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

Part 5: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement) under reduced oxygen atmospheres

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

Part 5:
Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement) under reduced oxygen atmospheres

1 Scope

This document specifies the apparatus and procedure for measuring reaction to fire behaviour under reduced oxygen atmospheres. Continuous measurements are made to calculate heat release rates, smoke and specific gas production rates, and mass loss rates. Ignition time measurements are also made and ignition behaviour is obtained. Pyrolysis parameters of specimens exposed to controlled levels of irradiance and controlled levels of oxygen supply can be determined as well.

Different reduced oxygen atmospheres in the test environment are achieved by controlling the oxygen volume concentration of input gas fed into the chamber (vitiation) or by controlling the total volume of atmosphere fed into the chamber (ventilation). Ranges of oxygen volume concentration below 20,95 % of oxygen can be studied. The apparatus is not intended to control enriched oxygen conditions above atmospheric 20,95 % oxygen concentration.

The measurement system prescribed in this document is based on the cone calorimeter apparatus described in ISO 5660-1. Therefore, this document is intended to be used in conjunction with ISO 5660-1.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 13927:2015, *Plastics — Simple heat release test using a conical radiant heater and a thermopile detector*

ISO 13943, *Fire safety — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5660-1, ISO 13943 and the following apply.

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

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

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

3.1
ambient atmosphere
atmosphere with an oxygen volume concentration of approximately 20,95 % in a control volume and unrestricted air flow into the same control volume

3.2
reduced oxygen atmosphere
atmosphere with either one of the following conditions that differ from ambient atmospheres:

a) vitiated atmosphere: atmosphere with less oxygen molecules than in ambient air in the same volume at same temperature and pressure levels (oxygen concentration below 20,95 %; vitiated conditions), with the remaining molecules balanced by inert gas molecules

b) under ventilated atmosphere: atmosphere with a limited air supply that leads to fewer oxygen molecules per time supplied to a combustion reaction than needed to allow stoichiometric reactions to take place (under-ventilated conditions)

3.3
vitiation-controlled conditions
conditions under which the volume concentration of oxygen is intentionally controlled or reduced in the combustion environment

Note 1 to entry: Vitiation-controlled conditions represent an oxygen depleted fire environment.

3.4
ventilation-controlled conditions
conditions in which the supply rate of (ambient or vitiated) air to the combustion environment is intentionally controlled or limited

Note 1 to entry: Ventilation-controlled conditions represent a fire environment with limited fresh air supply.

4 Symbols

For the purposes of this document, the symbols given in ISO 5660-1 and the following apply.

Table 1 — Symbols and their designations and units

Symbol	Designations	Unit
A_S	initially exposed surface area of the specimen	m^2
C	orifice flow meter calibration constant	m^{½} g^{½} K^{½}
γ	thermal expansion factor	(dimensionless)
$\tilde{\gamma}$	thermal changeable dilution factor	(dimensionless)
Δh_c	net heat of combustion	kJ g^{-1}
$\dot{m}_e$	mass flow rate in the exhaust duct during the test	kg s^{-1}
$\dot{m}_e^0$	initial mass flow rate in the exhaust duct	kg s^{-1}
$\dot{m}_f$	mass flow rate of fuel, burning rate of the specimen	kg s^{-1}
$\dot{m}_g^E$	mass flow rate of the incoming gas mixture to the enclosure	kg s^{-1}
Δp	orifice meter pressure differential	Pa
$\dot{q}(t)$	heat release rate	kW
$\dot{q}_A(t)$	heat release rate per unit area	kW m^{-2}
ϕ	oxygen depletion factor	(dimensionless)

Table 1 *(continued)*

Symbol	Designations	Unit
ϕ_{GER}	global equivalence ratio	(dimensionless)
T_e	absolute temperature of gas at the orifice meter	K
$\dot{V}_A$	volume flow rate of Air	L/min
$\dot{V}_g^E$	volume flow rate of Gas to the enclosure	L/min
$\dot{V}_N$	volume flow rate of Nitrogen gas	L/min
$X_{O_2\text{, Air}}$	oxygen concentration in air (bottled, pressurized)	(dimensionless)
$X_{O_2}^1$	value of combustion gas oxygen analyser reading, before delay time correction	(dimensionless)
$X_{O_2}^A$	actual value of combustion gas oxygen analyser reading	(dimensionless)
$X_{O_2}^{A^0}$	initial baseline value of combustion gas oxygen analyser reading (with enclosure environment established)	(dimensionless)
$X_{O_2}^{A^S}$	surrounding baseline value of oxygen analyser reading (before enclosure environment established – enclosure door open)	(dimensionless)
X_{CO}^A	actual value of combustion gas carbon monoxide analyser reading	(dimensionless)
$X_{CO_2}^A$	actual value of combustion gas carbon dioxide analyser reading	(dimensionless)
$X_{O_2}^E$	oxygen concentration in the enclosure	(dimensionless)
$X_{CO_2}^{A^S}$	surrounding baseline value of combustion gas carbon dioxide analyser reading (before enclosure environment established – enclosure door open)	(dimensionless)
$X_{H_2O}^S$	surrounding value of water vapor	(dimensionless)

