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BSI Standards Publication

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



National foreword

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Part 5:

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Foreword

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

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

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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 <u>www.iso.org/members.html</u>.

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 <u>http://www.electropedia.org/</u>

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.

Symbol	Designations	Unit
A _S	initially exposed surface area of the specimen	m ²
С	orifice flow meter calibration constant	$m^{1/2} g^{1/2} K^{1/2}$
γ	thermal expansion factor	(dimensionless)
$\tilde{\gamma}$	thermal changeable dilution factor	(dimensionless)
$\Delta h_{\rm c}$	net heat of combustion	kJ g ^{−1}
, 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
, m _f	mass flow rate of fuel, burning rate of the specimen	kg s ⁻¹
$\dot{m}_{ m g}^E$	mass flow rate of the incoming gas mixture to the enclosure	kg s⁻¹
Δp	orifice meter pressure differential	Ра
$\dot{q}(t)$	heat release rate	kW
$\dot{q}_A(t)$	heat release rate per unit area	kW m ⁻²
ϕ	oxygen depletion factor	(dimensionless)

Table 1 — Symbols and their designations and units

Symbol	Designations	Unit
$\phi_{\rm GER}$	global equivalence ratio	(dimensionless)
T _e	absolute temperature of gas at the orifice meter	К
<i>V</i> _A	volume flow rate of Air	L/min
$\dot{V}^{\mathrm{E}}_{\mathrm{g}}$	volume flow rate of Gas to the enclosure	L/min
$\dot{V}_{ m N}$	volume flow rate of Nitrogen gas	L/min
X ₀₂ , Air	oxygen concentration in air (bottled, pressurized)	(dimensionless)
$X_{0_2}^1$	value of combustion gas oxygen analyser reading, before delay time correction	(dimensionless)
$X_{0_2}^A$	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}	actual value of combustion gas carbon monoxide analyser reading	(dimensionless)
X ^A _{CO2}	actual value of combustion gas carbon dioxide analyser reading	(dimensionless)
<i>X</i> ^E ₀₂	oxygen concentration in the enclosure	(dimensionless)
$X_{\rm CO_2}^{\rm A^S}$	surrounding baseline value of combustion gas carbon dioxide analyser reading (before enclosure environment established – enclosure door open)	(dimensionless)
X ^S _{H₂0}	surrounding value of water vapor	(dimensionless)

Table 1 (continued)

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 $13,1 \times 10^3$ 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

 $13,1 \times 10^3$ 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 <u>Annex A</u>.

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 <u>Figure 1</u>. Components described in ISO 5660-1 are marked. Components specific to reduced oxygen atmosphere testing are specified in <u>6.1</u> to <u>6.7</u> 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. <u>Annex C</u> 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 $0.012 \pm 0.002 \text{ m}^3/\text{s}$.

6.2.1 Enclosure

A stainless steel enclosure shall have internal dimensions of $W \times D \times H$ (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 <u>Annex A</u>).

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.

6.5 Air- and gas-supply system

The pre-mixed mixture of air or gases shall be created in a gas mixing and supply unit. This unit shall comprise at least the following:

- shut-off valves for each gas component connected;
- manual or electric flow regulators accurate in a range of 5 L/min to 200 L/min;
- flow meters for monitoring each gas component flow rate (accurate within the same range);
- flow stabilizing devices for each gas component;
- a gas mixing chamber;
- a flow meter reading the total flow rate to the enclosure. The total gas flow rate to the enclosure shall be measured and monitored during the test.

The gas supply system shall be capable of delivering air or gas mixtures with an oxygen concentration ranging from approximately 1 % of oxygen to 20,95 % oxygen at each flow rate from 10 L/min to 180 L/min. For pyrolysis studies, oxygen concentrations of approximately 1 % of oxygen shall be adjustable.

Ideally, environmental conditions start from 0 % of oxygen. As this is technically challenging and does not add value in the context of controlled-atmosphere testing as intended by this document, the above mandatory wording of "approximately 1 %" has been chosen. The actual capability of the apparatus should be as close to 0 % as feasible with reasonable effort.

