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Plastics — Determination of burning behaviour by oxygen index —

Part 4: High gas velocity test

Plastiques — Détermination du comportement au feu au moyen de l'indice d'oxygène —

Partie 4: Essai à vitesse élevée de gaz

Reference number
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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 4, *Burning behaviour*.

A list of all parts in the ISO 4589 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.

Introduction

This document has been prepared to extend the test methods available for the determination of flammability by oxygen index to higher gas velocity of oxygen/nitrogen mixture to which plastic materials can be exposed in a service situation where the gas velocity is higher than that specified in ISO 4589-2. The gas velocity at the position of the test specimen is measured prior to the test.

The output of the test described in this document can be used, for example, in the evaluation of the burning behaviour of plastics materials used in circumstances where forced ventilation air flow governs the supply of oxygen to the fire. See References [10] to [16].

Plastics — Determination of burning behaviour by oxygen index —

Part 4: High gas velocity test

1 Scope

This document specifies a test method for determining the minimum volume fraction of oxygen, in admixture with nitrogen, at ambient temperature, that supports combustion of small vertical sheet test specimen under a specified gas velocity that is higher than that specified in ISO 4589-2.

NOTE The result is expressed as a high gas velocity oxygen index (HOI).

In addition, this document specifies the testing apparatus for determining the HOI.

The test method is applicable to materials in the form of sheets up to 2 mm thick. It is also applicable to flexible sheet materials that are supported vertically by a specified specimen holder.

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 291:2008, *Plastics — Standard atmospheres for conditioning and testing*

ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

ISO 2859-2, *Sampling procedures for inspection by attributes — Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*

ISO 13943, *Fire safety — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in 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
high gas velocity oxygen index
HOI
minimum volume fraction of oxygen, in a mixture of oxygen and nitrogen, at a specified gas velocity that supports flaming combustion of a material under specified test conditions

Note 1 to entry: The specified gas velocity is greater than 40 mm/s and is typically between 600 mm/s and 1 000 mm/s.

Note 2 to entry: The HOI is usually expressed as a percentage, at the gas velocity used (e.g. HOI = 34,6 %, 800 mm/s).

4 Principles for determination of HOI

A small test specimen is supported vertically in a mixture of oxygen and nitrogen that flows upwards through a transparent chimney. The volume fraction of oxygen in the gas mixture is pre-determined, controlled and measured. The vertical velocity of the gas mixture gas is pre-determined, controlled and measured. The upper end of the specimen is ignited, and its subsequent burning behaviour is observed in order to compare the burnt length of the specimen, with respect to the limits specified. The HOI is determined from a series of tests using different volume fractions of oxygen (see 8.7).

5 Apparatus

5.1 Test chimney, which shall consist of a heat-resistant glass tube supported vertically on a base through which the oxygen/nitrogen gas mixture can be introduced (see Figure 1).

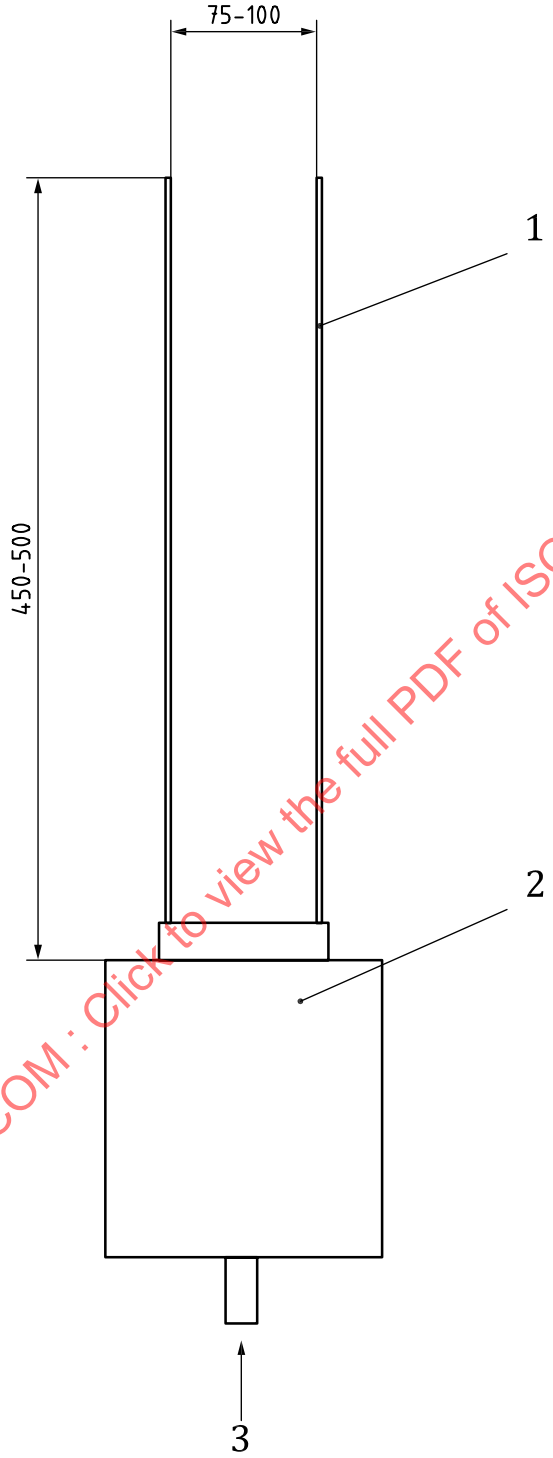
The recommended dimensions of the chimney are 450 mm to 500 mm in height and an inside diameter of 75 mm to 100 mm. The upper outlet shall be open without any restriction.

NOTE It has been found that the outlet cup defined in ISO 4589-2:2017, 5.1 creates flow turbulence in the chimney at a high gas velocity.

A chimney having a height more than 500 mm or less than 450 mm can be used, if it is shown to give the specified gas velocity within the permitted limit of variance.

The lower end of the chimney, or the base upon which the chimney is supported, shall incorporate a device for evenly distributing the gas mixture entering the chimney as shown in Figure 1. The mounting of a porous screen below the level of the specimen holder is helpful to prevent falling combustion debris from fouling the gas entry and distribution paths. One option is to construct the chimney in such a way that it can be divided in half, so as to make the setting of samples and cleaning easier.

