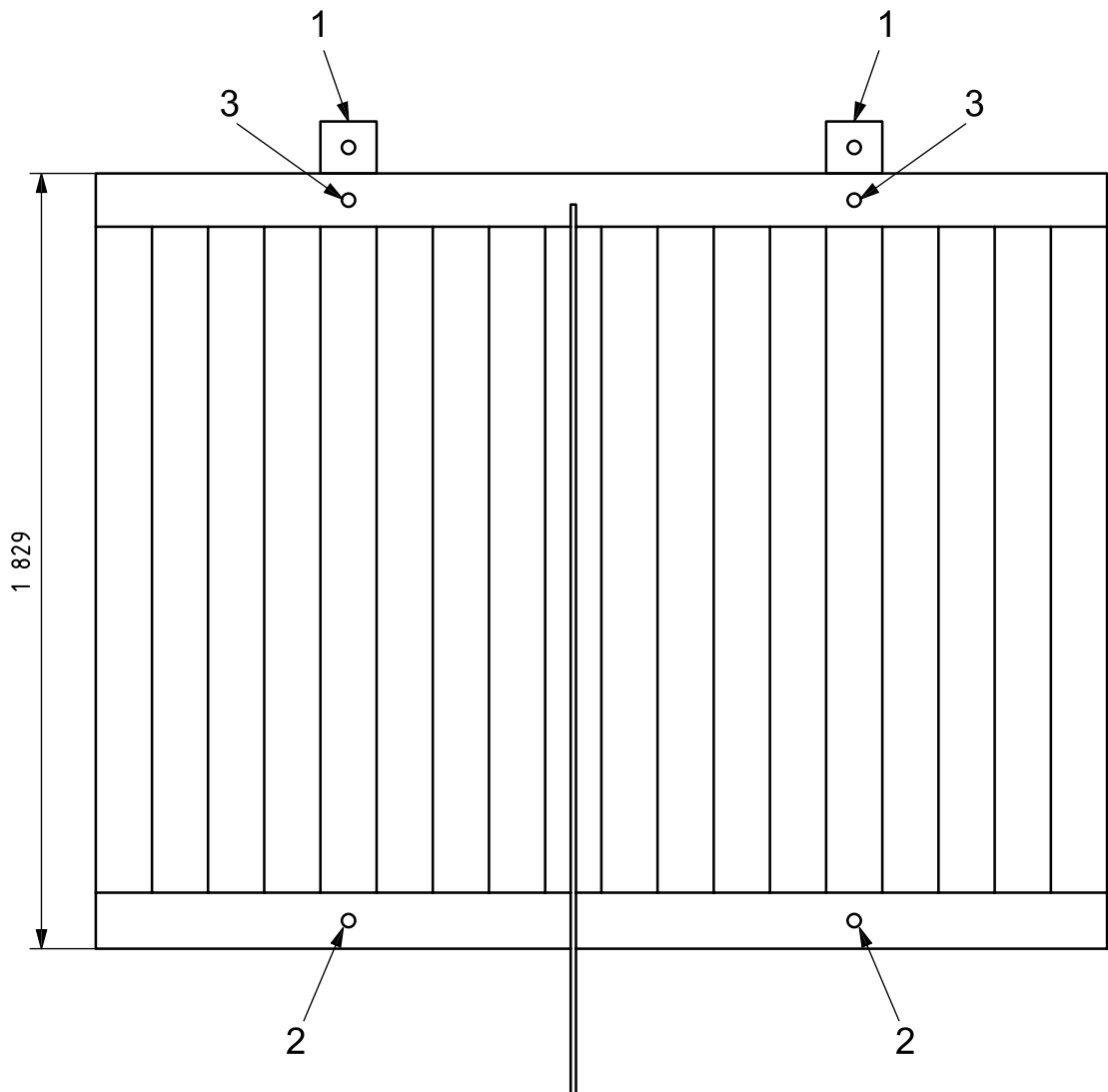


**Key**

- |   |                     |    |  |
|---|---------------------|----|--|
| 1 | gas sampling port   | 7  | specimen holder assembly                 |
| 2 | steel hood          | 8  | specimen holder trolley                  |
| 3 | supporting frame    | 9  | weighing platform                        |
| 4 | natural gas burners | 10 | specimen                                 |
| 5 | radiant panel       | 11 | air-stream-interrupting projection plate |
| 6 | wire igniter        |    |  |

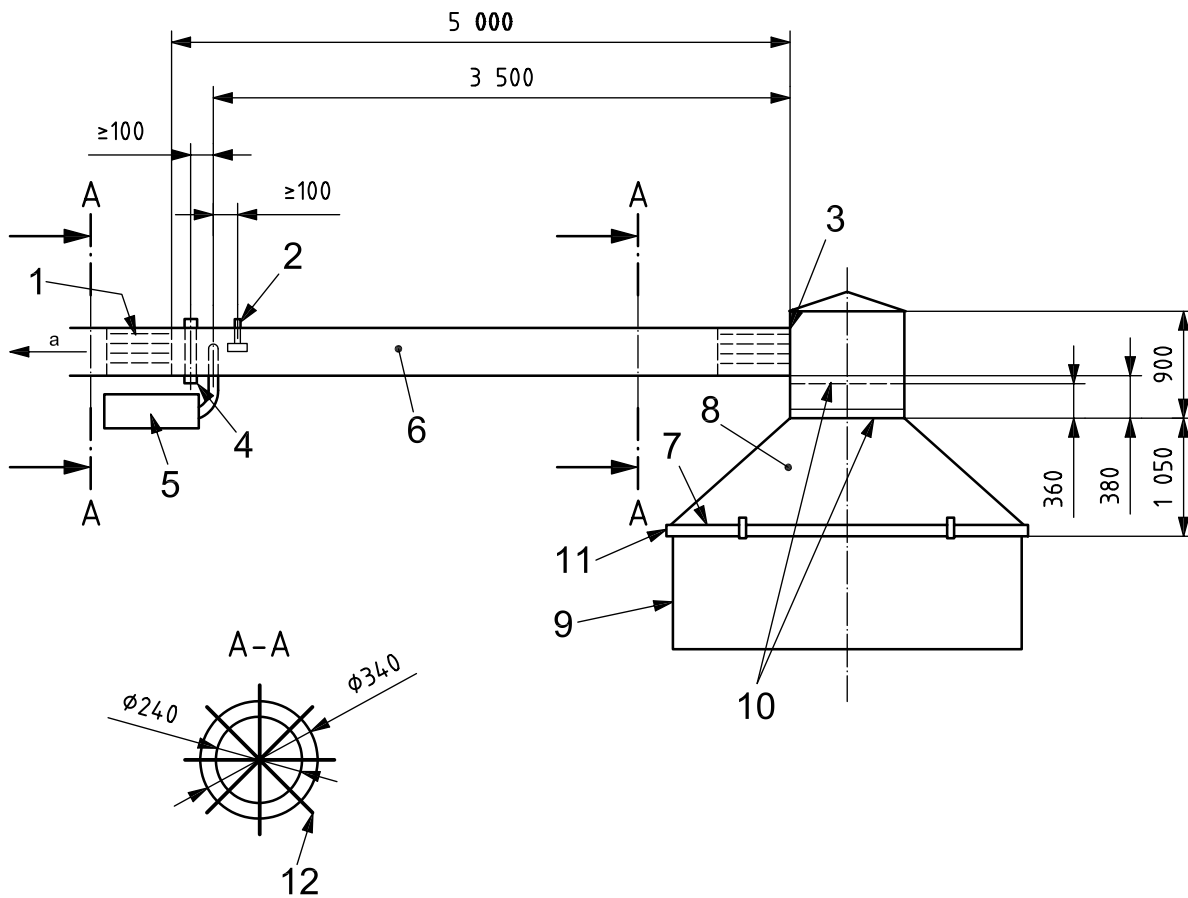
Figure 17 — Intermediate-scale calorimeter

Dimensions in millimetres

**Key**

- 1 support trolley attachment point
- 2 water inlet point
- 3 water outlet point