6.6 Enclosure oxygen analyser

Oxygen levels within the enclosure shall be measured and monitored during the test to facilitate concentration setting and control. A paramagnetic oxygen gas analyser with a range of 0 % oxygen to 25 % oxygen and an accuracy of 0,1 % shall be used for this purpose.

Sampling is done via a probe located inside the enclosure at the height of the sample surface and (20 ± 5) mm horizontally distant from it. The probe shall be connected to a gas port in the enclosure envelope.

6.7 Data collection and analysis system

A data collection and analysis system shall have facilities for recording the outputs of the flow meter reading the total flow rate to the enclosure as specified in <u>6.6</u> and the enclosure oxygen analyser as specified in <u>6.7</u> of this document. This data collection system shall be incorporated into the data collection and analysis system described in ISO 5660-1:2015, 6.15.

7 Suitability of product for testing

See ISO 5660-1:2015, Clause 7.

8 Specimen construction and preparation

See ISO 5660-1:2015, Clause 8. The following also applies.

Three different specimens shall be tested at each atmospheric condition (vitiation and/or ventilation).

9 Test environment

See ISO 5660-1:2015, Clause 9.

10 Calibration

Calibrations shall be performed in accordance with ISO 5660-1:2015, Clause 10 and the following shall be applied in addition. All calibrations shall be performed without gas supply to the enclosure. The enclosure door shall be open during calibration processes.

10.1 Operating calibrations

10.1.1 Enclosure oxygen analyser

The enclosure oxygen analyser shall be zero and span calibrated in accordance with the requirements given in ISO 5660-1:2015, 10.2.3. Differing from that standard, the response shall be adjusted to $(0,00 \pm 0,1)$ % respectively $(20,95 \pm 0,1)$ %. After each specimen has been tested, the enclosure door shall be opened and non-ambient gas supply shall be turned off. A response level of $(20,95 \pm 0,1)$ % oxygen shall be achieved within a reasonable time, but not more than 120 s.

10.1.2 Enclosure flow rate measurement

Calibrate enclosure supply flow rate meter if applicable based on manufacturer's manual.

10.1.3 Heater calibration

Sufficient time shall be provided to allow the steel enclosure temperature to reach steady state during calibration of the heater. Operate the cone heater for at least 30 min when stable at set point before collecting heat flux calibration data.

11 Test procedure

11.1 General precautions

WARNING — So that suitable precautions are taken to safeguard health, the attention of all concerned in fire tests is drawn to the possibility that flammable, toxic or harmful gases can be evolved during exposure of test specimens.

Insufficient combustion air supply can lead to a release and accumulation of incomplete combustion products, e.g. flammable gases, inside the enclosure. The hazard of delayed ignition and instantaneous combustion (backdraft) may therefore exist once oxygen is supplied to the enclosure (also due to accidental and unintentional supply). The accumulation of unburned combustion products within the enclosure shall, therefore, be prevented.

11.2 Initial preparation

ISO 5660-1:2015, 10.2 and 11.2 shall be followed for initial preparation.

Alternatively to the requirements in ISO 5660-1:2015, 11.2.4, the exhaust flow rate may be set to any value within the range of $(0,012 \pm 0,002)$ m³/s to $(0,024 \pm 0,002)$ m³/s.

11.3 Procedure

When using a configuration as described in <u>6.2.1</u> of this document (chimney not linked to the exhaust hood), the test procedure detailed in <u>11.3.1</u> to <u>11.3.14</u> shall be followed to collect all the data needed for calculation.

NOTE Details of the procedure are described in Reference [2].

When using a configuration as described <u>6.2.1</u> of this document (chimney/enclosure linked to the exhaust hood), the standard procedure as per ISO 5660-1:2015, 11.3 may be used with slight adjustments described in <u>Annex B</u> of this document.

11.3.1 Pre-test conditions

- Apparatus calibrated according to the requirements mentioned in <u>Clause 10</u> of this document.
- Radiation shield open and thermal barrier on top of the weighing device.
- Ignition circuit switched off.
- Enclosure door open.
- Gas supply to the enclosure turned off.
- Sampling pumps on and gas analysis units running.