5 Principle

The principle of this test method is based on the observation that, generally, both thermal and chemical products of a combustion reaction vary in quantity and quality depending on the atmospheric environmental conditions in which the reactions occurs. This test method provides a controlled environment to assess the contribution that a product under test can make to the rate of heat release, the production rate of gaseous products, and the smoke production rate, in different reduced oxygen atmospheres and/or differently ventilated atmospheres during the product's involvement in fire. The properties are determined on small representative specimens. Specimens in the test are burned in ambient atmospheres or predetermined reduced oxygen atmospheres, while being subjected to a predetermined external irradiance within the range of 0 kW m^{-2} to 50 kW m^{-2}. Measurements are made of oxygen and other gas concentrations in the exhaust, light transmission, exhaust gas flow rates, and specimen mass.

Heat release rate measurement is based on the observation that the net heat of combustion is proportional to the amount of oxygen required for combustion. The relationship is that approximately 13,1 × 10^3 kJ of heat are released per kilogram of oxygen consumed. This is accurate within ±5 % for complete combustion and differs by ±20 % considerably for incomplete combustion. Measurements of oxygen concentrations and total exhaust gas flow rates are conventionally made. Enhanced measurements of carbon dioxide concentrations, carbon monoxide concentrations, other species concentrations, soot, water vapor, and unburnt fuel allow application of appropriate corrections depending on stoichiometries of the combustion reactions. These measurements are used to calculate the mass of oxygen consumed. Results are reported as heat release rate and total heat release, both normalized to exposed specimen surface area. The heat release rate of a burning specimen is calculated as the product of the oxygen mass consumed by the fire and the averaged proportionality

13,1 × 10^{3} kJ kg$^{−1}$ with corrections for incomplete combustion. The enhanced measurements for carbon dioxide, carbon monoxide, and water vapor are applied for general corrections in this document. Where available, specific values for the proportionality can be used as quotient of the heat of combustion of a burning fuel and its stochiometric oxygen to fuel mass ratio. The total heat release is calculated by numerical integration of the heat release rate over the time interval being considered. Both variables are normalized to area because heat release is proportional to the burning surface area.

The principle of the smoke measurement is based on the observation that, generally, the intensity of light that is transmitted through a volume of combustion products is an exponentially decreasing function of distance. Measurements are made of exhaust gas obscuration, exhaust gas flow rate, and mass loss rate of the specimen. Exhaust gas obscuration is measured as the fraction of laser light intensity that is transmitted through the mixture of gases, aerosols, and particles in the exhaust duct. This fraction is used to calculate the extinction coefficient according to Bouguer's law. In particular, with non-flaming and anaerobic pyrolysis processes, extinction coefficients differ from extinction values for combustion smoke. The test results are reported in terms of smoke production and smoke production rate, both normalized to exposed specimen surface area. Smoke production rate is calculated as the product of the extinction coefficient and the volumetric flow rate of the smoke in the exhaust duct. Smoke production is calculated by numerical integration of the smoke production rate over the time interval being considered. The variables reported are normalized to area because smoke production is proportional to area.

Gas production measurements are performed by measuring gas concentrations in the exhaust duct. Gas production rates are calculated from those concentration measurements utilizing general equations and relations. Species yields are derived from the specific gas mass flow rate divided by the actual fuel mass loss rate at the same time interval.

Atmospheric environmental conditions may range from approximately 1 % to 20,95 % of oxygen and 10 L/min to 180 L/min volume flow rate. They are predetermined and controlled within the combustion environment by maintaining the ratio and volume flow rate of air and nitrogen gas respectively. The oxygen concentration in the atmospheric conditions and the total gas flow rate to the environment are monitored with relevant measurement devices. Air and nitrogen gas shall be provided either as bottled gases or as oil free pressurized air from a compressor, and liquid nitrogen vaporizer respectively.