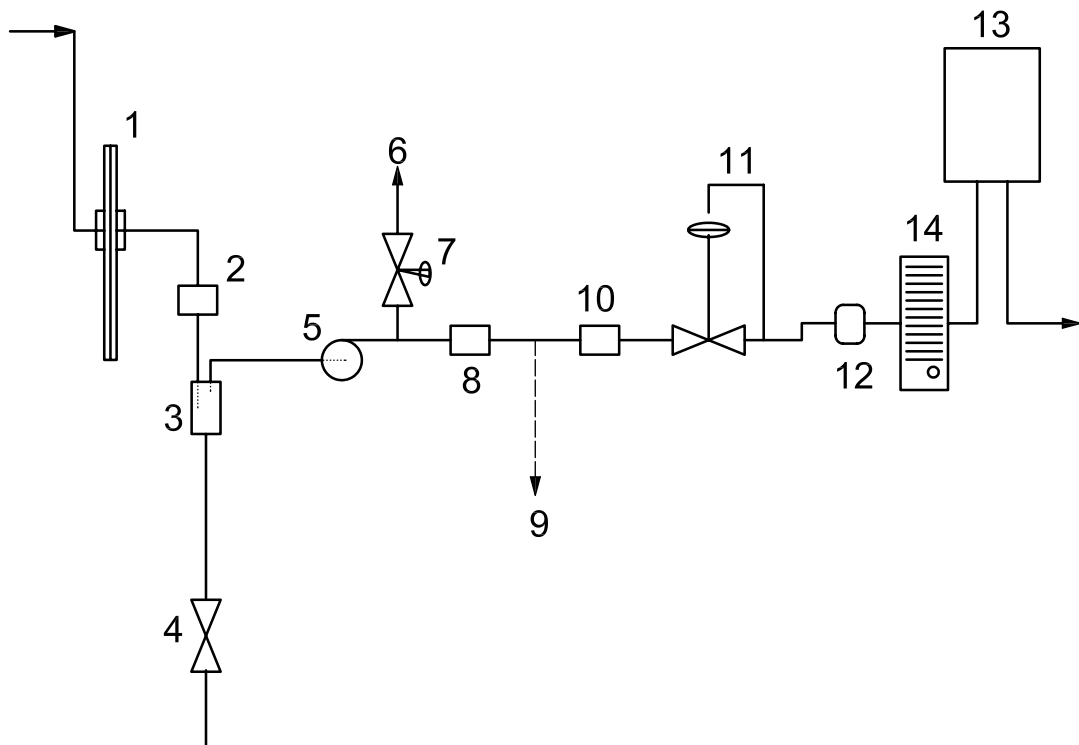
**Figure 18 — Heat shield**



**Key**

- |   |                               |    |   |
|---|-------------------------------|----|---|
| 1 | guide vanes                   | 7  | opening, 3 100 mm × 2 400 mm                  |
| 2 | bi-directional probe          | 8  | hood of 2 mm thick steel plates               |
| 3 | opening, $\varnothing$ 400 mm | 9  | steel plates (optional), 1 000 mm × 2 400 mm  |
| 4 | lamp, photocell system        | 10 | steel plates, 2 mm × 500 mm × 900 mm          |
| 5 | gas analysis                  | 11 | steel frame of profile 50 mm × 10 mm × 3,2 mm |
| 6 | exhaust duct                  | 12 | four steel plates, 395 mm × 400 mm            |
| a | To exhaust.                   |    |   |

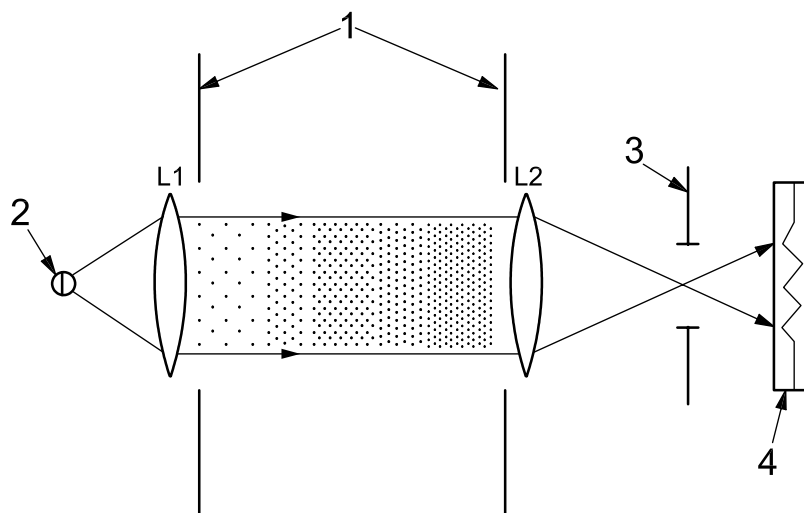
**Figure 19 — Sample design of collection hood and exhaust that meets the requirements of this International Standard**



**Key**

- |                      |                                      |                    |
|----------------------|--------------------------------------|--------------------|
| 1 soot filter        | 6 waste vent                         | 11 flow controller |
| 2 cold trap          | 7 waste vent regulator               | 12 7 µm filter     |
| 3 separation chamber | 8 desiccant                          | 13 oxygen analyser |
| 4 drain              | 9 to CO <sub>2</sub> and CO analyser | 14 rotameter       |
| 5 pump               | 10 CO <sub>2</sub> removal media     |                    |

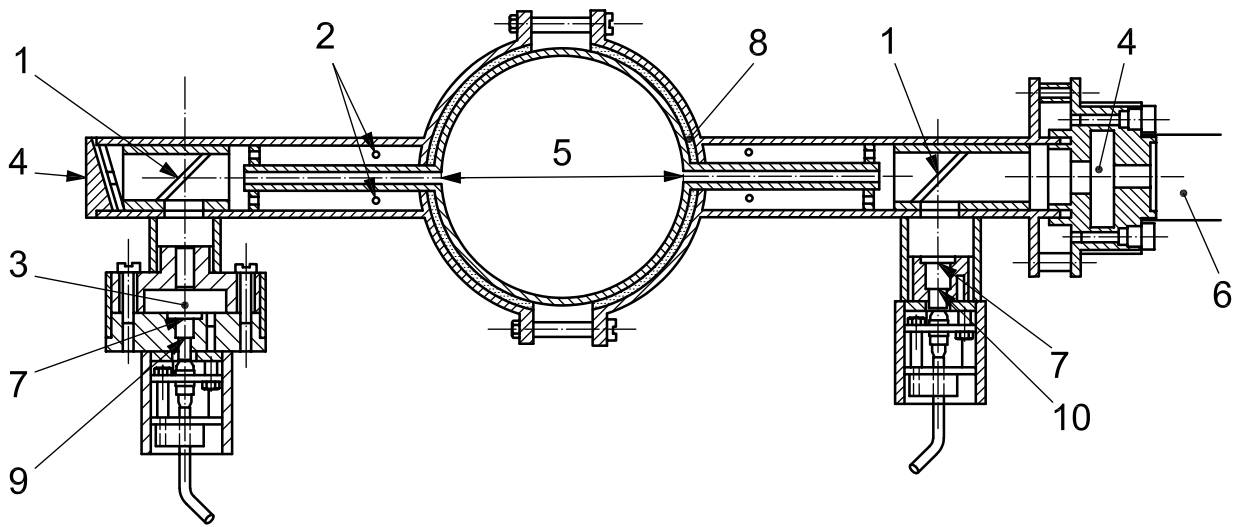
**Figure 20 — Schematic diagram of gas sampling train**



**Key**

- |                        |            |
|------------------------|------------|
| 1 wall of exhaust duct | 3 aperture |
| 2 lamp                 | 4 detector |
- L1 and L2: lenses of focal length  $f$

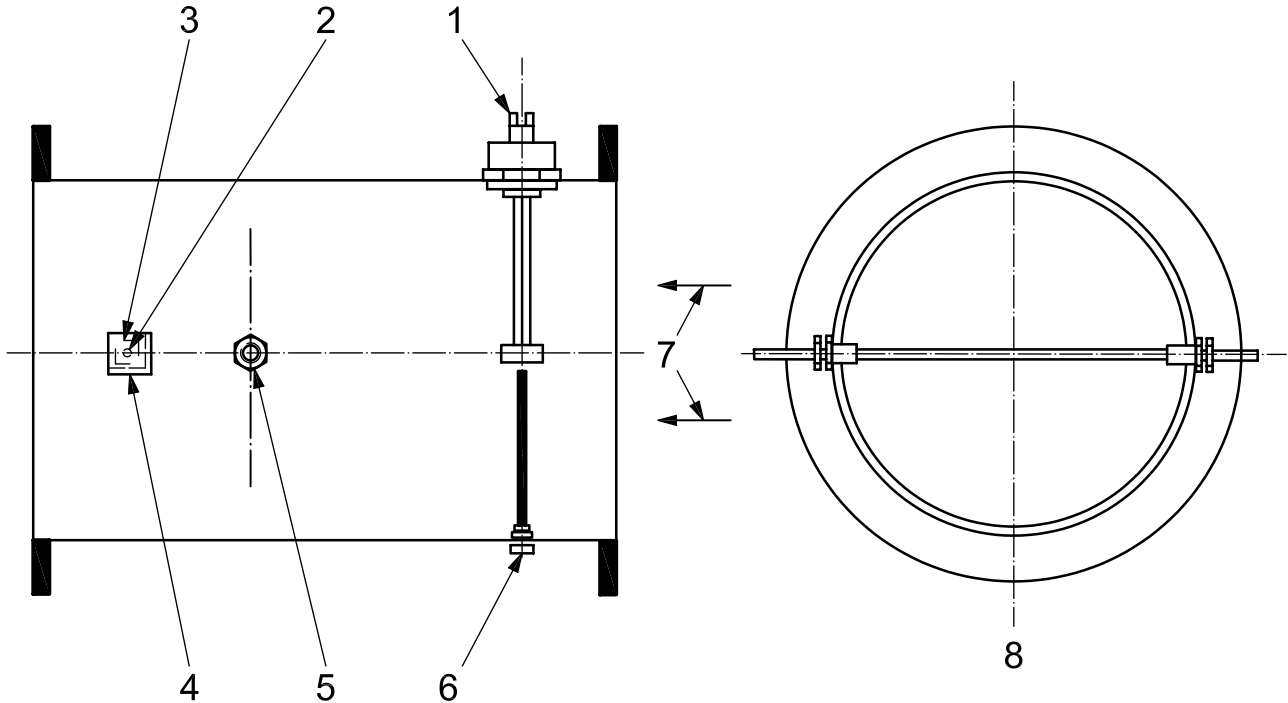
**Figure 21 — White light optical system**



**Key**

- |                      |                            |
|----------------------|----------------------------|
| 1 beam splitter      | 6 0,5 mW helium neon laser |
| 2 purge air orifices | 7 opal glass               |
| 3 filter slot        | 8 ceramic fibre packing    |
| 4 cap                | 9 main detector            |
| 5 optical path       | 10 compensation detector   |