11.3.2 Start data collection. Collect 60 seconds of *surroundings* baseline data. The standard scan interval is 5 s, unless a short burning time is anticipated (see ISO 5660-1:2015, 7.3).

11.3.3 Close enclosure door, turn gas supply to the enclosure on, and set flow rate and level of oxygen concentration to the desired points by adjusting and maintaining volume flow rates of each gas involved. Set settings as accurate as ± 5 % of desired flow rate and $\pm 0,1$ % of oxygen.

When ventilation-controlled conditions are not intended, the total inlet gas flow rate to the enclosure shall not fall below 150 L/min and shall also not exceed 180 L/min. This applies for ambient air as well as for vitiated air.

When ventilation-controlled conditions are intended, the total gas inlet flow rate to the enclosure shall not fall below 10 L/min.

When vitiation-controlled conditions are intended, the required flow rates of each gas involved for a given concentration of oxygen are, mathematically, solutions of the following simultaneous formulae:

$$\dot{V}_{\rm N} + \dot{V}_{\rm A} = \dot{V}_{\rm g}^{\rm E} \tag{1}$$

 $X_{0_{2}}^{E} [\%] = \frac{X_{0_{2},Air} \cdot \dot{V}_{A}}{\dot{V}_{N} + \dot{V}_{A}} \cdot 100$ ⁽²⁾

$$\dot{V}_{A} = \frac{V_{g}^{E}}{X_{O_{2}, Air}} \cdot X_{O_{2}}^{E}$$
(3)

$$\dot{V}_{\rm N} = \dot{V}_{\rm g}^{\rm E} - \dot{V}_{\rm A} \tag{4}$$

Table D.1 in Annex D shows flow rates of air and nitrogen gas respectively for different oxygen concentrations and for 150 L/min total inlet gas flow rate.

11.3.4 Purge the enclosure and allow the system to stabilize and the atmosphere to equilibrate in the enclosure once proper gas flow settings have been established. Allow the system to flow at least for 90 s. Validate flow rate and oxygen concentration to be at the set point and readjust accordingly if needed. Time for stabilization restarts with each re-adjustment.

11.3.5 Once system has stabilized, do not change any gas flow settings. Collect 60 s of *initial* baseline data. Mark and record start and end of this reading.

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11.3.6 Once initial baseline data have been collected, insert radiation shield in position, open enclosure door, and remove the thermal barrier protecting the weighing device. Then place the specimen holder and specimen, prepared according to <u>Clause 8</u>, on the weighing device.

The radiation shield should be cooler than 100 °C immediately prior to the insertion.

Close the enclosure door not later than 20 s after opening it. Insert the spark igniter in position.

11.3.7 Purge the enclosure and allow the system to equilibrate. Conditions identical to those during initial baseline shall be achieved. Equilibration time depends on the gas supply flow rate to the enclosure. A maximum 60 s equilibration time should be targeted, however, 120 s shall not be exceeded. The equilibration time shall be kept constant within test series to reduce the potential of different pre-heating conditions.

11.3.8 Remove the radiation shield and mark this point of time as start of the test.

11.3.9 Record the times when flashing or transitory flaming occurs. When sustained flaming occurs, record the time, turn off the spark, and remove the spark igniter. If the flame extinguishes in less than 60 s after turning off the spark, re-insert the spark igniter and turn on the spark within 5 s, do not remove the spark until the entire test is completed. Report these events in the test report.

11.3.10 Record the times when flashing or sustained flaming occurs above the chimney if applicable. Also, and if applicable, report times when changes in the atmospheric conditions occur such as a change in the enclosure oxygen analyser's reading. Report these events in the test report.

11.3.11 Collect all data until either:

- a) 32 min after the time to sustained flaming (the 32 min consist of a 30 min test period, and an additional 2 min post-test period to collect data that will be time-shifted). Data is processed to time to sustained flaming plus 30 min.
- b) 30 min have elapsed and the specimen has not ignited; applies only for tests at 20,95 % oxygen.
- c) Current X₀₂ reading returns to a value greater than the initial baseline value minus 100 parts per million of oxygen concentration for 10 min. The end of test is the beginning of the 10 min period.
- d) The mass loss of the specimen is less than 0,1 g for 60 s. The end of test is the beginning of the 60 s period.