Dimension in millimetres



Key

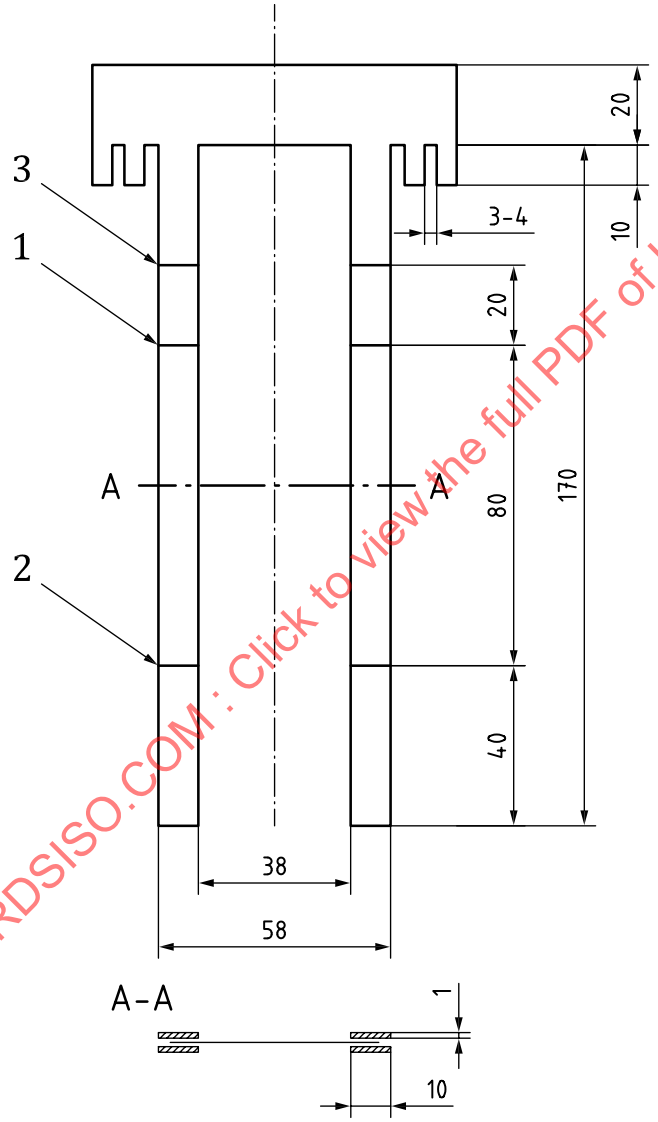
1 chimney glass tube
2 gas flow controlling chamber
3 inlet of oxygen/nitrogen gas mixture

Figure 1 — Typical chimney arrangement

5.2 Specimen holder, which shall be suitable for supporting a specimen vertically in the centre of the chimney.

The specimen holder can be hanged from the upper edge of the chimney (see Figure 2) or supported by a vertical rod raised from the base of the chimney (see Figure 3). The specimen shall be supported by the vertical edges of the frames of the specimen holder. The vertical frames of the specimen holder shall have reference marks at 20 mm, 100 mm and 140 mm as shown in Figure 2 and Figure 3. The surfaces of the specimen holder and its support shall be smooth in order to minimize the turbulence of the gas flow in the chimney.

Dimensions in millimetres
with tolerances ± 0,25 mm



Key

1 upper reference mark

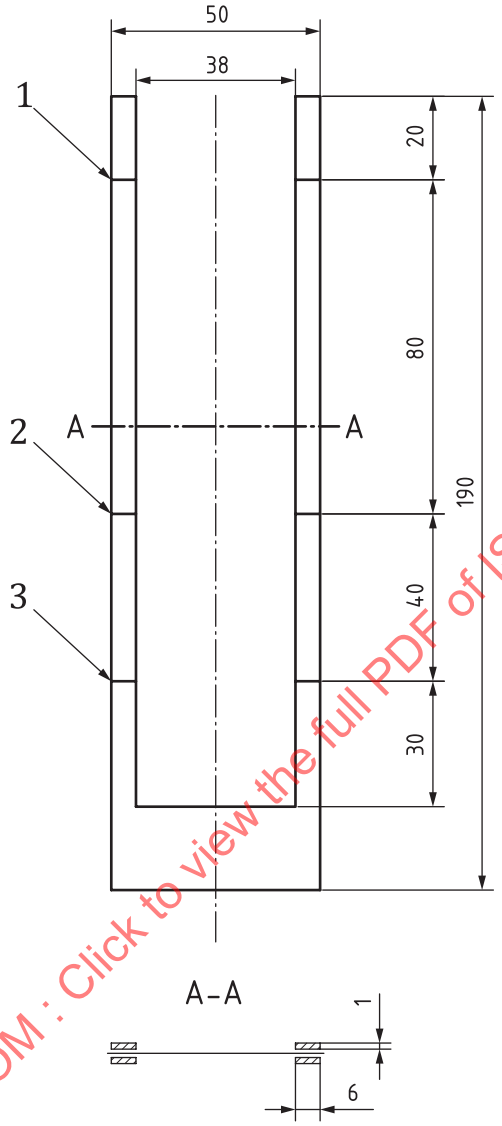
2 lower reference mark

3 positioning mark for the top edge of the test specimen

NOTE The test specimen is held securely along both upright edges between forks made of stainless steel.

Figure 2 — Suspended specimen holder (Option A hanging specimen holder)

Dimensions in millimetres
with tolerances ± 0,25 mm



Key

1 upper reference mark
2 lower reference mark
3 positioning mark for the bottom edge of the test specimen

NOTE The test specimen is held securely along both upright edges between forks made of stainless steel.

Figure 3 — Supported specimen holder
(Option B: Specimen holder supported from the bottom)

5.3 Gas supplies, which shall be suitable for supporting an oxygen/nitrogen gas mixture with a gas velocity of up to 1 000 mm/s in the chimney. The gas velocity shall be within 50 mm/s of the desired value.

The supply rate of oxygen/nitrogen gas mixture in this document is greater than that specified in ISO 4589-2 and it is necessary to have higher gas supply capability than that for ISO 4589-2.