**Figure 22 — Smoke obscuration measurement system**



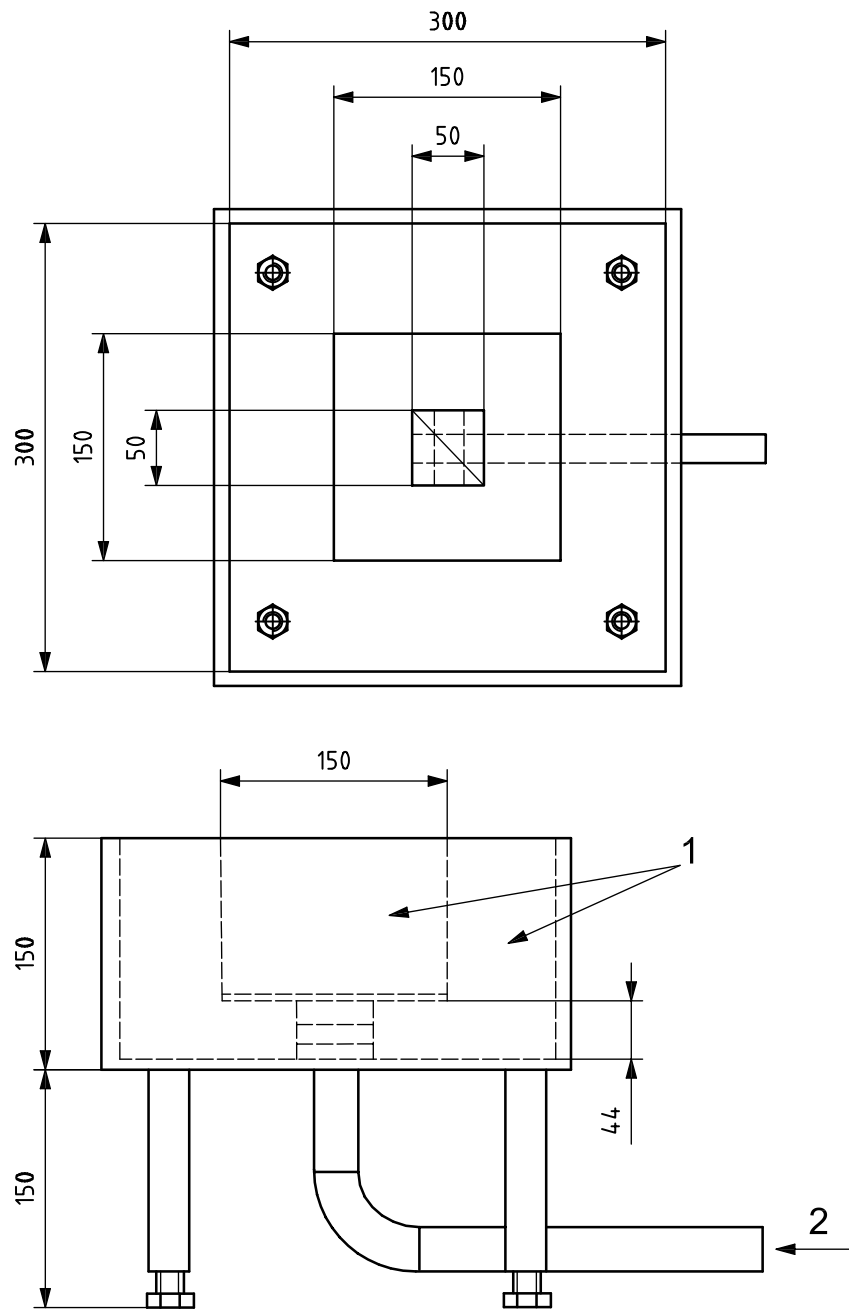
**Key**

- |                         |  |
|-------------------------|--|
| 1 bi-directional probe  | 5 gas sampling probe   |
| 2 laser extinction beam | 6 flow thermocouple  |
| 3 laser optics          | 7 flow direction   |
| 4 laser source          | 8 view of gas sampling probe (holes downstream to reduce clogging) |