Whichever occurs first, but in any case, minimum test duration shall be 5 min.

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

- **11.3.12** Open enclosure door, remove specimen holder, place thermal barrier on weighing device.
- **11.3.13** Turn gas supply to enclosure off.
- **11.3.14** Stop data collection.

11.4 Criteria to consider a test as successful

The following list of criteria shall be met to consider a test as being successful and the data as being reasonably unaffected by exterior influences. If one of the criteria is not met, the changes suggested

in $\underline{Annex} \underline{A}$ can be considered to prevent unfavourable conditions, otherwise the data shall not be reported.

a) No flames above the chimney have been observed and/or reported during the test. This applies both to flames attached to the specimen surface and flames detached from the specimen surface.

Flames above the chimney indicate combustion in an atmosphere that potentially differs from the one set in the enclosure (secondary burning). The changes mentioned in <u>Annex A</u> can be applied in order to prevent this from happening.

- b) The oxygen measurement within the enclosure has been continuously recorded and does not show any significant changes greater than 1 % of oxygen during the test that indicates a local oxygen depletion due to extensive oxygen consumption (changes in atmospheric conditions).
- c) Ignition times under ambient atmospheres in the apparatus described in this document do not significantly differ from those obtained for the same material in the standard apparatus according to ISO 5660-1. It shall be verified through a set of triplicate tests, that the use of the enclosure does not affect ignition times of a specific material in excess of the repeatability and reproducibility range described in ISO 5660-1:2015, Annex C.

12 Calculations

12.1 General

All gas concentration data used for calculation purposes are measured in the exhaust duct with the optional gas analysis system per ISO 5660-1. This optional setup includes O_2 , and in addition CO_2 and CO measurement. The formulae from 12.2 to 12.4 assume that O_2 , CO_2 and CO data are measured and collected. Less gas analysis setups are not considered appropriate for measuring of heat release rates and production rate of gaseous products using this apparatus.

Measurement data from the enclosure oxygen analyser are rather used for data validation purposes per Section b) of this document than for calculation purposes.

Mass loss rate shall be calculated using the formulae in ISO 5660-1:2015, 12.5. A least-square method may be also used.

The calculation of smoke obscuration described in ISO 5660-1:2015, 12.6 applies without changes to this document. It shall be noted that a general correlation between exhaust gas light obscuration and smoke, soot, aerosol, and particle production rates is not defined. In particular, for non-flaming and anaerobic pyrolysis results, the mass specific extinction coefficient can be generalized and taken from Reference [3].

12.2 Calibration constant for oxygen consumption analysis

ISO 5660-1:2015, 12.2 applies.

12.3 Correct time delay

Prior to performing other calculations, calculate all (O_2 , CO_2 , CO) analyser readings from the recorded analyser data and the delay time, t_d , using ISO 5660-1:2015, Formula (7).

12.4 Heat release rate

When using a configuration as described in <u>6.2.1</u> of this document (chimney not linked to the exhaust hood), the following formulae in <u>12.4.1</u> shall be used for heat release rate calculation as recommended in Reference [2].

When using a configuration as described in 6.2.2 of this document (chimney/enclosure linked to the exhaust hood), the standard procedure as per ISO 5660-1:2015, 12.3.2 may be used.

12.4.1 Calculate the heat release rate, $\dot{q}(t)$, from the values corrected according to <u>12.3</u> as follows:

$$\dot{q}(t) = E \cdot 1, 10 \cdot \left(X_{O_2}^{A^0} \gamma - X_{O_2}^{A^S} (\gamma - 1) \right) \cdot C \sqrt{\frac{\Delta p}{T_e}} \left[\frac{\phi - \left(\frac{E_{CO} - E}{2E} \right) (1 - \phi) X_{CO}^A / X_{O_2}^A}{(1 - \phi) + \phi \left(1 + 0, 5 \left(X_{O_2}^{A^0} \gamma - X_{O_2}^{A^S} (\gamma - 1) \right) \right)} \right] (1 - X_{H_2O}^S \tilde{\gamma})$$
(5)

where

$$E = \Delta h_c / r_0$$
 for the specimen is taken as $(13, 1 \times 10^3 \text{ kJ kg}^{-1})$, unless a more accurate value is known;

$$X_{O_2}^{A^0}$$
 is determined as the average of the oxygen analyser output measured during the 1-min initial baseline measurement;

