
**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

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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.

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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)

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/m² to 50 kW/m². 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

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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	m^2
A_s	Exposed specimen area	m^2
E	Net heat released for complete combustion, per unit of oxygen consumed	13,1 MJ/kg O_2
E_{CO}	Net heat released per unit mass of oxygen consumed for combustion of CO to CO_2	17,6 MJ/kg O_2
$E_{propane}$	Net heat released for complete combustion of propane, per unit of oxygen consumed	12,78 MJ/kg O_2
$E_{methane}$	Net heat released for complete combustion of methane, per unit of oxygen consumed	12,51 MJ/kg O_2
F_{OD}	Relative optical density	dimensionless
f_x	Yield of gas x	kg/kg
$f_{(Re)}$	Reynolds number correction for bi-directional probe differential pressure measurement	—
$\Delta H_{c,ng}$	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	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_{CO}	Relative molecular mass of carbon monoxide	28 kg/kmol
M_{CO_2}	Relative molecular mass of carbon dioxide	44 kg/kmol
M_{dry}	Relative molecular mass of dry air	29 kg/kmol
M_e	Relative molecular mass of exhaust gases	kg/kmol
M_{H_2O}	Relative molecular mass of water	18 kg/kmol
M_{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
Δp	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 ²
$\dot{q}''_{A,180}$	Average heat release rate per unit area of specimen for the first 180 s after ignition	kW/m ²
\dot{q}''_{peak}	Peak heat release rate per unit area of specimen	kW/m ²
q''_s	Total heat released per unit area of specimen	MJ/m ²
\dot{q}''_s	Heat release rate per unit area of specimen	kW/m ²
$q''_{s,i}$	Heat released per unit area of specimen, in incoming air	MJ/m ²
R_{inst}	Instantaneous rate of production of light-obscuring smoke	m ² /s
R_{tot}	Total amount of smoke	m ²
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 ³ /s
\dot{V}_s	Volumetric flow at location of smoke meter (value adjusted for smoke measurement calculations)	m ³ /s
Δt	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 ₂ in exhaust flow	dimensionless
$X_{CO_2,i}$	Measured mole fraction of CO ₂ in incoming air	dimensionless
$X_{O_2,e}$	Measured mole fraction of O ₂ in exhaust flow	dimensionless
$X_{O_2,i}$	Measured mole fraction of O ₂ in incoming air	dimensionless
[x]	Relative mass fraction of gas x	kg/kg
α	Combustion expansion factor (an average value of 1,105 is used for mixed fuels or when the exact factor is unknown)	dimensionless
ρ	Density of air at the temperature in exhaust duct	kg/m ³
ρ_0	Density of air at 273,15 K: 1,293	kg/m ³
ϕ	Oxygen depletion factor	dimensionless

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4 Principle

4.1 This test method is designed to measure the heat release rate from a 1 m² 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/m² 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 [1], [2]. 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 ± 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 ¹⁾.

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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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 Chromel²⁾ 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-Boelter³⁾ (thermopile) type, with a design range of 50 kW/m² to 100 kW/m².

The target receiving radiation, and possibly to a small extent convection, shall be flat, circular, of approximately 12,5 mm in diameter and coated with a durable matt-black finish. The target shall be water-cooled. Radiation shall not pass through any window before reaching the target. The instrument 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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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 m³/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

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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 ascerite⁴⁾ 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.

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.