**Figure 23 — Laser beam and other instrumentation in exhaust duct**



Dimensions in millimetres

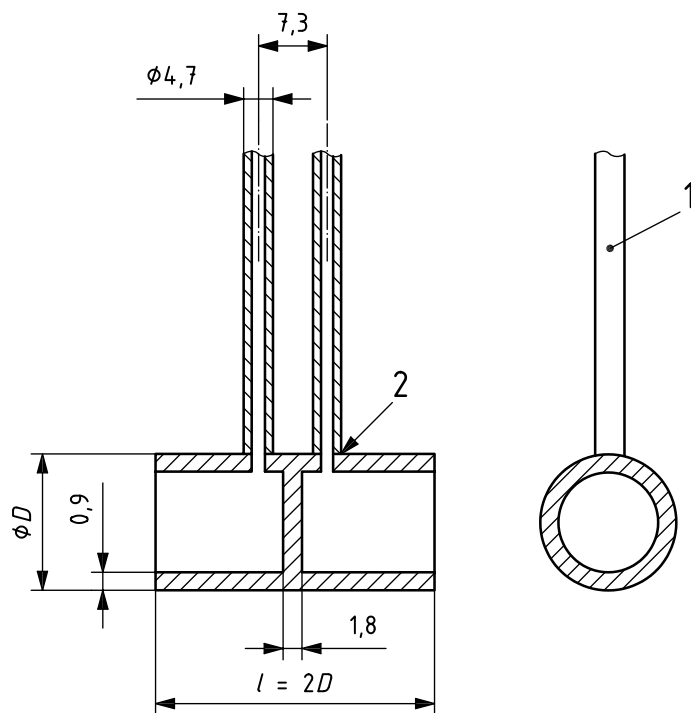


**Key**

- 1 space filled with silica sand
- 2 propane fuel

**Figure 24 — Sand burner**

Dimensions in millimetres

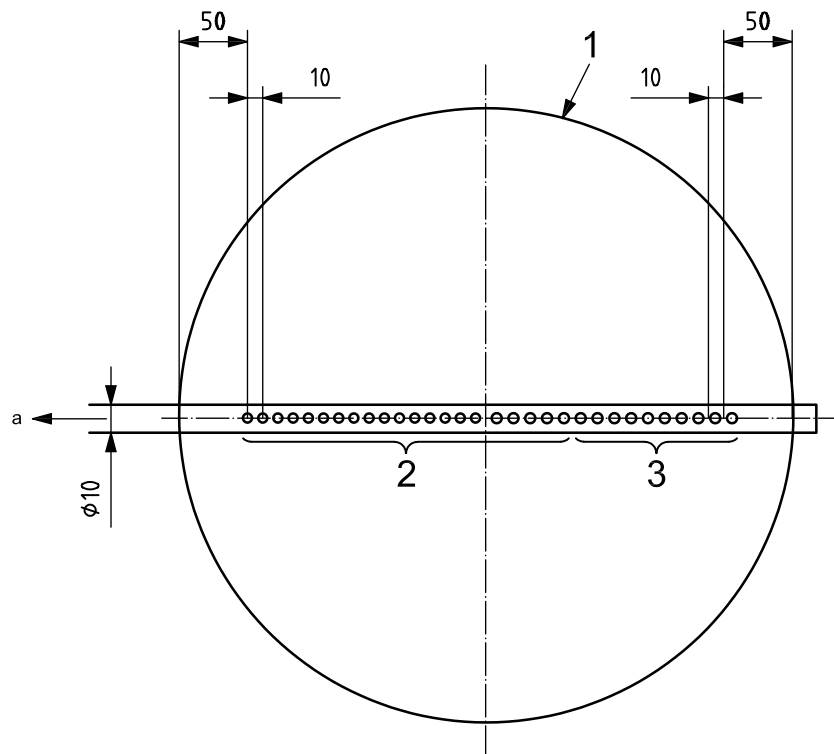


**Key**

- 1 variable-length support tube (to  $\Delta p$  instrument)
- 2 weld

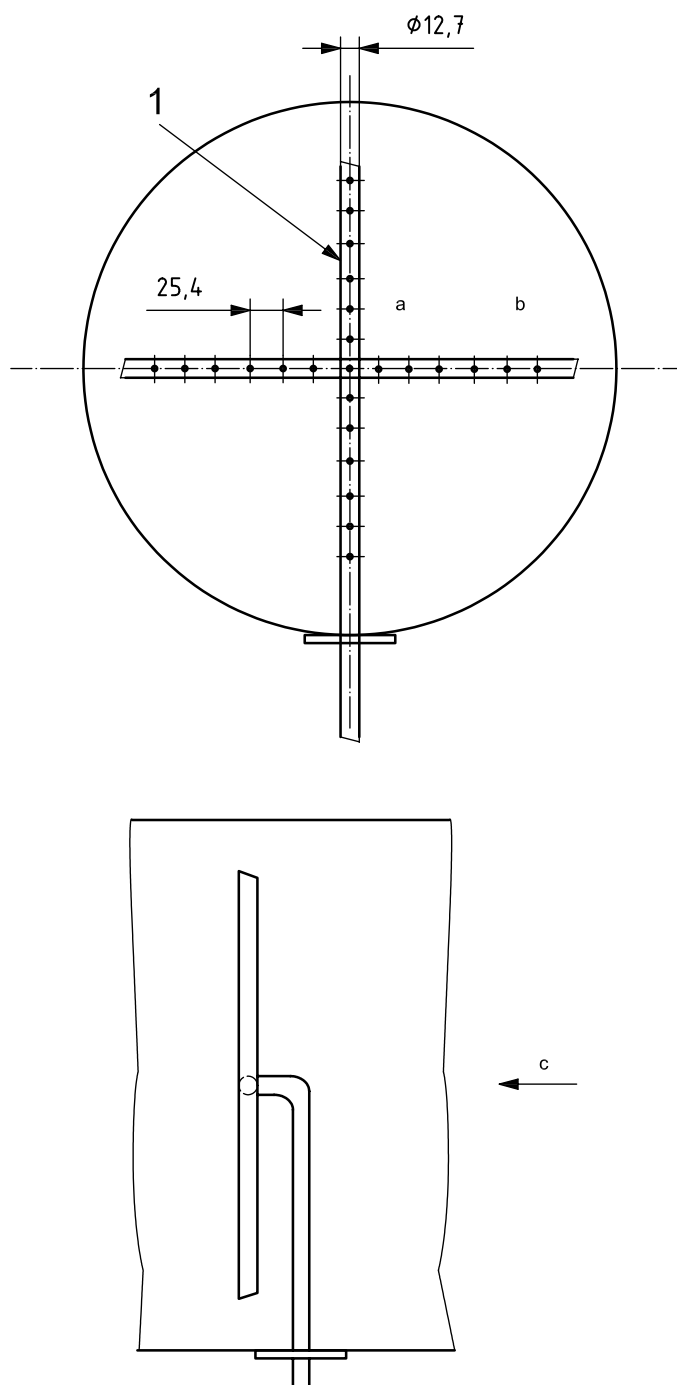
**Figure 25 — Bi-directional probe**

Dimensions in millimetres

**Key**

- 1 exhaust duct
- 2 2 mm holes on downstream side of flow (two thirds of the length of the pipe with holes)
- 3 3 mm holes on upstream side of flow (one third of the length of the pipe with holes)
- a Sample flow.

**Figure 26 — Horizontal gas sampling probe**

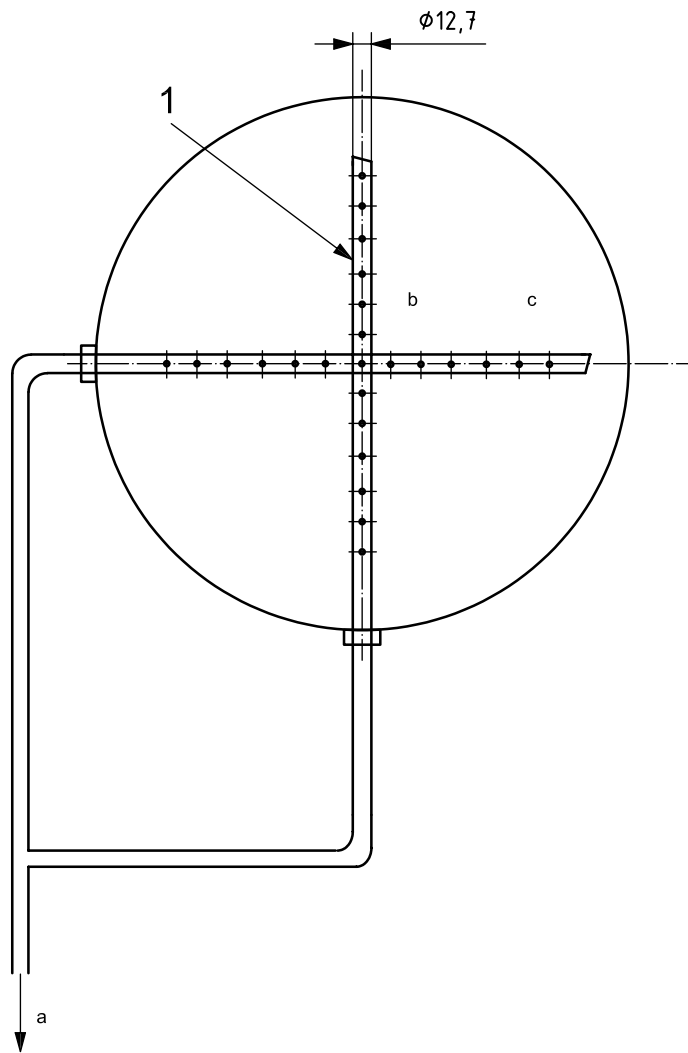


**Key**

- 1 SS tube
- a Small holes (centre, 9), 2,4 mm (3/32 in) diameter.
- b Large holes (outer, 16), 3,2 mm (1/8 in) diameter, distance between holes 25,4 mm (1 in).
- c Air flow.

**Figure 27 — Cross-type sampling probe**

Dimensions in millimetres

**Key**

- 1 SS tube
- a To filter and gas analysers.
- b Small holes (centre, 9), 2,4 mm (3/32 in) diameter.
- c Large holes (outer, 16), 3,2 mm (1/8 in) diameter, distance between holes 25,4 mm (1 in).

**Figure 28 — Cross-type gas sampling probe**

## Annex A (normative)

### Design of exhaust system

**A.1** Collect the combustion gases from the burning specimen by means of a hood. Described below, is an exhaust system (5.1.6), which has been tested in practice and proven to fulfil the specifications given in the method.

**A.2** The bottom dimensions of the hood of 3,1 m × 2,4 m have been found satisfactory, with a height of the hood itself of 1,0 m (see Figure 19). A vertical skirt on the hood will help ensure all the fire gases are collected at the least duct flow. The hood feeds into a plenum having a 0,9 m × 0,9 m cross-sectional area. The plenum has a height of 0,9 m. The maximum acceptable plenum height is 1,8 m, depending on building constraints. A system with different dimensions is acceptable, provided equivalence has been demonstrated. The hood shall not leak.

**A.3** In the plenum chamber, the incorporation of two plates approximately 0,5 m × 0,9 m in size (see Figure 19) increase mixing of the combustion gases. Alternative gas mixing methods can be used, if equivalence has been demonstrated.

**A.4** If a laser is used for smoke measurement, a means of mounting it together with the combustion gas sampling probes is shown in Figure 23.

**A.5** Connect an exhaust duct to the plenum chamber. The inner diameter of the exhaust duct is 0,4 m to 1,0 m. To facilitate flow measurements, guide vanes, if necessary, are located at both ends of the exhaust duct (Figure 19). Alternatively, the rectilinear part of the exhaust duct shall have such a length that a fully developed flow profile is established at the point of measurement. Connect the exhaust duct to an evacuation system.

**A.6** The evacuation system shall be designed so as to exhaust minimally all combustion gases leaving the specimen. This requires an exhaust capacity of at least 2,7 kg s<sup>-1</sup> (about 8 000 m<sup>3</sup>h<sup>-1</sup> at standard atmospheric conditions) corresponding to a driving under-pressure of about 2 kPa at the end of the duct. Provide a means to control the exhaust flow from about 0,5 kg s<sup>-1</sup> up to maximum flow as stated above during the test process. Ensure that the measurement system has sufficient sensitivity for measuring low rates of heat release. Mixing vanes in the duct are an adequate means of solving the problem if concentration gradients are found to exist.

**A.7** An alternative exhaust system design can be used if it is shown to produce equivalent results. Equivalency is demonstrated by conformance to the calibration requirements in Clause 8. Exhaust system designs based on natural convection are not recommended.

## Annex B (normative)

### Instrumentation in exhaust duct

#### B.1 Flow measurement

**B.1.1** One technique for measuring the flow is a bi-directional probe located at the centreline of the duct. The probe shown in Figure 25 consists of a stainless steel cylinder 44 mm long and with an inner diameter of 22 mm. The cylinder has a solid diaphragm in the centre, dividing it into two chambers. The pressure difference between the two chambers is measured by a differential pressure transducer.

**B.1.2** Use a differential pressure transducer with accuracy of at least  $\pm 0,25$  Pa and of the capacitance type. A suitable range of measurement is 0 Pa to 150 Pa.

**B.1.3** Place one thermocouple within 152 mm of the bi-directional probe. Use an Inconel sheathed thermocouple, type K Chromel-Alumel<sup>2)</sup>. The wire gauge shall be in the range 24 AWG (0,51 mm diameter) to 30 AWG (0,36 mm diameter). Place the thermocouple wire, within 13 mm of the bead, along expected isotherms to minimize conduction errors. Between the Chromel and Alumel wires use insulation that is stable to at least 1 100 °C. Ensure that the thermocouple does not disturb the flow pattern around the bi-directional probe.

#### B.2 Sampling line

**B.2.1** Locate the sampling probe in a position where the exhaust duct flow is uniformly mixed. Construct the probe with a cylindrical cross-section so as to minimize disturbance of the air flow in the duct. Collect the combustion gas samples across the entire diameter of the exhaust duct (see Figure 23, Figure 26, Figure 27 and Figure 28).

**B.2.2** Remove the particulates contained in the combustion gases with inert filters, to the degree required by the gas analysis equipment. Filter combustion gases in more than one step. Cool the combustion gas mixture to a maximum of 4 °C. The combustion gas samples taken to each analyser shall be completely dried.

**B.2.3** Transport the combustion gases using a pump. Use a pump which prevents gases from making contact with oil, grease or similar products, all of which can contaminate the gas mixture. A diaphragm pump (coated with polytetrafluoroethylene) is suitable. Alternative pumps shown to have the same effect are acceptable, but they have often been shown to need frequent replacement.

**B.2.4** Suitable sampling probes are shown in Figure 23, Figure 26, Figure 27 and Figure 28. These sampling probes are of the bar and cross type. Ring-type sampling probes are also acceptable, although they do not collect gas samples across the full diameter of the duct. Turn the intake of the sampling probe downstream to avoid soot clogging the probe.