The oxygen depletion factor ϕ follows from:

$$\phi = \frac{\left[\left(X_{O_2}^{A^0} \gamma - X_{O_2}^{A^S} (\gamma - 1) \right) \left(1 - X_{CO_2}^A - X_{CO}^A \right) \right] - \left[X_{O_2}^A \left(1 - X_{CO_2}^{A^S} \tilde{\gamma} \right) \right]}{\left(1 - X_{O_2}^A - X_{CO}^A \right) \left(X_{O_2}^{A^0} \gamma - X_{O_2}^{A^S} (\gamma - 1) \right)}$$
(6)

The thermal expansion factor γ is the quotient of the 1-minute average of the exhaust mass flow rate reading during initial baseline measurement $\dot{m}_{\rm e}^0$ and the actual exhaust mass flow rate during a test $\dot{m}_{\rm e}$.

$$\gamma = \frac{\dot{m}_{\rm e}^0}{\dot{m}_{\rm e}} \tag{7}$$

Respectively, the actual air/gas mass flow rate to the enclosure \dot{m}_{g}^{E} divided by the exhaust mass flow rate.

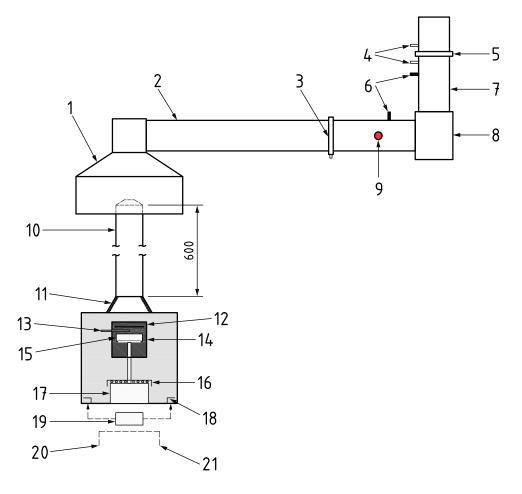
$$\tilde{\gamma} = \frac{\dot{m}_{\rm g}^{\rm E}}{\dot{m}_{\rm e}} \tag{8}$$

12.4.2 Heat release per unit area can be obtained from ISO 5660-1:2015, Formula (9).

13 Test report

The test report shall contain all of the applicable information described in ISO 5660-1:2015, Clause 13 and, additionally the following:

- a) intended test conditions: vitiation-controlled or ventilation-controlled;
- b) oxygen concentration in the enclosure during the test;
- c) (total) gas flow rate to the enclosure during the test;
- d) equilibration time after specimen loading;
- e) the time of test start.

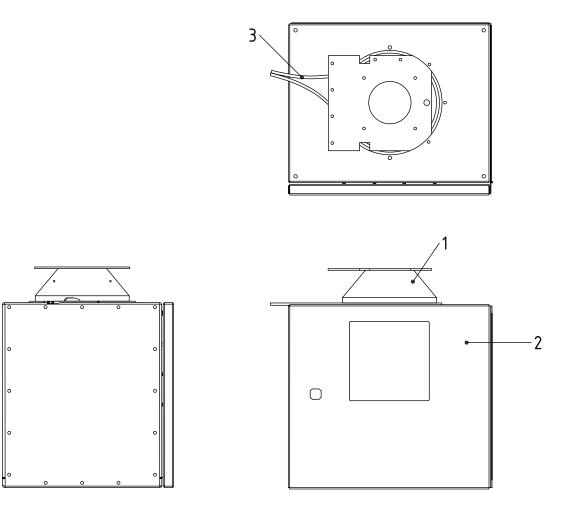


Кеу

- 1 exhaust hood
- 2 exhaust duct
- 3 sampling ring
- 4 pressure ports
- 5 orifice plate
- 6 thermocouple
- 7 stack
- 8 fan/blower
- 9 laser extinction beam
- 10 chimney
- 11 cone heater