5.4 Gas control system, which shall be suitable for setting and adjusting the volume fraction of oxygen in a gas mixture entering into the chimney with a resolution of 0,1 % and an accuracy of ±0,2 % when the

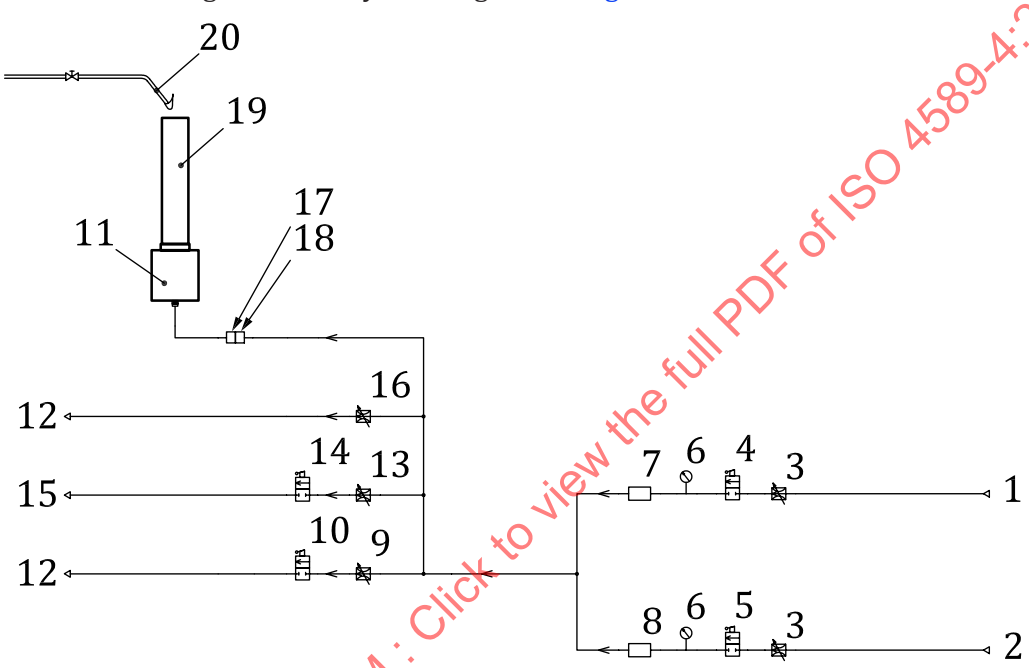
gas velocity of the mixture within the chimney is 800 mm/s ± 50 mm/s at 23 °C ± 2 °C. The gas velocity shall be controllable within the range of 600 mm/s to 1 000 mm/s.

The gas control system shall also be capable of supplying the oxygen/nitrogen gas mixture at the gas velocity of 100 mm/s or less for the ignition stage of the test (see 8.3).

Means shall be provided for ensuring that the temperature of the gas mixture entering into the chimney is 23 °C ± 2 °C. If such means involves an internal probe, its position and profile shall be designed to minimize the turbulence within the chimney.

NOTE It has been found that the temperature measurement can be done by a device specified in 5.9 and/or 5.10.

An example of the suitable gas control system is given in Figure 4.



Key

1	N_2 supply port	11	gas flow controlling chamber
2	O_2 supply port	12	bypass outlet
3	pressure regulator	13	flow rate control valve
4	N_2 supply valve	14	open/shut valve
5	O_2 supply valve	15	mixed gas sampling port
6	pressure gauge	16	flow rate control valve
7	digital mass flow controller [for N_2]	17	coupler (plug)
8	digital mass flow controller [for O_2]	18	coupler (socket)
9	bypass flow rate control valve	19	test chimney glass tube
10	open/shut valve	20	igniter

Figure 4 — Example of diagram of gas control system

5.5 Oxygen analyser, which shall be suitable for measuring the volume fraction of oxygen in the gas mixture entering the chimney with a resolution of 0,1 % and an accuracy of ±0,1 % of the mixture.

NOTE 1 It has been found that paramagnetic oxygen analysers meet the accuracy requirements.

NOTE 2 In case the volume fraction of oxygen around the position of the specimen differs from that controlled at the air supply line, troubleshooting will be necessary.

5.6 Flame igniter, which shall comprise a tube that can be inserted into the chimney to apply to the test specimen a flame issuing from an outlet of 2 mm ± 1 mm diameter at the end of the tube.

The flame fuel shall be propane, without premixed air. The purity of the propane gas shall be at least 98 %. The fuel supply shall be adjusted so that, for the set volume fraction of oxygen, the total flame height is 16 mm ± 4 mm when the tube is vertical within the chimney.

Use of a flame height gauge is recommended.

5.7 Timing device, which shall be capable of measuring periods up to 5 min with an accuracy of ±0,5 s.

5.8 Fume extraction system, which shall be capable of providing sufficient ventilation or exhaust to remove fumes or soot expelled from the chimney without disrupting the gas flow rate or temperatures in the chimney.

5.9 Gas velocity measurement device, i.e. anemometer, which shall be capable of measuring vertical gas velocity at the specimen position in the test chimney and have a resolution of 0,01 m/s.

NOTE A hot-wire anemometer has been found suitable for this purpose. It has been found that this device usually can also measure gas temperature as specified in 5.10.

5.10 Gas temperature device. The temperature measurement device for measuring the gas temperature in the chimney shall have an accuracy and a resolution of 0,5 K.

6 Calibration of equipment

For conformance with this method, calibrate the equipment periodically in accordance with the instructions given in Annex A so that the maximum interval between recalibration and the use conforms to the periods stated in Table 1.

Table 1 — Equipment calibration frequencies

Item	Maximum period between calibrations
Leak tests for gas system joints (as required by A.1)	
a) For joints disturbed during use or cleaning of the apparatus	Immediately
b) For undisturbed equipment	6 months
Gas flow rate (as required by A.2)	6 months
Gas velocity measurement device (as required by A.3)	1 year
Oxygen analyser (as required by A.4)	1 week
Mass flow controller (as required by A.5)	1 year
Gas temperature measurement device (as required by A.6)	1 year

7 Preparation of test specimens

7.1 Test specimen form

Of the six specimen forms listed in ISO 4589-2:2017, Table 2, only one form “V” is used in this test method. Test specimen form “V” is flexible film or sheet and shall have the length of (140^{+0}_{-5}) mm and width of (52 ± 0,5) mm. The thickness of twat specimen shall be up to 2 mm.

7.2 Sampling

Obtain a sample sufficient for preparation of at least 15 test specimens for tests in a gas velocity. The sample shall be taken in accordance with the material specification. Otherwise, it shall be taken in accordance with ISO 2859-1 or ISO 2859-2, as applicable.