NOTE Atmospheric environmental conditions can be characterised in terms of the Global Equivalence Ratio. Details are given in Annex A.

6 Apparatus

The apparatus described in this document allows measurement of reaction to fire behaviour under reduced oxygen atmospheres. Ranges of oxygen volume concentration below 20,95 % of oxygen can be studied. For those conditions above 15 % of oxygen, flaming combustion is usually expected. For those below 15 % of oxygen, flaming may occur but is generally not expected to occur for many products. Anaerobic pyrolysis experiments at close to 0 % of oxygen can be carried out in absence of the oxygen depletion measurements.

The apparatus utilizes the components and controls of the apparatus specified in ISO 5660-1 supplemented by apparatus modification detailed in this document to facilitate testing under reduced oxygen atmospheres. This principally consists of replacing the standard cone heater assembly by a second unit housed in a chamber that can by supplied with metered mixtures of air and nitrogen. Measurements are otherwise similar to those made in ISO 5660-1.

Optional gas measurement equipment shall be used as detailed in ISO 5660-1:2015, Annex G.

An apparatus exclusively for anaerobic pyrolysis experiments may alternatively utilize components and controls of the apparatus specified in ISO 13927.

A schematic representation of the apparatus required for this document is given in Figure 1. Components described in ISO 5660-1 are marked. Components specific to reduced oxygen atmosphere testing are specified in 6.1 to 6.7 of this document.

6.1 General

The conical shaped radiant heater described in ISO 5660-1:2015, 6.1 shall be integrated into the top face of an enclosure described in in 6.2.1. The cabinet shall also include the radiation shield (ISO 5660-1:2015, 6.3), the weighing device (ISO 5660-1:2015, 6.4) with an additional cooling shield as described in 6.3, the specimen holder (ISO 5660-1:2015, 6.5), and the ignition circuit (ISO 5660-1:2015, 6.9). The heat flux meter and housing (ISO 5660-1:2015, 6.12) and the calibration burner (ISO 5660-1:2015, 6.13) shall be provided as well. Appropriate mountings shall be available to perform calibration measurements using the heat flux meter and calibration inside the enclosure. A gas mixing and supply system shall be connected to the enclosure to allow adjusting the atmospheric conditions.

6.2 Heater and enclosure and chimney arrangement with cone calorimeter as per ISO 5660-1

The test enclosure described in 6.3 replaces the standard cone heater assembly in the ISO 5660-1 apparatus. It shall be centred underneath the exhaust hood and can be used in each of the following configurations using a chimney on top of the enclosure as described in 6.5.

1) When testing with an enclosure gas supply rate lower than the exhaust flow rate, the enclosure and chimney should not be linked directly to the exhaust hood. Air from the surroundings shall be allowed to enter the exhaust hood.

   NOTE 1 The effect of the chimney in the unlinked configuration on various results are discussed in Reference [2].

   NOTE 2 An exhaust flow rate that exceeds the enclosure supply rate would cause under pressure, leakages, and potentially uncontrolled conditions in the enclosure.

2) When undertaking anaerobic pyrolysis experiments these can be carried out in either unlinked or linked apparatus depending on the applicable gas supply rate or if the enclosure is stand-alone without the oxygen depletion measurement equipment running. Annex C specifies more details about the stand-alone arrangement.

Regardless of the configuration or the enclosure inlet gas flow rate, the exhaust flow rate in the duct shall be sufficiently high to reliably entrain all combustion/pyrolysis products released during the process. The minimum exhaust flow rate at the beginning of the test shall be at least 0,012 ± 0,002 m^3/s.

6.2.1 Enclosure

A stainless steel enclosure shall have internal dimensions of W × D × H (370 ± 20) mm × (300 ± 20) mm × (320 ± 20) mm. A door shall be mounted on the front of the enclosure to provide access to all inner parts and to allow specimen loading. When opened for specimen loading, a door may allow significant amounts of air entering the enclosure. This may unintentionally change the predetermined controlled atmosphere. An alternative opening scheme may be used if it allows only minimum air entering the enclosure during specimen loading. At least one wall or door element shall contain a window to allow the specimen to be observed during a test. At least one gas connection port shall be mounted at the level of the sample that allows gas sampling of the enclosure atmosphere. Additional ports may be present for cooling water entry, additional gas sampling, and/or temperature measurement as well as extinguishing, and radiation measurement equipment.