**B.2.5** A suitable pump has a capacity of 10 L min<sup>-1</sup> kPa to 50 L min<sup>-1</sup> kPa (minimum), as gas analysis instruments consume about 1 L min<sup>-1</sup>. A pressure differential of at least 10 kPa, as generated by the pump, reduces the risk of smoke clogging the filters.

**B.2.6** Install a soot filter, capable of removing all particles greater than 25 µm in size.

**B.2.7** A refrigerated column is the most successful approach to cool and dry the gases. Provide a drain plug to remove the collected water from time to time. Alternative devices can be used.

**B.2.8** If carbon dioxide is to be removed, use carbon dioxide removal media, as indicated in Figure 20.

## B.3 Combustion gas analysis

### B.3.1 Oxygen concentration

#### B.3.1.1 Oxygen analyser output noise and drift

**B.3.1.2** Check the noise and drift of the oxygen analyser output as described in B.3.1.3 using the data acquisition system (5.1.8) after set-up, maintenance, repair or replacement of the oxygen analyser or other major components of the gas analysis system and at least every six months.

**B.3.1.3** The procedure for checking the noise and drift of the oxygen analyser output shall be as follows.

- a) Feed the oxygen analyser with oxygen-free nitrogen gas, until the analyser reaches equilibrium.
- b) After at least 10 min in oxygen-free conditions, adjust the volume flow in the exhaust duct and begin flowing gases through the gas train at the same flow rate, pressure and drying procedure as for sample gases. When the analyser reaches equilibrium, adjust the analyser output to  $(20,95 \pm 0,01) \%$ .
- c) Within 1 min, start recording the oxygen analyser output at 2 s intervals for a period of 30 min.
- d) Determine the drift using the least squares fitting procedure to fit a straight line through the data points. The absolute value of the difference between the readings at 0 min and at 30 min of this linear trend line represents the drift.
- e) Determine the noise by computing the root-mean-square (RMS) deviation around the linear trend line.

**B.3.1.4** The drift and noise (both taken as positive values) shall be not more than  $0,01 \%$  ( $V_{O_2}/V_{air}$ ).

### B.3.2 Carbon monoxide and dioxide concentration

Analysers found suitable are non-dispersive IR analysers.

### B.3.3 Time shift

Gas concentration measurements require the use of appropriate time shifts in order to account for gas transit time within the sampling system.

## B.4 Smoke obscuration

### B.4.1 White light system

**B.4.1.1** A suitable light-measuring system based on white light has the following components: a lamp, plano convex lenses, an aperture, a photocell and an appropriate power supply. Mount lenses, lamp and photocell inside two housings, located on the exhaust duct, diametrically opposite each other. A system consisting solely of a white light and a photocell, along the exhaust duct, across from each other and at an angle to the vertical, can be used in some cases.

**B.4.1.2** Use a lamp of the incandescent filament type, which operates at a colour temperature of  $(2\,900 \pm 100)$  K. Supply the lamp with stabilized direct current, stable within  $\pm 0,2 \%$  (including temperature and short-term and long-term stability). Centre the resultant light beam on the photocell.

**B.4.1.3** Select the lens system so that the lens L2, according to Figure 21, has a diameter,  $d$ , chosen with regard to the focal length,  $f$ , of L2 so that  $d/f \geq 0,04$ .

**B.4.1.4** Place the aperture in the focus of lens L2 as shown in Figure 21.



**B.4.1.5** Use a detector with a spectrally distributed response approximating that of the CIE (Commission Internationale d'Éclairage) photopic curve and which is linear within 5 % over an output range of at least 3,5 decades. Check this linearity over the entire range of the instrument periodically with calibrated optical filters.

**B.4.1.6** The system described in ISO 9705 is an example of a light-measuring system that can be used.

**B.4.1.7** Design a system that is easily purged of soot deposits. The use of holes in the periphery of the two housings is a means of achieving this objective.

#### **B.4.2 Laser system**

An alternative system for measurements of smoke obscuration uses a laser beam. A 0,5 mW to 2,0 mW helium-neon laser beam is projected across the exhaust duct. Couple the two halves of the device rigidly together (see Figure 22).

## Annex C (informative)

### Considerations for heat release measurements

#### C.1 Measurement of heat release rate by oxygen consumption

**C.1.1** In 1917, Thornton <sup>[1]</sup> showed that for a large number of organic fuels, a more or less constant net amount of heat is released per unit of oxygen consumed for complete combustion. Huggett <sup>[2]</sup> obtained an average value for this constant of 13,1 MJ/kg of oxygen. This value may be used for practical applications and is accurate, with very few exceptions, to within  $\pm 5\%$ .

**C.1.2** Thornton's rule indicates that it is sufficient to measure the oxygen consumed in a combustion system in order to determine the net heat released. This is particularly useful for full-scale fire test applications. For example, for compartment fires, the oxygen consumption technique is much more accurate and easier to implement than methods based on measuring all the terms in a heat balance of the compartment.

**C.1.3** The first application of the oxygen consumption principle in fire research was by Parker <sup>[3]</sup> using the ASTM E84 <sup>[15]</sup> tunnel test. Later, Sensenig applied it to an intermediate-scale room test <sup>[4]</sup>. During the late seventies and early eighties, the O<sub>2</sub> consumption technique was refined at the National Institute of Standards and Technology (NIST, formerly the National Bureau of Standards). A paper by Parker <sup>[5]</sup> gives equations to calculate heat release rate by oxygen consumption for various applications. The technique is now used extensively in many laboratories all over the world, both in bench-scale <sup>[6]</sup> and full-scale <sup>[7]</sup>, <sup>[8]</sup> fire test applications.

**C.1.4** The objective of this annex is to provide a comprehensive set of equations and guidelines to determine the heat release rate in ICAL fire tests based on the oxygen consumption principle. The approach followed here is somewhat different from Parker <sup>[5]</sup>, as the emphasis is on intermediate-scale fire test applications and the use of volumetric flows is avoided. Volumetric flows require specification of temperature and pressure. Various investigators have used different combinations of reference pressure and temperature. This leads to confusion, which is greatly minimized if mass flows are used.

**C.1.5** The basic requirement is that all combustion products be collected in a hood and removed through an exhaust duct. At a distance downstream of the hood sufficient for adequate mixing, both flow and composition of the combustion gases are measured. It is assumed here that it is not possible to measure the air flow into the system, as this is generally the case for full-scale fire tests. The differences in treatment and equations to be used are mainly due to the extent to which combustion gas analysis is made. At least oxygen shall be measured. However, heat release rate measurements will be more accurate by measuring CO<sub>2</sub> and CO additionally.

**C.1.6** It should be emphasized that the analysis is approximate. The following describes the main simplifying assumptions made:

- a) The amount of energy released by complete combustion per unit of oxygen consumed is taken as:  
 $E = 13,1$  MJ/kg of oxygen.
- b) All combustion gases are considered to behave as ideal gases, in other words one mole of any gas is assumed to occupy a constant volume at the same pressure and temperature.
- c) Incoming air consists of O<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and N<sub>2</sub>. All inert gases, which do not take part in the combustion reaction, are lumped into the nitrogen.
- d) O<sub>2</sub>, CO<sub>2</sub>, and CO are measured on a dry basis, i.e. water vapour is removed from the specimen before combustion gas analysis measurements are made.

**C.1.7** In the analysis to follow, initial emphasis will be placed on the flow measurement. Equations used to calculate flow are applicable, unless otherwise indicated, irrespective of the configuration of the combustion gas analysis system. In subsequent subclauses, distinction is made between various combustion gas analyser combinations.

## C.2 Flow measurements

**C.2.1** The mass flow rate through the duct is obtained from the velocity measured with a bi-directional probe at one point in the duct, usually along the centreline. The flow is then calculated using a predetermined shape of the velocity profile in the duct. The latter is obtained by measuring velocity at a sufficient number of representative points over the diameter or cross-section of the duct prior to any fire tests. Detailed procedures to obtain this profile are described by ASME [8] and in Ower and Pankhurst [9]. Usually, conditions in intermediate-scale fire tests are such that the flow in the duct is turbulent, resulting in a shape factor  $k_c$  (equal to the ratio of the average velocity to the velocity along the centreline) close to unity.

**C.2.2** Due to considerable soot production in many fires, pitot static tubes cannot be used because of the potential for clogging of the holes. In order to deal with this problem, a more robust bi-directional probe was designed by McCaffrey and Heskestad [10]. This involves measuring the differential pressure across the probe and the centreline velocity, and is valid in the range of Reynolds numbers,  $Re$ :

$$40 < Re < 3\,800.$$

In many intermediate-scale fire test applications, duct diameter and flow are such that the Reynolds number is:

$$Re > 3\,800.$$

In this case  $f_{(Re)}$  is taken as a constant (1,08), which greatly simplifies the calculations. This calculation [Equation (D.1)] is preferred for intermediate-scale measurements of heat release rate. Further details of this and of all other calculations discussed in this Annex are found in a paper by Janssens [11]. For additional details, see also ISO 9705.

## C.3 Heat release rate measurement if only oxygen is measured

**C.3.1** In this case, all water vapour and  $CO_2$  are eliminated by the use of appropriate filtering media. This leads to the assumption that the specimen combustion gas only consists of  $O_2$  and  $N_2$ . This is approximately true provided  $CO$  production is negligible, which is usually the case owing to the abundant availability of oxygen. As the composition of the incoming air is unlikely to change during a test, and as the temperatures in building fires are usually not high enough to generate noticeable amounts of nitrogen oxides by nitrogen fixation, the mole fraction of oxygen in the air as measured by the analyser prior to a test can be written on the basis of  $O_2$  and  $N_2$  exclusively. The mole fraction of oxygen in the exhaust combustion gases, as measured by the oxygen analyser, can be written likewise. As nitrogen is conserved and does not participate in the combustion reactions, the equations are derived on the basis of its conservation.