7.3 Test specimen dimensions and preparation

The test specimen shall be made by any applicable procedures that comply with the appropriate material specification (see NOTE 1) or ISO methods (see NOTE 2) for preparing specimens, mould or cut test specimens.

NOTE 1 Some material specifications can require selection and identification of the "state of the test specimen" used (e.g. in a "defined state" or "basic state" for a styrene-based polymer or copolymer).

NOTE 2 In the absence of a relevant specification, one or more procedures in ISO 293, ISO 294, ISO 295, ISO 2818 or ISO 3167 can be used.

Ensure that the surfaces of specimens are clean and free from flaws that could affect burning behaviour (e.g. peripheral moulding flash or burrs from machining).

Note the position and orientation of test specimens with respect to any asymmetry in the sample material (see NOTE 3).

NOTE 3 HOI can be significantly affected by differences in the ease of ignition or burning behaviour, due to material inhomogeneity (e.g. different levels of shrinkage when heated for specimens cut in different directions from asymmetrically oriented thermoplastics film).

7.4 Marking of test specimen

The test specimens shall be marked at 20 mm and 100 mm from the end at which the specimen is to be ignited.

7.5 Conditioning

The test specimens shall be conditioned for 24 h at 23 °C ± 2 °C and (50 ± 5) % relative humidity. After 24 h conditioning, if the stability of the mass of the test specimen reaches to 0,1 % or less changes, then allow to conduct test. Otherwise, follow ISO 291:2008, 8.1, to keep specimen in conditioning chamber for at least 88 h (maximum duration is 88 h).

Test specimens of cellular materials that may contain volatile flammable material should preferably be purged of such volatile material prior to conditioning at 23 °C and 50 % relative humidity. Test specimens may be purged satisfactorily by pre-conditioning at 60 °C in suitable ventilated ovens for 168 h. Larger blocks of such materials may require longer pre-treatment. It is important that facilities for cutting test specimens from cellular material that may contain volatile flammable material are suitable for the hazards involved.

8 Procedure for determination of HOI

8.1 Setting up the apparatus

8.1.1 Maintain ambient temperature for the test apparatus at 23 °C ± 2 °C. If necessary, keep the test specimens in an enclosure at 23 °C ± 2 °C and relative humidity (50 % ± 5 %) from which each test specimen can be taken when required.

8.1.2 Recalibrate equipment components, if necessary (see Clause 6 and Annex A). Before conducting a test, clean the glass chimney to maintain good visibility. If necessary, clean the gas inlets or inlet screen, and the temperature sensor (if fitted).

8.1.3 Ensure that the test chimney is set in vertical (see Figure 1).

8.2 Setting volume fraction of oxygen and gas flow velocity

8.2.1 Select an initial volume fraction of oxygen to be used. When possible, base this volume fraction on past results for similar materials. Alternatively, try to ignite a test specimen in air, and note the burning behaviour. If the test specimen burns rapidly, select an initial volume fraction of about 18 % oxygen; if the specimen burns gently or unsteadily, select an initial oxygen volume fraction of about 21 %; if the specimen does not continue to burn in air, select an initial volume fraction of at least 25 %, depending on the difficulty of ignition or the period of burning before extinguishing in air.

8.2.2 Select a gas velocity between 600 mm/s to 1 000 mm/s to which HOI is to be determined.

NOTE It is found suitable to conduct the tests with gas velocity of 600, 800 and 1 000 mm/s for determining blow-off behaviour in terms of volume fraction of oxygen. See Annex D.

8.2.3 Shut the bypass valve (device 10 in Figure 4). Set the gas mixing and flow controls for nitrogen and oxygen supply (devices 3 to 8 in Figure 4) so that an oxygen/nitrogen mixture at 23 °C ± 2 °C containing the pre-determined volume fraction of oxygen is flowing at the selected gas velocity within the chimney.

8.2.4 Measure the volume fraction of the oxygen using oxygen analyser (see 5.5) which samples the mixture gas at the gas sampling port (device 15 of Figure 3). Record the volume fraction of oxygen.

8.2.5 The gas velocity in the test chimney at the level of top of the specimen and 20 mm horizontally from the centre of the chimney shall be measured by the gas flow velocity measurement device as specified in 5.9. The gas velocity shall be within 50 mm/s of the desired value. The gas velocity can be controlled by the flow rate control valve (device 16 in Figure 3).

8.2.6 Once the pre-determined volume fraction of oxygen and gas velocity are obtained, keep left the mass flow controller of oxygen and nitrogen untouched (device 7 and 8 in Figure 3). For shutting the flow of oxygen and nitrogen, use supply valves of these gases (device 4 and 5 in Figure 3).

8.3 Procedure for ignition of the test specimen

8.3.1 Mount a test specimen vertically in the centre of the chimney so that the top of the test specimen is at least 30 mm below the open top of the chimney, and the lowest exposed part of the test specimen is at least 100 mm above the top of the gas distribution device at the base of the chimney.

8.3.2 Open the bypass valve (device 10 in Figure 3) and set the gas velocity to 100 mm/s or less (see 5.4).

8.3.3 Supply oxygen and nitrogen to the chimney by opening the gas supply valves (device 4 and 5 in Figure 3). Let the gas flow to purge the test chimney for at least 20 s prior to the ignition of the test specimen and maintain the flow without change during the ignition.

8.3.4 After the purge, ignite the test specimen using flame ignitor (see 5.6) to burn only on the top surface of the test specimen by sweeping the ignitor flame along the top surface of the test specimen. This ignition process shall not be continued for more than 30 s.

8.3.5 After ignition of the test specimen, shut the bypass valve (device 10 in Figure 3) so that the gas velocity in the chimney becomes the selected velocity.

8.3.6 Consider the specimen to be ignited, for the purpose of measuring the extent of burning, as soon as any part of the visible burning portion reaches the level of the upper reference mark.

8.3.7 Observe the burning behaviour of the test specimen and record if the limit of burning was or was not exceeded (see 8.4) at the pre-determined volume fraction of oxygen and the pre-determined gas velocity.

8.4 Assessing the burning behaviour of test specimen

When the combustion continues and exceed the lower reference level, note the burning behaviour accordingly, and extinguish the flame. This is recorded as an "X" response.

When the combustion does not exceed the lower reference level, note the burning behaviour accordingly. This is recorded as an "O" response.