- 12 radiation shield
- 13 ignition circuit
- 14 controlled-atmosphere chamber (enclosure)
- 15 sample holder/specimen
- 16 cooled shield
- 17 weighing device
- 18 baffles
- 19 mass flow meter
- 20 air
- 21 nitrogen

Figure 1 — Overview of the arrangement of enclosure, chimney and cone calorimeter according to ISO 5660-1



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Кеу

- 1 radiant heater
- 2 enclosure
- 3 water-cooled collar

Figure 2 — Overview of the enclosure (*top-right*: top view; *bottom right*: front view; *bottom left*: side view)

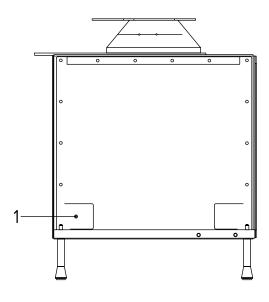
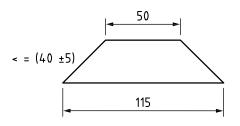
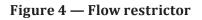




Figure 3 — Baffles at base of chamber





Annex A (informative)

Commentary and guidance notes for operators

A.1 Introduction

This annex aims to provide the test operator, and perhaps the user of the test results, with background information on the method, the apparatus and the data obtained. The information given in ISO 5660-1:2015, Annex A should also be considered.

A.2 Observation of secondary burning

If either attached or detached flames have been observed beyond the chimney during a test, secondary burning cannot be excluded. As potential exterior influences may be effective, then test conditions should be adjusted to prevent flames from occurring beyond the chimney or the data should not be reported. Test conditions can be adjusted as follows:

- a) Reduce the external irradiance in order to reduce the burning rate of the sample and, thus, to reduce the heat release rate and flame length accordingly.
- b) Reduce the sample surface in order to reduce the burning surface.
- c) Extend the chimney length used to provide appropriate containment for the actual flame length.

A.3 Characterisation of vitiation and ventilation conditions

Both, vitiation and ventilation conditions can be characterised in terms of the Global Equivalence Ratio $\Phi_{\rm GER}$.

$$\phi_{\rm GER} = \frac{\dot{m}_{\rm f} / (\dot{m}_{\rm g}^{\rm E} \cdot X_{\rm O_2}^{\rm E})}{(\dot{m}_{\rm f} / (\dot{m}_{\rm g}^{\rm E} \cdot X_{\rm O_2}^{\rm E}))_{\rm sto}}$$
(9)

It expresses the ratio of the ratio of fuel mass released from the specimen (simplified as burning rate, $\dot{m}_{\rm f}$) to the oxygen mass introduced from supplied air/gas, divided by that mass ratio required for stochiometric burning of the fuel.

With <u>Formula (9)</u>, variations in burning rate of some materials are captured also to describe time-specific vitiation and ventilation conditions.

Annex B

(informative)

Additional information for using the linked configuration

B.1 General

When using a configuration as described in $\underline{6.2.1}$ of this document (chimney/enclosure linked to the exhaust hood), the standard procedure as per ISO 5660-1:2015, 11.3 may be used with the following additional steps applied.

B.2 Procedure

Start with step <u>11.3.1</u> of this document.

Prior to collecting *baseline* data as per ISO 5660-1:2015, 11.3.1, apply steps <u>11.3.3</u> and <u>11.3.4</u> of this document. The total inlet gas flow rate to the enclosure should match with the exhaust flow rate to avoid under- or over-pressure within the enclosure. As in <u>11.2</u> of this document, the gas inlet flow should consequently not fall below (0,012 ± 0,002) m³/s.

After collecting baseline data per ISO 5660-1:2015, 11.3.1, continue with insertion of the radiation shield as in step <u>11.3.6</u> of this document and complete the rest of the paragraph.

Replace steps ISO 5660-1:2015, 11.3.2 and 11.3.3 with steps <u>11.3.7</u> through <u>11.3.8</u> of this document.

<u>11.4</u> a) of this document does not apply.

Calculate results per <u>Clause 12</u> and ISO 5660-1:2015, Annex G for a setup with additional gas analysis. Less gas analysis setups are not considered appropriate for measuring of heat release rates and production rate of gaseous products using this apparatus.