**C.3.2** In this case the heat release rate, in kW, is calculated as a function of the heat released per unit of oxygen consumed ( $E$ , 13,1 MJ/kg of  $O_2$ ), the ratio of the relative molecular mass of oxygen ( $M_{O_2}$ , 32,0 kg/kmol) and relative molecular mass of the incoming air ( $M_a$ , generally taken as 28,97 kg/kmol) and the mass flow of the incoming air, in kg/s. The flow measured is that of the smoke within the exhaust duct and not that of the incoming air. In order to find a relation between the two, it is necessary to define the oxygen depletion factor. The oxygen depletion factor is the fraction of the incoming air which is fully depleted of its oxygen [Equation (D.4)]. It has been demonstrated (see Annex of ASTM E1354 [16]) that the heat release rate is a function of  $E$ ,  $M_{O_2}$ ,  $M_a$  and the oxygen depletion factor, plus the expansion factor.

The expansion factor has to be assigned and a recommended value is 1,105, the value for methane. The value for propane is 1,084, carbon in dry air is 1,0 and hydrogen is 1,21.

**C.3.3** The resulting equation, Equation (D.4), is expected to be accurate to within  $\pm 5\%$  provided combustion is complete and all carbon is converted to  $\text{CO}_2$ . Errors will be greater if CO or soot production is considerable or if a significant amount of the combustion products are other than  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . It is unlikely that these errors will be of concern for the ICAL tests since oxygen is not limited.

#### **C.4 Heat release rate measurement if oxygen and carbon dioxide are being measured**

This case is similar to that covered in C.3. It is now assumed that only water vapour is trapped before the specimen reaches the combustion gas analysers. The equations are derived on the basis of conservation of  $\text{N}_2$ . The mole fraction of  $\text{CO}_2$  in the incoming air is taken to be 440 ppm. A new equation is now necessary, of course, for the oxygen depletion factor, Equation (D.5). The equation for heat release rate [Equation (D.3)] is accurate to within  $\pm 5\%$  provided combustion is complete and all carbon is converted to  $\text{CO}_2$ .

#### **C.5 Heat release rate measurement if oxygen, carbon dioxide and carbon monoxide are being measured**

This case reverts to that covered in C.4 if CO production is negligible. Taking CO into account, however, changes the equations. It means that a new oxygen depletion factor is required, Equation (D.6), as well as a new heat release rate equation altogether, Equation (D.7).

#### **C.6 Conclusions**

**C.6.1** Depending on the configuration of combustion gas analysers and the type of flow measurement, one of the following procedures should be used to calculate heat release rate.

**C.6.2** Case 1: Only  $\text{O}_2$  is measured.

**C.6.2.1** Calculate the mass flow of the exhaust combustion bases.

**C.6.2.2** Calculate the oxygen depletion factor.

**C.6.2.3** Calculate the heat release rate.

**C.6.3** Case 2: Both  $\text{O}_2$  and  $\text{CO}_2$  are measured.

**C.6.3.1** Calculate the mass flow of the exhaust combustion gases as in C.6.2.

**C.6.3.2** Calculate the new oxygen depletion factor.

**C.6.3.3** Calculate the new heat release rate.

**C.6.4** Case 3:  $\text{O}_2$  and  $\text{CO}_2$  and CO are measured.

**C.6.4.1** Calculate the mass flow of the exhaust combustion gases as in C.6.2.

**C.6.4.2** Calculate the new oxygen depletion factor.

**C.6.4.3** Calculate the new heat release rate.

**C.6.5** The following numerical values are recommended for use in the equations.

a) Energies:

- $E = 13,1$  MJ/kg of  $O_2$ ,
- $E_{CO} = 17,6$  MJ/kg of  $O_2$ ,
- $E_{\text{propane}} = 12,73$  MJ/kg of  $O_2$ .

b) Relative molecular mass:

- $M_a = 29$  kg/kmol (cases 1, 2 and 3),
- $M_{CO} = 28$  kg/kmol,
- $M_{CO_2} = 44$  kg/kmol,
- $M_{\text{dry}} = 29$  kg/kmol,
- $M_e = 29$  kg/kmol (cases 1, 2 and 3),
- $M_{H_2O} = 18$  kg/kmol,
- $M_{N_2} = 28$  kg/kmol,
- $M_{O_2} = 32$  kg/kmol.

c) Expansion factor = 1,105 (cases 1, 2 and 3).

**C.6.6** If a  $CO_2$  analyser is used, this eliminates the need for removal of  $CO_2$  from the combustion gas specimen. This is mainly of practical importance as the scrubbing agent used to remove  $CO_2$  usually requires careful handling and is rather expensive. If a significant amount of CO is produced (for example at or beyond flashover in ventilation-controlled room fires), the accuracy of the heat release rate measurement is improved if CO is measured.

**C.6.7** The presence of a water vapour analyser simplifies the analysis and improves accuracy even more. Unfortunately, implementation of a water vapour analyser is not straightforward because sampling lines, filters, etc. shall be heated to avoid condensation. Thus, the use of a water vapour analyser precludes the need to estimate the expansion factor.

## Annex D (normative)

### Measurement equations

#### D.1 Heat release rate

**D.1.1** The equation used to calculate the mass flow rate,  $\dot{m}_e$ , is Equation (D.1):

$$\dot{m}_e = 26,54 \cdot \frac{A \cdot k_c}{f_{(Re)}} \cdot \sqrt{\left(\frac{\Delta p}{T_e}\right)} \quad (D.1)$$

The function of the Reynolds number correction,  $f_{(Re)}$ , is taken to be 1,08 as described in C.2.

**D.1.2** In Case 1, only oxygen is measured.

Calculate the mass flow using Equation (D.1) and the oxygen depletion factor,  $\phi$ , using Equation (D.2):

$$\phi = \frac{X_{O_2,i} - X_{O_2,e}}{(1 - X_{O_2,e}) X_{O_2,i}} \quad (D.2)$$

Then, calculate the heat release rate,  $\dot{q}$ , using Equation (D.3):

$$\dot{q} = E \frac{M_{O_2}}{M_a} \frac{\phi}{1 + \phi(\alpha - 1)} \dot{m}_e X_{O_2,i} \quad (D.3)$$

If only oxygen is measured, Equation (D.3) simplifies to Equation (D.4):

$$\dot{q} = E \cdot 1\,010 \cdot C \sqrt{\left(\frac{\Delta p}{T_e}\right)} \cdot \left(\frac{X_{O_2,i} - X_{O_2,e}}{1,105 - 1,5 X_{O_2,e}}\right) \quad (D.4)$$

**D.1.3** In Case 2, only O<sub>2</sub> and CO<sub>2</sub> are measured.

Calculate the mass flow,  $\dot{m}_e$ , using Equation (D.1) and the oxygen depletion factor,  $\phi$ , using Equation (D.5):

$$\phi = \frac{X_{O_2,i}(1 - X_{CO_2,e}) - X_{O_2,e}(1 - X_{CO_2,i})}{X_{O_2,i}(1 - X_{O_2,e} - X_{CO_2,e})} \quad (D.5)$$

Calculate the heat release rate using Equation (D.3).

**D.1.4** In Case 3, O<sub>2</sub> and CO<sub>2</sub> and CO are measured.

Calculate the mass flow,  $\dot{m}_e$ , using Equation (D.1), and the oxygen depletion factor,  $\phi$ , using Equation (D.6):

$$\phi = \frac{X_{O_2,i}(1 - X_{CO_2,e} - X_{CO,e}) - X_{O_2,e}(1 - X_{CO_2,i})}{X_{O_2,i}(1 - X_{O_2,e} - X_{CO_2,e} - X_{CO,e})} \quad (D.6)$$

Calculate the heat release rate,  $\dot{q}$ , using Equation (D.7):

$$\dot{q} = \left[ E\phi - (E_{\text{CO}} - E) \frac{1-\phi}{2} \frac{X_{\text{CO}_2,\text{e}}}{X_{\text{O}_2,\text{e}}} \right] \cdot \frac{M_{\text{O}_2}}{M_{\text{a}}} \cdot \frac{\dot{m}_{\text{e}}}{1+\phi \cdot (\alpha-1)} \cdot X_{\text{O}_2,\text{i}} \quad (\text{D.7})$$

Finally, calculate the specimen heat release rate,  $\dot{q}_{\text{s}}''$ , per unit area using Equation (D.8):

$$\dot{q}_{\text{s}}'' = \left[ \dot{q} - (\Delta H_{\text{c,ng}} \cdot \dot{m}_{\text{ng}} \cdot 1,05) \right] / A_{\text{s}} \quad (\text{D.8})$$

NOTE A correct factor [1,05 used in Equation (D.8)] should be calculated for the natural gas based on its composition.

## D.2 Total heat release equations

Determine the total specimen heat released per unit area during combustion,  $q_{\text{s}}''$ , expressed in kW/m<sup>2</sup>, by summation, using Equation (D.9):

$$q_{\text{s}}'' = \sum_i q_{\text{s},i}''(t) \Delta t \quad (\text{D.9})$$

where the summation begins with the first reading after exposure and continues until the final reading of the test.