All connections of wall assemblies, ports and openings shall be tightly sealed to prevent surrounding air from penetrating in the enclosure during the test.

The conical heater, specified in ISO 5660-1:2015, 6.1 shall be mounted in the centre of the top face of the enclosure. It shall be capable of producing an irradiance level on the surface of the specimen of 0 kW m^{-2} to 50 kW m^{-2}. Higher heat flux levels may be possible if the equipment is suitable for high temperature conditions. A water-cooled collar should be mounted between the heater and the top of the enclosure to minimize warping of the top plate due to the hot electrical heater. Heat resistant sealing material shall be used for sealing the cone heater openings against unintended air diffusion/penetration into the enclosure.

In accordance with applicable sections of ISO 5660-1, the enclosure shall contain a radiation shield (ISO 5660-1:2015, 6.3), an ignition circuit (ISO 5660-1:2015, 6.10), and a specimen holder (ISO 5660-1:2015, 6.6). The enclosure shall be capable of incorporating and operating the weighing device per ISO 5660-1:2015, 6.5 as well. The weighing device may be located outside the enclosure if proper sealing of the connection rod is ensured and accurate sample mass measurement is provided.

Two local entry points or a mesh of points shall be provided in the base of the enclosure to feed the enclosure with a pre-mixed mixture of air and gases in a suitable ratio to create the desired test atmosphere. The entry points shall be designed in a way that minimizes high local flow rates inside the enclosure. A baffle design that has been used to meet these requirements is shown in Figure 3. Alternative equipment, such as screens and beads, or similar may be used if it minimizes high local flow rates. Screens and beads at the bottom of the enclosure are expected to provide uniformly consistent and upward inlet flow velocity. Baffles as per Figure 3 shall be used. When using an alternative to the baffles in Figure 3, comparative tests between an ISO 5660-1 apparatus and the apparatus described in this document shall be conducted for the same product at 20,95 % of oxygen. Time to ignition and heat release rate measurement results shall be compared.

6.3 Water-cooling for weighing device

A water-cooled shield or housing shall be provided on top or around the weighing device to ensure proper weight measurement while protecting the weighing device from the heat inside the enclosure during a test. The device's connection rod may be cooled as well. However, water-cooling shall not affect the specimen mass measurement at any time before or during a test. Weighing devices that are located outside the enclosure do not require water-cooling.

6.4 Chimney

A circular cross-section chimney shall be mounted on top of the top-plate of the conical heater. The axis of the chimney shall coincide with the axis of the heater. The chimney shall have a length of (600 ± 2) mm and an internal diameter of (115 ± 2) mm following the chimney design in ISO 13927:2015, 6.4.

NOTE The internal diameter of 115 mm is a suitable empiric compromise that ensures appropriate exhaust gas flow rates and flame lengths for the expected range of heat release rate results. Large diameters lower the gas flow rate within the chimney and delay proper transportation to the gas sampling point which bias the time resolution of the burning behaviour. Smaller diameters increase the gas flow rate and the flame length which can lead to unintended burning of flames in uncontrolled atmospheric conditions above the chimney (secondary burning; see Annex A).

The chimney shall be made of 1-mm-thick stainless steel or glass. Its upper end shall overlap the bottom of the exhaust hood and reaches (45 ± 5) mm into it. The height of the enclosure shall be positioned appropriately.

The purpose of the chimney is to limit potential secondary burning when flames access ambient air. If secondary burning is observed above the chimney, the test shall be considered as unsuccessful as per 11.4 of this document, and test data shall be void. Data from controlled-atmosphere testing, using a longer chimney length can be considered as an option, as can a directly linked setup used according to 6.2. However, the chimney length shall be limited to not exceed the overlapping criteria as mentioned above. The enclosure should also not be positioned too close to the floor so that conflicts with occupational health and safety regulations are a concern.

To prevent exhaust gases from escaping the exhaust hood, the end of the chimney shall be designed to provide flow velocity and flow direction suitable to entirely collect gases through the hood. A flow restrictor reducing the chimney diameter has been found to be effective to ensure sufficient flow velocity for the smoke to reach into the hood. Figure 4 shows a flow restrictor with dimensions that work properly. Other designs may be used as well. This device shall not be included into the 600 mm length of the chimney. It, however, shall also not add more than (40 ± 5) mm to the chimney.