Annex C (informative)

Additional information for using the enclosure as standalone device with ISO 13927 controls

C.1 General

When undertaking anaerobic pyrolysis measurement close to 0 % of oxygen concentration, using the controlled-atmosphere enclosure alone without the oxygen depletion measurement equipment might be considered as an alternative test setup that limits the potential of extensive smoke deposit on high value equipment parts. In this particular case, controls of the apparatus specified in ISO 13927 should be utilized.

C.2 Arrangement with a mass loss calorimeter as per ISO 13927

The test enclosure described in <u>6.3</u> replaces the standard cone heater assembly in the ISO 13927 apparatus. The chimney housing in the thermopiles for heat release rate measurement should still be used. The controls according to ISO 13927 should be used to control the heater and ignition circuit and to allow collection of mass loss data and appropriate calibration procedures. Heat release rate data may be obtained by thermopile measurements and appropriate procedures described in ISO 13927.

Annex D (informative)

(informative)

Gas flow rates

D.1 General

Table D.1 shows flow rates of air and nitrogen gas respectively for different oxygen concentrations and for 150 L/min total inlet gas flow rate.

Table D.1 — Flow rates of air and nitrogen for different enclosure oxygen concentrations for 150 L/min total inlet gas flow rate

O2 Conc.Air flowNitrogen flor[%][L/min][L/min]20,95150,00,020,90149,60,420,80148,91,120,70148,21,820,60147,52,520,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,18,919,60140,39,719,50139,610,419,30138,211,819,20137,512,5	
20,95150,00,020,90149,60,420,80148,91,120,70148,21,820,60147,52,520,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00144,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,30138,211,8	w
20,90149,60,420,80148,91,120,70148,21,820,60147,52,520,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,18,919,70141,18,919,50139,610,419,40138,911,119,30138,211,8	
20,80148,91,120,70148,21,820,60147,52,520,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00144,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,30138,211,8	
20,70148,21,820,60147,52,520,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,50139,610,419,40138,911,119,30138,211,8	
20,60147,52,520,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,30138,211,8	
20,50146,83,220,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,30138,211,8	
20,40146,13,920,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
20,30145,34,720,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
20,20144,65,420,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
20,10143,96,120,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
20,00143,26,819,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
19,90142,57,519,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
19,80141,88,219,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
19,70141,18,919,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
19,60140,39,719,50139,610,419,40138,911,119,30138,211,8	
19,50139,610,419,40138,911,119,30138,211,8	
19,40138,911,119,30138,211,8	
19,30 138,2 11,8	
19,20 137,5 12,5	
19,10 136,8 13,2	
19,00 136,0 14,0	
18,90 135,3 14,7	
18,80 134,6 15,4	
18,70 133,9 16,1	
18,60 133,2 16,8	
18,50 132,5 17,5	
18,40 131,7 18,3	
18,30 131,0 19,0	
18,20 130,3 19,7	
18,10 129,6 20,4	

O2 Conc.	Air flow	Nitrogen flow
[%]	[L/min]	[L/min]
17,90	128,2	21,8
17,80	127,4	22,6
17,70	126,7	23,3
17,60	126,0	24,0
17,50	125,3	24,7
17,40	124,6	25,4
17,30	123,9	26,1
17,20	123,2	26,8
17,10	122,4	27,6
17,00	121,7	28,3
16,90	121,0	29,0
16,80	120,3	29,7
16,70	119,6	30,4
16,60	118,9	31,1
16,50	118,1	31,9
16,40	117,4	32,6
16,30	116,7	33,3
16,20	116,0	34,0
16,10	115,3	34,7
16,00	114,6	35,4
15,90	113,8	36,2
15,80	113,1	36,9
15,70	112,4	37,6
15,60	111,7	38,3
15,50	111,0	39,0
15,40	110,3	39,7
15,30	109,5	40,5
15,20	108,8	41,2
15,10	108,1	41,9
15,00	107,4	42,6