NOTE The lower reference level is 40 mm above the bottom edge of the test specimen.

8.5 Selecting successive volume fraction of oxygen

The procedure described in 8.6 and 8.7 is based upon Reference [17] using the specific case where $N_T - N_L = 5$ (see 8.7.2 and 8.7.3), with an arbitrary step size for certain changes to be made in the oxygen volume fraction used.

During the testing, select the oxygen volume fraction to be used for testing the next test specimen as follows:

a) decrease the oxygen volume fraction if the burning behaviour of the preceding specimen gave an "X" response;

otherwise

b) increase the oxygen volume fraction if the preceding specimen gave an "O" response.

Choose the size of the change in oxygen volume fraction in accordance with 8.6 or 8.7, as appropriate.

8.6 Determining the preliminary volume fraction of oxygen

Repeat the procedures specified in 8.3 to 8.5 inclusive, using volume fraction of oxygen changes of any convenient step size, until the volume fractions of oxygen have been found that differ by ≤ 1,0 % and of which one gave an "O" response and the other an "X" response. From this pair of volume fractions of oxygen, note that which gave the "O" response as the preliminary volume fraction of oxygen level and then proceed in accordance with 8.7.

NOTE 1 The two results, at volume fractions of oxygen ≤1,0 % apart, which give opposite responses need not be from successive specimens.

NOTE 2 The volume fraction which gave the "O" response does not have to be lower than that which gave the "X" response.

NOTE 3 A format convenient for recording the information required by this and subsequent clauses is illustrated in Annex B.

8.7 Volume fraction of oxygen changes

8.7.1 Using the preliminary volume fraction of oxygen (see 8.6), test one specimen by repeating 8.3 to 8.4 inclusive. Record both the oxygen volume fraction (co) used and the response, "X" or "O", as the first of the N_L and of the N_T series of results.

8.7.2 Change the volume fraction of oxygen, in accordance with 8.5, using volume fraction changes (d) of 0,2 % (see 8.7.2.2) of the total gas mixture to test further specimens in accordance with 8.3 to

8.5 inclusive, noting the values of *c*o and the corresponding responses until a different response to that obtained in 8.7.1 is recorded.

8.7.2.1 The result from 8.7.1, plus those of like response from 8.7.2, constitute the N_L series of results (see example in B.3).

8.7.2.2 Where experience has shown that the requirements of 8.7.4 are usually satisfied by a value of d other than 0,2 %, that value may be selected as the initial value of d.

8.7.3 Test four more specimens, in accordance with 8.3 to 8.5 inclusive, maintaining d = 0,2 %; and note the volume fraction of oxygen *c*o used, and response of, each specimen. Designate the volume fraction of oxygen used for the last specimen as c_f.

These four results, together with the last result from 8.7.2 (i.e. that which differed in response from that of 8.7.1), constitute the remainder of the N_T series, as shown in Formula (1):

$$N_T = N_L + 5 \quad (1)$$

where

N_L is the series of "X" or "O" results;

N_T is the series of "X" or "O" results plus five.

See example in B.3.

8.7.4 Calculate the estimated standard deviation, σ, of the oxygen volume fraction measurements from the last six responses in the N_T series (including c_f), in accordance with Formula (4). If the condition shown as Formula (2):

$$2\sigma/3 < d < 1{,}5\sigma \quad (2)$$

is satisfied, calculate the HOI in accordance with Formula (3); otherwise

a) if $d < 2\sigma/3$, repeat steps 8.7.2 to 8.7.4, using increased values for d, until the condition is satisfied; or

b) if $d > 1{,}5\sigma$, repeat steps 8.7.2 to 8.7.4, using decreased values for d, until the condition is satisfied, except that d shall not be reduced below 0,2 unless so required by the relevant material specification.

9 Calculation and expression of results

9.1 Calculation of the HOI

$$\text{HOI} = c_f + k\,d \quad (3)$$

where

c_f is the final value of the volume fraction of oxygen, reported in three decimal places, used in the series of N_T measurements in accordance with 8.7;

d is the interval, to at least three decimal places, between the oxygen volume fraction levels used and controlled in accordance with 8.7;

k is a factor to be obtained from Table 2.

For the purposes of calculating σ as required by 8.7.4 and 9.3, the HOI shall be calculated up to four decimal places (i.e. two decimal places when HOI is expressed as a percentage).

For the purposes of reporting HOI results, express HOI values to the nearest 0,001 (i.e. to the nearest 0,1 when the HOI is expressed as a percentage), with actual intermediate results being rounded down.

Table 2 — Values of *k* for calculating the HOI from determinations made by Dixon's "up-and-down method"

1	2	3	4	5	6
Responses for the last five measurements	**Values of k for which the first N_L determination are**				
	a) O	OO	OOO	OOOO	
XOOOO	−0,55	−0,55	−0,55	−0,55	OXXXX
XOOOX	−1,25	−1,25	−1,25	−1,25	OXXXO
XOOXO	0,37	0,38	0,38	0,38	OXXOX
XOOXX	−0,17	−0,14	−0,14	−0,14	OXXOO
XOXOO	0,02	0,04	0,04	0,04	OXOXX
XOXOX	−0,50	−0,46	−0,45	−0,45	OXOXO
XOXXO	1,17	1,24	1,25	1,25	OXOOX
XOXXX	0,61	0,73	0,76	0,76	OXOOO
XXOOO	−0,30	−0,27	−0,26	−0,26	OOXXX
XXOOX	−0,83	−0,76	−0,75	−0,75	OOXXO
XXOXO	0,83	0,94	0,95	0,95	OOXOX
XXOXX	0,30	0,46	0,50	0,50	OOXOO
XXXOO	0,50	0,65	0,68	0,68	OOOXX
XXXOX	−0,04	0,19	0,24	0,25	OOOXO
XXXXO	1,60	1,92	2,00	2,01	OOOOX
XXXXX	0,89	1,33	1,47	1,50	OOOOO
	Values of k for which the first N_L determinations are				Responses for the last five measurements
	b) X	XX	XXX	XXXX	
	are as given in this table opposite the appropriate response in column 6, but with the sign of k reversed (i.e. HOI = $c_f - kd$; see 9.1).				