## D.3 Smoke measurement equations

The relative optical density,  $F_{\text{OD}}$ , is calculated using Equation (D.10):

$$F_{\text{OD}} = \log(I_0 / I) \quad (\text{D.10})$$

Extinction coefficient,  $k$ , is calculated using Equation (D.11):

$$k = (1/L_p) \ln(I_0 / I) \quad (\text{D.11})$$

The volumetric flow is calculated as the product of the mass flow and the density of air, at the corresponding temperature. Thus, both the volumetric flow and the density of air shall undergo temperature corrections. The volumetric duct flow,  $\dot{V}$ , is adjusted because it is measured in the exhaust duct, but required at the temperature near the photodetector, as shown in Equation (D.12):

$$\dot{V}_{\text{s}} = V_{\text{e}} \cdot \left( \frac{T_{\text{s}}}{T_{\text{e}}} \right) \quad (\text{D.12})$$

The density of air is adjusted between the literature value, measured at 273,15 K, and the value at the temperature in the exhaust duct, as shown in Equation (D.13):

$$\rho = \rho_0 \cdot \frac{273,15}{T_{\text{e}}} \quad (\text{D.13})$$

Then, the final equation for the volumetric flow is Equation (D.14) as follows:

$$\dot{V}_{\text{s}} = \frac{\dot{m}_{\text{e}}}{\rho_0} \cdot \frac{T_{\text{e}}}{273,15} \quad (\text{D.14})$$

The instantaneous rate of production of light-obscuring smoke,  $R_{\text{inst}}$ , is defined using Equation (D.15):

$$R_{\text{inst}} = (\dot{V}_s k) \quad (\text{D.15})$$

The total amount of smoke released,  $R_{\text{tot}}$ , is found using Equation (D.16):

$$R_{\text{tot}} = \int_0^t k \dot{V}_s dt \quad (\text{D.16})$$

#### D.4 Mass loss rate

Compute the required mass loss rate,  $-dm/dt$ , at each time interval using five-point numerical differentiation. The equations to be used are Equations (D.17), (D.18), (D.19), (D.20) and (D.21) as follows:

a) For the first scan ( $i = 0$ ) use Equation (D.17):

$$-\left(\frac{dm}{dt}\right)_{i=0} = \frac{25m_0 - 48m_1 + 36m_2 - 16m_3 + 3m_4}{12\Delta t} \quad (\text{D.17})$$

b) For the second scan ( $i = 1$ ) use Equation (D.18):

$$-\left(\frac{dm}{dt}\right)_{i=1} = \frac{3m_0 + 10m_1 - 18m_2 + 6m_3 - m_4}{12\Delta t} \quad (\text{D.18})$$

c) For any scan for which  $1 < i < n - 1$  (where  $n =$  total number of scans) use Equation (D.19):

$$-\left(\frac{dm}{dt}\right)_i = \frac{-m_{i-2} + 8m_{i-1} - 8m_{i+1} + m_{i+2}}{12\Delta t} \quad (\text{D.19})$$

d) For the next to last scan ( $i = n - 1$ ) use Equation (D.20):

$$-\left(\frac{dm}{dt}\right)_{i=n-1} = \frac{-10m_n - 3m_{n-1} + 18m_{n-2} - 6m_{n-3} + m_{n-4}}{12\Delta t} \quad (\text{D.20})$$

e) For the last scan ( $i = n$ ) use Equation (D.21):

$$-\left(\frac{dm}{dt}\right)_{i=n} = \frac{-25m_n + 48m_{n-1} - 36m_{n-2} + 16m_{n-3} - 3m_{n-4}}{12\Delta t} \quad (\text{D.21})$$



## Annex E (informative)

### Commentary

#### E.1 Introduction

**E.1.1** Heat release rate is the most important fire quantity associated with a burning object, room or building. It describes the size of the fire at any time during a specific fire scenario.

**E.1.2** Bench-scale test methods like the cone calorimeter (ASTM E1354<sup>[16]</sup>) and the Ohio State University rate of heat release apparatus (ASTM E906<sup>[17]</sup>) have been used for testing materials and to some extent sandwich panels and composites. The resulting heat release rate (RHR) data have mainly been used in computer fire models. Classifications of building materials and products have also been proposed on the basis of heat release data from the cone calorimeter alone or in combination with a large-scale fire test.

**E.1.3** However, some materials, and especially assemblies, cannot be successfully tested on a bench-scale apparatus for several reasons. One of the most important limitations is the composition of assemblies. The very small samples used in the bench-scale apparatus cannot adequately represent an assembly. Also other features, such as joints of assembly panels, sheets of non-combustible protective materials, cracking, material collapse, melting and spilling of thermoplastic materials, cannot be represented in a bench-scale sample. Neither can these features be mathematically modelled.

**E.1.4** Large-scale tests such as a standard room fire test have been successfully used for heat release measurements of assemblies. Attempts to measure heat release from fire endurance furnaces have not been successful (unpublished). These tests are expensive and time consuming. The ICAL test method overcomes most of the problems identified with large and bench-scale testing.

#### E.2 Factors affecting radiant panel assembly performance

**E.2.1** Because the surface temperature of the specimen may change during a test, the surface temperature and therefore irradiance from the radiant panel may also change. In order to maintain a constant irradiance, the flow of gas to the radiant panel should be adjusted to compensate for the re-radiation from the sample.

**E.2.2** Experience in operating the radiant panel in a relatively small laboratory shows that high heat flow from the radiant panel may increase the temperature of the room and gas plumbing while the radiant panel is operating. This increase in the plumbing temperature will cause the gas temperature and flow to increase also. Therefore, the heat produced by the radiant panel will also change. Such changes may influence the results of the tests of longer duration.

#### E.3 Ignition source

**E.3.1** Different ignition sources were examined for use in the ICAL. The desired ignition system should ignite the specimen surface instantaneously as it reaches the ignition temperature. A line pilot burner was positioned near the top of the specimen as a first attempt. Ignition by this mode did not provide for uniform ignition over the whole surface especially at irradiances greater than 30 kW/m<sup>2</sup>. Rather, the flaming ignition was isolated to the burning of the pyrolysis gases above the specimen and did not spread downward over the surface.

**E.3.2** When the pilot line burner was positioned near the bottom of the specimen, it caused localized heating and premature ignition. The rest of the specimen had not reached the ignition temperature which resulted in upward flame spread.

**E.3.3** Spark igniters are used extensively when gas phase ignition is desired. This approach was not suitable for the ICAL since the width of the sample was quite large and would have required many spark sources near the bottom edge. Even more spark igniters would be required at the top of the specimen for certain assemblies which generate pyrolysis gases near the top of the specimen only.

**E.3.4** Hot wire igniters at the top and bottom of the specimen were determined to be the best solution to the problems mentioned above. The hot wires have performed well in all the tests to date on several different materials and assemblies.

## **E.4 Factors affecting heat flux exposure range**

Heat flux exposure for the heat release measurements at the low flux levels are limited by the exhaust hood size. Heat flux values between 25 kW/m<sup>2</sup> and 50 kW/m<sup>2</sup> are achieved at distances less than 1 m. Heat fluxes higher than 50 kW/m<sup>2</sup> require small separations between the radiant panel and specimen which can cause convective heat transfer between the radiant panel and specimen by interference of the radiant panel boundary layer with the specimen boundary layer [16].

## **E.5 Air-stream-interrupting projection plate**

Ignition in the ICAL at lower flux exposures occurs at much longer times and much higher surface temperatures than in small-scale apparatus. It seems reasonable to assume that the difference may be due to the high upward air velocity induced by the heated large-area specimen. One of the ignition criteria that shall be met is that the downward flame spread velocity relative to the moving air (i.e. the velocity with no air movement) shall be equal to or greater than the upward air velocity. Otherwise the flame will “blow off” and not attach to the specimen. In order to reduce the upward air velocity near the bottom of the specimen a 100 mm horizontal projection plate is placed along the lowest exposed portion so that the air flowing upward would have to turn a sharp corner and thus create an eddy there. This technique works well for wood materials and is therefore incorporated into the test method.

## **E.6 Radiant panel ignition safety system**

Ignition of the burners can be accomplished by individual, automatic spark igniters and pilot flames. The spark igniters are used to ignite the pilot flames on each end of each row of burners, which in turn are used to ignite the burners. The natural gas flow to the burners is released through the safety interlock only when optical sensors on one side of the burner rows can see the pilot flames. The pilot flames remain on while natural gas is flowing to the burners and are not to be turned off until the burners are extinguished.

## Annex F (informative)

### Measurement and determination of other parameters and values needed in computer fire models

#### F.1 Introduction

The ICAL is also used to determine many of the parameters or values needed in computer fire models. Examples of these values include effective heat of combustion, surface temperature, ignition temperature, temperature gradients in the specimen and combustion gas yields.

#### F.2 Effective heat of combustion

##### F.2.1 General

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 exhibit more than one mode of degradation and a varying effective heat of combustion. For materials having more than one mode of degradation, or for composites or non-homogeneous materials, the effective heat of combustion is not necessarily constant. Effective heat of combustion can be used to provide additional information on the fire behaviour of materials.

##### F.2.2 Symbols

$\Delta h_{c,eff}$  effective net heat of combustion, expressed in megajoules per kilogram, MJ/kg;

$m_f$  final specimen mass, expressed in kilograms;

$m_i$  initial specimen mass, expressed in kilograms.