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02 COIIC.	All HOW	Microgen In
[%]	[L/min]	[L/min]
18,00	128,9	21,1
14,80	106,0	44,0
14,70	105,3	44,7
14,60	104,5	45,5
14,50	103,8	46,2
14,40	103,1	46,9
14,30	102,4	47,6
14,20	101,7	48,3
14,10	101,0	49,0
14,00	100,2	49,8
13,90	99,5	50,5
13,80	98,8	51,2
13,70	98,1	51,9
13,60	97,4	52,6
13,50	96,7	53,3
13,40	95,9	54,1
13,30	95,2	54,8
13,20	94,5	55,5
13,10	93,8	56,2
13,00	93,1	56,9
12,90	92,4	57,6
12,80	91,6	58,4
12,70	90,9	59,1
12,60	90,2	59,8
12,50	89,5	60,5
12,40	88,8	61,2
12,30	88,1	61,9
12,20	87,4	62,6
12,10	86,6	63,4
12,00	85,9	64,1
11,90	85,2	64,8
11,80	84,5	65,5
11,70	83,8	66,2
11,60	83,1	66,9
11,50	82,3	67,7
11,40	81,6	68,4
11,30	80,9	69,1
11,20	80,2	69,8
11,10	79,5	70,5
11,00	78,8	71,2
10,90	78,0	72,0
10,80	77,3	72,7
10,70	76,6	73,4

O2 Conc.

Air flow

Nitrogen flow

O2 Conc.	Air flow	Nitrogen flow
[%]	[L/min]	[L/min]
14,90	106,7	43,3
9,80	70,2	79,8
9,70	69,5	80,5
9,60	68,7	81,3
9,50	68,0	82,0
9,40	67,3	82,7
9,30	66,6	83,4
9,20	65,9	84,1
9,10	65,2	84,8
9,00	64,4	85,6
8,90	63,7	86,3
8,80	63,0	87,0
8,70	62,3	87,7
8,60	61,6	88,4
8,50	60,9	89,1
8,40	60,1	89,9
8,30	59,4	90,6
8,20	58,7	91,3
8,10	58,0	92,0
8,00	57,3	92,7
7,90	56,6	93,4
7,80	55,8	94,2
7,70	55,1	94,9
7,60	54,4	95,6
7,50	53,7	96,3
7,40	53,0	97,0
7,30	52,3	97,7
7,20	51,6	98,4
7,10	50,8	99,2
7,00	50,1	99,9
6,90	49,4	100,6
6,80	48,7	101,3
6,70	48,0	102,0
6,60	47,3	102,7
6,50	46,5	103,5
6,40	45,8	104,2
6,30	45,1	104,9
6,20	44,4	105,6
6,10	43,7	106,3
6,00	43,0	107,0
5,90	42,2	107,8
5,80	41,5	108,5
5,70	40,8	109,2

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O2 Conc.	Air flow	Nitrogen flow
[%]	[L/min]	[L/min]
10,60	75,9	74,1
10,50	75,2	74,8
10,40	74,5	75,5
10,30	73,7	76,3
10,20	73,0	77,0
10,10	72,3	77,7
10,00	71,6	78,4
9,90	70,9	79,1
2,50	17,9	132,1
2,40	17,2	132,8
2,30	16,5	133,5
2,20	15,8	134,2
2,10	15,0	135,0
2,00	14,3	135,7
1,90	13,6	136,4
1,80	12,9	137,1
1,70	12,2	137,8
1,60	11,5	138,5
1,50	10,7	139,3
1,40	10,0	140,0
1,30	9,3	140,7

Table D.1 (continued)

O2 Conc.	Air flow	Nitrogen flow
[%]	[L/min]	[L/min]
5,60	40,1	109,9
5,50	39,4	110,6
5,40	38,7	111,3
5,30	37,9	112,1
5,20	37,2	112,8
5,10	36,5	113,5
5,00	35,8	114,2
4,90	35,1	114,9
1,20	8,6	141,4
1,10	7,9	142,1
1,00	7,2	142,8
0,90	6,4	143,6
0,80	5,7	144,3
0,70	5,0	145,0
0,60	4,3	145,7
0,50	3,6	146,4
0,40	2,9	147,1
0,30	2,1	147,9
0,20	1,4	148,6
0,10	0,7	149,3
0,00	0,0	150,0

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