9.2 Determination of *k*

The value and sign of k are dependent upon the pattern of the responses of specimens tested in accordance with 8.7, and shall be determined from Table 2 as follows:

a) if the response of the specimen tested in accordance with 8.7.1 was "O", so that the first contrary response (see 8.7.2) was an "X", refer to column 1 of Table 2 to select the row for which the last four response symbols correspond to those found when testing in accordance with 8.7.3. The value and sign of k will be that shown in column 2, 3, 4 or 5 for which the number of "O"s shown in row a) of Table 2 corresponds to the number of "O" responses found for the N_L series, in accordance with 8.7.1 and 8.7.2;

or

b) if the response of the specimen tested in accordance with 8.7.1 was "X", so that the first contrary response was an "O", refer to the sixth column of Table 2 to select the row for which the last four response symbols correspond to those found when testing in accordance with 8.7.3. The value of k will be that shown in column 2, 3, 4 or 5 for which the number of "X"s shown in row b) of Table 2 corresponds to the number of "X" responses found for the N_L series, in accordance with 8.7.1 and

8.7.2, but the sign of k is reversed, so that negative values shown in Table 2 for k become positive, and vice versa.

NOTE An example of the determination of k and the calculation of an HOI is given in Annex B.

9.3 Standard deviation of oxygen volume fraction measurements

For the purposes of 8.7.4, calculate the estimated standard deviation σ of the oxygen volume fraction measurements from the relationship as shown in Formula (4):

$$\sigma = \left[\sum_{i=1}^{n} (c_i - \mathrm{HOI})^2 / (n-1) \right]^{1/2} \tag{4}$$

where

c_i is the representation of, in turn, each of the oxygen volume fractions used during measurement of the last six responses in the N_T series of measurements;

HOI is the oxygen index value, calculated in accordance with 9.1;

n is the number of measurements of the oxygen volume fraction contributing to $\Sigma(c_i\text{-HOI})^2$.

NOTE For this method, n = 6 in accordance with 8.7.4. For n < 6, the method loses precision. For n > 6, different statistical criteria applies.

10 Precision of test results

An interlaboratory trial was conducted in 2019 among four laboratories in three nations using five materials to obtain the precision data of this test method. The results together with the precision data is described in Annex C for information.

11 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 4589-4:2021;

b) a statement that the test results relate only to the behaviour of the test specimens under the conditions of this test, and that these results shall not be used to infer the fire hazards of the materials in other forms or under other fire conditions;

c) all details necessary for identification of the material tested, including, where relevant, the type of material, its density, its previous history, and the specimen orientation with respect to any anisotropy in the material or sample;

d) the test specimen dimensions;

e) the igniter used, if other than the standard propane flame;

f) the HOI, value and gas velocity (e.g. HOI= 35,6 %, 800 mm/s);

g) the estimated standard deviation and oxygen volume fraction increment used, if other than 0,2 %;

h) a description of any relevant ancillary characteristics or behaviour, such as charring, dripping, severe shrinkage, erratic burning or afterglow;

i) any deviations from the requirements of this document.

Annex A
(normative)

Calibration of equipment

A.1 Leak tests

Leak tests shall be carried out thoroughly on all joints where leaks could change the volume fraction of oxygen levels in the chimney from the volume fraction levels set or indicated.

A.2 Gas flow rates

The mass flow controller shall be checked according to the manufacturer's recommendation.

A.3 Gas velocity

Gas velocity measurement device shall be calibrated by a comparison with calibrated gas velocity meter in an air flow test chamber.

A.4 Oxygen analyser

For zeroing, feed the analyser with oxygen-free nitrogen gas (purity 99,99 % or better), with the same flow rate and pressure as for the sample gases. Adjust the analyser response to (0,0 ± 0,2) %. Calibration shall be similarly achieved using the gas satisfying following conditions and adjusting the analyser response to the volume fraction of the oxygen source.

a) Oxygen gas with purity of 99,99 % or more.

b) Volume fraction of oxygen of gas for span adjustment should have volume fraction of oxygen higher than that of what is going to be measured, and the accuracy of volume fraction of that gas should be higher than ±0,2 %.

A.5 Mass flow controller

The mass flow controllers shall be calibrated by the manufacturer or by a qualified calibration laboratory.

A.6 Temperature measurement device

Temperature measurement device shall be calibrated by a comparison with calibrated temperature measurement device (thermos-couple) in a temperature chamber.

Annex B
(informative)

Example of test results sheet for HOI

B.1 Test results sheet for HOI

Material: Phenolic laminate

HOI (volume fraction, %): 29,5
(rounded to 0,1 %)

Specimen form: V (1 mm thick)

Testing flow velocity (mm/s): 800

Ignition procedure:

σ : 0,152

Conditioning procedure: 23 23 °C /50 RH%

Date of test: 2016-05-24

Oxygen volume fraction increment (d): 0,2 %

Laboratory No.: 19 Test No.: 1

B.2 Determination of oxygen volume fraction for one pair of "X" and "O" responses at ≤1 % O_2 volume fraction interval

See 8.6.

Oxygen volume fraction, %	25,0	35,0	30,0	32,0	31,0				
Length burnt, mm	0	>80	62	>80	>80				
Response ("X" or "O")	O	X	O	X	X				

Oxygen volume fraction of the "O" response for the relevant pair (see 8.6) = 30,0 % (This is the volume fraction to be used again for the first measurement in B.3.).

B.3 Determination of HOI

See 8.7.

Step size to be used for successive changes *d* in oxygen volume fraction = 0,2 % (initially to be 0,2 %, unless otherwise instructed).

	N_T series measurements											
	N_L series measurements (8.7.1 and 8.7.2)							(8.7.3)				c_f
Oxygen volume fraction, %	30,0	29,8	29,6	29,4				29,4	29,6	29,4	29,6	29,8
Length burnt, mm	>80	>80	>80	60				60	>80	58	64	>80
Response ("X" or "O")	X	X	X	O				O	X	O	O	X
	Column (2, 3, 4 or 5): 4							Row (1 to 16): 7				
	k value from Table 2; 1,25											
						Hence k = –1,25						

HOI = $c_f + kd$ = 29,8 + (–1,25 × 0,2)

= 29,5 % (to one decimal place, for reporting HOI)

= 29,55 % (to two decimal places, for calculation of and verification of d as required in B.4.)

k is determined by 9.2.

B.4 Verification of step size d % oxygen volume fraction

See 8.7.4 and 9.3.