##### F.2.3 Calculation

The mass loss rate,  $-dm/dt$ , computed for each time interval starting from time of ignition, can be used to determine a time-varying value of the effective heat of combustion, given by Equation (F.1):

$$\Delta h_{c,eff}(t) = \frac{q_i(t)}{-(dm/dt)} \quad (F.1)$$

As the mass loss rate requires numerical differentiation for its determination and is, therefore, noisier than measurements obtained directly from instrument readings, it is better to calculate average values of  $\Delta h_{c,eff}$ . To obtain such averages the numerator and denominator in Equation (F.1) shall be averaged separately rather than calculating the average of the ratio. For instance  $\Delta h_{c,eff}$  averaged over the whole test is obtained using Equation (F.2):

$$\Delta h_{c,eff} = \frac{\sum_i q_i(t)\Delta t}{m_i - m_f} \quad (F.2)$$

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

### F.3 Sample ignition temperature

The ignition temperature can be measured with the IR pyrometer which is more fully described by Urbas and Parker [12].

The pyrometer<sup>6)</sup> views the specimen surface through the 33 mm gap in the radiant panel. The pyrometer is positioned behind the radiant panel assembly (5.1.1) at a height appropriate to view the geometrical centre of the specimen.

### F.4 Surface temperature during burning

The IR pyrometer used for the ignition temperature measurements can also be used to follow the surface temperature during burning [12]. A wavelength band of 8 µm to 10 µm is chosen to minimize the effects of water vapour, carbon dioxide and soot radiation and to take advantage of the high spectral emissivity of the surface.

### F.5 Sample temperature measurements

Heat flow through a specimen can be measured with thermocouples positioned at different locations through the specimen.

All thermocouples shall be 0,127 mm [or 0,005 in or 5 mils<sup>7)</sup>] Type K, Chromel-Alumel<sup>2)</sup>.

The location of the minimum number of thermocouples for measurement of homogenous sample temperature shall be at the geometric centre of the sample and on the centreline through the sample as follows: surface, 1/4 depth, 1/2 depth, 3/4 depth and unexposed surface. Other temperatures may be taken as necessary to gain specific information such as interface temperatures between assemblies of materials.

The interior thermocouples shall be inserted in holes which have been pre-drilled from the unexposed face of the sample toward the exposed face to the desired depth. These thermocouples shall be sheathed with ceramic insulation. The two wires leading up to the junctions of surface thermocouples shall be bared for a distance of at least 20 mm on both sides of the junction. Each lead shall be pulled tight so that the bead is contacting the surface and stapled at a point on each wire 10 mm away from the junction. The bead shall be pushed, using the thumb, with moderate force into the surface if it will penetrate.

### F.6 Gas yield calculations

Gas yields can be calculated for carbon dioxide, carbon monoxide and any other measured combustion gases following the procedure described below.

Calculate the specimen carbon monoxide, carbon dioxide and other (if any) measured combustion gas concentrations by subtracting the radiant panel contributions (baseline data) from the total values.

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6) A Heitronics Infrared Pyrometer, Model KT19.81 having a temperature range from 0 °C to 1 000 °C and a wavelength band of 8 µm to 10 µm can be used for this application. This information is given for the convenience of users of ISO 14696 and does not constitute an endorsement by ISO of this product.

7) One milli-inch is equivalent to  $2,54 \times 10^{-5}$  m. Mils is deprecated.

Calculate the gas yields,  $f$ , using Equation (F.3):

$$f_x = \frac{\int_0^t \dot{m}_{e(x)} dt}{\Delta m} \quad (\text{F.3})$$

In Equation (F.3),  $\dot{m}_{e(x)}$  is the mass flow of gas  $x$ , expressed in grams per second, and  $\Delta m$  is mass loss of specimen over the period over which the gas yield is calculated, and is expressed in grams. The numerator in this equation is calculated as shown in Equation (F.4):

$$f_x = \frac{\left\{ \sum_i [x]_i \cdot \dot{m}_{e(i)} \cdot \Delta t_i \right\} \cdot 0,001 \cdot \frac{M_x}{M_a}}{\Delta m} \quad (\text{F.4})$$

In this equation,  $[x]$ , the concentration of gas  $x$ , is expressed in kg/kg,  $\dot{m}_{e(i)}$ , the mass flow, calculated using Equation (D.1), is expressed in kilograms per second,  $M_x$  is the relative molecular mass of gas species  $x$ ,  $\Delta t$  is the scan period, and mass loss of specimen is the mass lost over the period over which the gas yield is calculated, and is expressed in grams, with 0,001 as a conversion factor, to account for the unit changes.

Gas concentrations are reported on a dry gas basis, unless water concentrations are being measured.

Use ASTM E800<sup>[14]</sup> for details of suitable analysers when the concentrations of other combustion gases, such as water, total hydrocarbon, nitrogen oxide, hydrogen cyanide, or hydrogen chloride, are to be measured for special purposes.

## Annex G (informative)

### Determination of the precision and bias of the test method

#### G.1 General

An inter-laboratory trial was conducted using the protocol described in this International Standard<sup>8)</sup>. The materials tested were:

- 11 mm oriented strandboard ( $\rho = 685 \text{ kg/m}^3$ );
- 19 mm fire-retardant-treated plywood ( $\rho = 640 \text{ kg/m}^3$ );
- 3 mm fibre reinforced plastic ( $\rho = 1\,100 \text{ kg/m}^3$ );
- 51 mm paper-faced rigid polyisocyanurate foam ( $\rho = 58 \text{ kg/m}^3$ ).

Three replicates of each material were tested at two irradiance levels ( $25 \text{ kW/m}^2$  and  $40 \text{ kW/m}^2$ ) by four laboratories.

#### G.2 Precision and bias of the test method

The repeatability standard deviation,  $s_r$ , and reproducibility standard deviation,  $s_R$ , were calculated in accordance with ISO 5725-2 for five variables. These values include the test results identified as “stragglers” and exclude test results by laboratories identified as “outliers” determined by the procedures described in ISO 5725-2. The five variables were chosen as being representative of the test results:  $t_{ig}$ ,  $\dot{q}''_{peak}$ ,  $\dot{q}''_{A,60}$ ,  $\dot{q}''_{A,180}$ , and  $q_s''$ . In addition, for comparison with other apparatuses, the precision of which was evaluated in accordance with ISO 5725:1986<sup>9)</sup>, repeatability ( $r$ ) and reproducibility ( $R$ ) were calculated by multiplying  $s_r$  and  $s_R$  by 2,8. A linear regression model described in ISO 5725-2 was used to describe repeatability ( $r$ ) and reproducibility ( $R$ ) as a function of the mean over all replicates and over all laboratories for each of the five variables mentioned. The following are the regression equations. The range of mean values over which the fit was obtained is also indicated.

The results for  $t_{ig}$  in the range of 7 s to 160 s are given as Equations (G.1) and (G.2):

$$r = 18,83 + 0,183t_{ig} \quad (\text{G.1})$$

$$R = 23,79 + 0,784t_{ig} \quad (\text{G.2})$$

The results for  $\dot{q}''_{peak}$  in the range of  $91 \text{ kW/m}^2$  to  $342 \text{ kW/m}^2$  are given as Equations (G.3) and (G.4):

$$r = 43,81 + 0,232 \dot{q}''_{peak} \quad (\text{G.3})$$

$$R = -1,852 + 0,870 \dot{q}''_{peak} \quad (\text{G.4})$$

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8) This trial was conducted by ISO/TC 92/SC 1/WG 7.

9) ISO 5725:1986 has been withdrawn and replaced by ISO 5725-1, ISO 5725-2, ISO 5725-3, ISO 5725-4, ISO 5725-5 and ISO 5725-6.

The results for  $\dot{q}_{A,60}''$  in the range of 57 kW/m<sup>2</sup> to 233 kW/m<sup>2</sup> are given as Equations (G.5) and (G.6):

$$r = 35,15 + 0,105 \dot{q}_{A,60}'' \quad (\text{G.5})$$

$$R = 52,79 + 1,055 \dot{q}_{A,60}'' \quad (\text{G.6})$$

The results for  $\dot{q}_{A,180}''$  in the range of 49 kW/m<sup>2</sup> to 172 kW/m<sup>2</sup> are given as Equations (G.7) and (G.8):

$$r = 36,30 + 0,026 \dot{q}_{A,180}'' \quad (\text{G.7})$$

$$R = 9,290 + 0,595 \dot{q}_{A,180}'' \quad (\text{G.8})$$

The results for  $q_s''$  in the range of 10 MJ/m<sup>2</sup> to 56 MJ/m<sup>2</sup> are given as Equations (G.9) and (G.10):

$$r = 7,599 + 0,369 q_s'' \quad (\text{G.9})$$

$$R = 12,06 + 0,818 q_s'' \quad (\text{G.10})$$

### G.3 Example

The meaning of Equations (G.1) to (G.10) is best illustrated by means of an example. Assuming a laboratory tests a single sample of a certain material and determines that the average heat release rate 180 s after ignition is 100 kW/m<sup>2</sup>. If the same laboratory conducts a second test on the same material, the value of  $r$  is evaluated as Equation (G.11):

$$r = 36,30 + 0,026 \times 100 = 38,9 \text{ kW/m}^2 \quad (\text{G.11})$$

Then the probability is 95 % that the result of the second test falls between 61,1 kW/m<sup>2</sup> and 138,9 kW/m<sup>2</sup>. If the same material is tested by a different laboratory, the value of  $R$  is evaluated as Equation (G.12):

$$R = 9,290 + 0,595 \times 100 = 68,8 \text{ kW/m}^2 \quad (\text{G.12})$$

Then the probability is 95 % that the result from the test at that laboratory falls between 31,2 kW/m<sup>2</sup> and 168,8 kW/m<sup>2</sup>.

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