Last six results		Oxygen volume fraction, %			
		c_i [a]	HOI	c_i – HOI	$(c_i - \text{HOI})^2$
c_f	1	29,8	29,55	0,25	0,062 5
	2	29,6	29,55	0,05	0,002 5
	3	29,4	29,55	–0,15	0,022 5
	4	29,6	29,55	0,05	0,002 5
	5	29,4	29,55	–0,15	0,022 5
n	6	29,6	29,55	0,05	0,002 5
Total $\sum(c_i - \text{HOI})^2$					0,115

[a] Column c_i contains the oxygen volume fractions used for the measurements of c_f and for each of the 5 preceding measurements, for n = 6.

Estimation of standard deviation

$$\sigma = \left[\sum_{i=1}^{n} (c_i - \text{HOI})^2 / (n-1)\right]^{\frac{1}{2}}$$

= $(0,115/5)^{1/2}$ = 0,152

$2\sigma/3$ = 0,101

d = 0,2

$1,5\sigma$ = 0,227

If $2\sigma/3 < d < 1,5\sigma$, or if $0,2 = d > 1,5\sigma$, HOI is valid.

If $2\sigma/3 > d$, repeat B.3 using a larger value for d.

If $1,5\sigma < d$, repeat B.3 using smaller value for d.

Then verify the step size again, making further changes to the step size as necessary until one of the verification relationships is satisfied.

B.5 Ancillary information

a) These test results relate only to the behaviour of the specimens under the conditions of this test. These results shall not be used to infer the relative hazards presented by differing materials or shapes under these or other fire conditions.

b) Special material history/characteristics, if applicable.

c) Variations from standard procedure, if applicable.

d) Description of observed burning behaviour.

e) Results measured/reported by.

Annex C
(informative)

Interlaboratory test data on HOI measurement

An interlaboratory test was conducted in 2019 to assess the effects of different type specimens. Four laboratories in three nations (as indicated laboratory A, B, C and D) participated in this exercise, using type V test specimens of five materials (as indicated test specimen V, W, X, Y and Z).

The performance of all test equipment at each laboratory was checked as stipulated in Annex A prior to conducting this trial.

Each laboratory conducted the tests with two replicate sets on each material.

Due to the small number of the participating laboratories, the test results were analysed according to ASTM E2653[18].

As specified in 9.3, the estimated standard deviation of the oxygen volume fraction measurements σ at each laboratory for tests on each material were obtained. The repeatability r was calculated as $r = 2{,}8\ \sigma$.

Tables C.1 through C.3 show the results on individual test specimen.

Table C.1 — Precision data at gas velocity 600 mm/s

Material	Type	Thickness mm	Average HOI %	Repeatability, r %	Reproducibility, R %
V	Film	0,125	19,0	0,7	1,1
W	Textile	0,30	30,8	0,4	1,3
X	Textile	0,40	30,6	0,2	1,2
Y	Film	0,25	22,6	0,7	2,0
Z	Film	0,125	44,2	2,5	3,8

Table C.2 — Precision data at gas velocity 800 mm/s

Material	Type	Thickness mm	Average HOI %	Repeatability, r %	Reproducibility, R %
V	Film	0,125	19,6	1,6	1,9
W	Textile	0,30	31,1	1,0	1,2
X	Textile	0,40	30,7	0,7	1,4
Y	Film	0,25	23,4	0,7	2,9
Z	Film	0,125	44,7	0,8	4,0

Table C.3 — Precision data at gas velocity 1 000 mm/s

Material	Type	Thickness mm	Average HOI %	Repeatability, *r* %	Reproducibility, *R* %
V	Film	0,125	20,0	1,1	1,2
W	Textile	0,30	31,4	0,5	1,1
X	Textile	0,40	31,0	0,5	0,9
Y	Film	0,25	24,0	0,6	2,7
Z	Film	0,125	44,8	0,7	4,6

Annex D
(informative)

Blow-off behaviour at high gas velocity — How to predict the flammability of materials

D.1 Background

Recently, human activities in space have become more extensive and long-term manned missions, such as the construction of a moon base and the exploration of Mars, are being planned as international cooperative missions. In such plans, one of the most important tasks is to guarantee fire safety in microgravity and planetary habitation environments. In the past several decades, extensive research on flame spread over solid materials in microgravity environments has been conducted and many interesting phenomena have been reported. The flammability limit of solid materials in opposed flow is one of the major topics concerning fire safety. An important measure to discuss the flammability of a material is its limiting oxygen concentration (LOC). The LOC is defined as the oxygen concentration below which the material cannot sustain a flame under the conditions considered. It may vary depending on the flow speed and direction and the sample thickness. From the trend of a material's LOC, the flammability map of the material can be obtained. It has been found that the LOC of poly-methyl methacrylate (PMMA) sheets is lower in opposed flow velocities lower than the buoyant flow. [10] Such extension of the flammability limit is one of the notable characteristics observed in micro or reduced gravity environments. However, the flammability limit of a solid material is not always extended under low flow velocities; some flame retardant materials have higher LOCs in microgravity than in normal gravity. Hence, the flammability behaviour of a material in slow opposed flows should be discussed carefully, and it is important consider whether the flammability limit of the material under consideration expands.

Currently, the Japan Aerospace Exploration Agency (JAXA) is conducting the Flammability Limits at Reduced-g Experiment (FLARE) project, an orbital flight experiment in the International Space Station, in collaboration with the National Aeronautics and Space Administration (NASA), the European Space Agency (ESA), and several universities. One of the objectives of the FLARE project is to develop a simplified model for evaluating the LOC of a thin solid material, and expressing it as an index, in microgravity environments for fire safety in space environments. Using the simplified model developed in Reference [10], the trend of the flammability limit in microgravity was predicted by the trend of the blow-off limit, and the prediction was verified for PMMA and the meta-aramid fabric NOMEX. As the next step, we carried out blow-off tests and parabolic flight experiments for several flame retardant materials in opposed flow by varying the oxygen concentration and flow velocity. In the rest of this paper, we first show the results of the blow-off tests of these flame retardant materials, which were conducted on the ground, and then report their flammability limit trend in microgravity to discuss the relationship between the blow-off characteristics and the LOC in microgravity.

D.2 Analytical model for flammability limiting curve

Figure D.1 shows the typical flammability map of a thin solid material in opposed flow. The limiting curve is expressed by coupling the radiative extinction and blow-off limits. The LOC for downward spread tests in buoyant flow, LOC_{1g}, is a typical index for discussing the flammability of materials on the ground, but there can be a minimum LOC (MLOC) that is lower than the LOC_{1g}. It should be emphasized that the opposed flow velocity, V_{cr}, at which the MLOC is achieved, can be higher or lower than the buoyant flow velocity. If the MLOC is achieved in low velocity condition, the material is expected to be more flammable in microgravity, while at higher velocity conditions, the material is expected to be a flame retardant in microgravity.

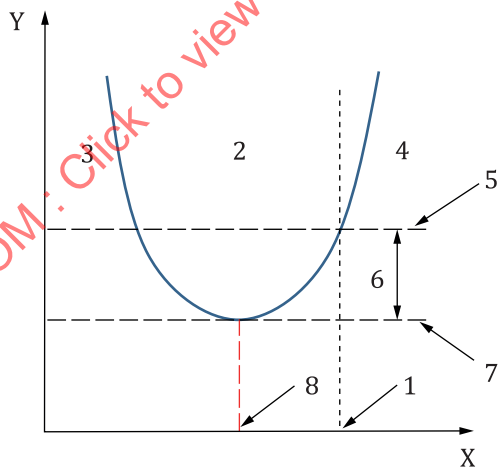
To obtain the expressions for the radiation extinction and the blow-off extinction, we built a heat balance around the two-dimensional flame spread over a thin solid material using the scale analysis technique. See Figure D.2. In the Takahashi's simplified model, the non-dimensional flame spread rate is expressed as Formula (D.1), and the limiting curve is calculated with the two non-dimensional parameters R_{rad} and Da, which represent the radiative heat loss factor and the Damkohler number, respectively.

$$\eta + R_{rad} + \frac{1}{Da} = 1 \tag{D.1}$$

$$R_{rad} = B_2 \frac{\varepsilon(1-a_{abs})\sigma(T_v^4 - T_\infty^4)}{\rho_g c_g V_g (T_f - T_v)} \tag{D.2}$$

$$Da = Pr \frac{x_d}{V_g} \rho_g Y_O A^* \exp(-E^*/RT_f) \tag{D.3}$$

The limiting curve lies on the points where the sum of R_{rad} and Da is unity. From Formula (D.2), it is found that R_{rad} is strongly affected by the pyrolysis temperature of the material, T_v. To calculate R_{rad}, we need to know the T_v of the material, which can be measured by TG/DTA. To evaluate D_a in Formula (D.3), we need to know the values of the activation energy, E, and the pre-exponential factor, A; however, these values are difficult to measure directly or to estimate. Additionally, some flame retardant materials may contain a small amount of additives to enhance their retardant property. In such a case, it becomes more difficult to take their effect into account. Hence, we introduced the blow-off tests to derive the empirical E^* and A^*, instead of actual E and A, in order to obtain the material's blow-off extinction limit accurately. The E^* and A^* obtained by the blow-off tests are empirical values and have no physical meaning, although they are helpful to understand the trend of the blow-off limit once there are specified.

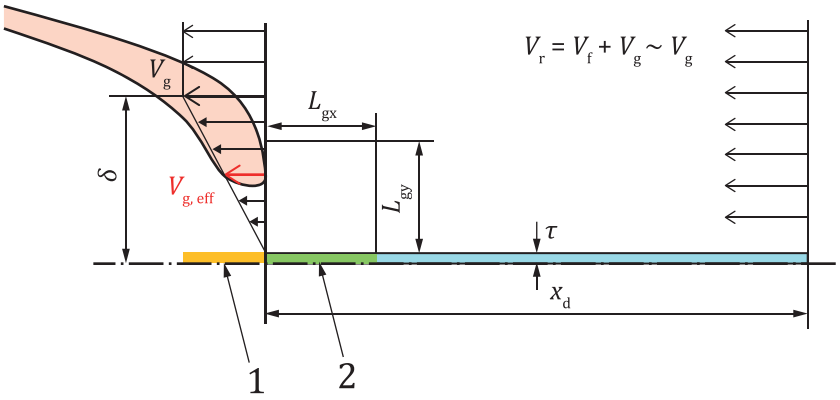


Key

X	opposed flow velocity	4	blow-off extinction
Y	oxygen concentration	5	LOC_{1g}
1	buoyant flow velocity approx. 350 mm/s	6	ΔO_2
2	flammable zone	7	MLOC
3	radiative extinction	8	critical gas velocity

NOTE The oxygen concentration and the velocity at the minimum of the U-shaped limiting curve are defined as MLOC and V_{cr}, respectively.

Figure D.1 — Typical flammability map of a solid material in opposed flow



Key

1 pyrolysis zone

2 preheat zone

Figure D.2 — Schematic of two-dimensional flame on a solid material

D.3 Relationship between the blow-off behaviour and flammability in microgravity

Blow-off behaviours of the materials listed in Table D.1 were investigated within the wind tunnel shown in Figure D.3. The forced flow velocity was varied from 60 cm/s to 130 cm/s by 10 cm/s steps; for material Z, the velocity was varied from 50 cm/s to 200 cm/s. If the flame propagated beyond the marked line, which was 4 cm away from the bottom sample edge (2nd line in Figure D.3), we considered the condition as flammable; otherwise, we considered it extinct. We repeated the blow-off test under the same conditions three times, each time decreasing the oxygen concentration by 0,5 %, until all samples were considered extinct. The minimum oxygen concentrations at which the material was flammable were defined as the LOC at the given flow velocity. The results of downward spread tests in buoyant flow were equivalent to the results where the opposed flow velocity was 35 cm/s.

Table D.1 — Properties of the materials used in this study

Type	Material	Thickness (mm)	Heat of combustion Δhc (kJ/g) [12]	Pyrolysis temperature T_v (K) [10][11]	LOC in buoyant flow LOC_{1g} (%)
Thermoplastic resin	S	0,125	25,9	670	17
	T	0,25	30,32	812	22
	U	0,2	30,16	863	28,5
	V	0,03	Unknown[a]	867	24
Thermosetting resin	W	0,125	25,45	906	45,5
Fibre and rubber	X	0,3	25,53	761	31,5
	Y	0,3	25,53	819	32,5
	Z	0,3	26,00	854	33

[a] The flame temperature was estimated assuming that the heat released per gram of oxygen consumed by the combustion was 13,10 kJ/g-O_2 [12].