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Space systems — Safety and compatibility of materials — Method to determine the flammability thresholds of materials

Systèmes spatiaux — Sécurité et compatibilité des matériaux — Méthode de détermination des seuils d'inflammabilité des matériaux

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Foreword

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

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

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An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 16697 was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

Introduction

Spacecraft fire safety emphasizes fire prevention, which is achieved primarily through the use of fire-resistant materials. Materials selection for spacecraft is based on conventional flammability acceptance tests, along with prescribed quantity limitations and configuration control for items that do not pass these acceptance tests or are questionable. ISO 14624-1 and ISO 14624-2 are the main methods used to evaluate flammability of polymeric materials intended for use in the habitable environments of spacecraft. These methods are upward flame-propagation tests initiated in static environments and using a well-defined igniter flame at the bottom of the sample.

The pass/fail test logic of ISO 14624-1 and ISO 14624-2 does not allow for a quantitative comparison with reduced-gravity or microgravity test results; therefore use of these methods is limited for in-depth theoretical analyses and realistic estimates of spacecraft fire extinguishment requirements. To better understand the applicability of laboratory test data to actual spacecraft environments, this Technical Specification has been proposed which, as an alternative to qualifying materials as pass/fail, measures the actual upward flammability limit for the material.[1] A working group established by NASA to provide recommendations for exploration spacecraft realized the importance of correlating laboratory data with real-life environments, and recommended the development of a flammability threshold test method[2]. The working group indicated that “the flammability threshold information will allow identification of materials with increased flammability risk from oxygen concentration and total pressure changes, minimize potential impacts, and allow for development of sound requirements for new spacecraft and extravehicular landers and habitats”. Furthermore, recent research using this method has shown that conventional normal-gravity materials flammability tests do not correlate with the flammability in ventilated, micro- or reduced-gravity conditions.[3][4] Currently, the materials selection for spacecraft is based on the assumed extension of ground flammability test results to spacecraft environments, an assumption which needs to be validated by further testing. In contrast to pass/fail data, materials flammability threshold data acquired in normal gravity can be compared with data obtained in microgravity or reduced-gravity experiments[5] and, consequently, a more accurate assessment of the margin of safety of the material in the real spacecraft environment can be made. In addition, this Technical Specification gives the option of selecting better or best space system materials, as opposed to what would be considered just “acceptable” from a flammability point of view, and realistic assessment of spacecraft fire extinguishment needs, which could result in significant weight savings. The knowledge afforded by this technique allows extrapolations of flammability behaviour to conditions not specifically tested and this could potentially result in significant cost and time savings.[6] This Technical Specification presents a method for evaluating oxygen-concentration flammability extinguishment limits when a material is exposed to a standard ignition source under total pressure, temperature, convective flow, and gravity-level conditions. However, the method can also be used to determine other flammability extinguishment limits, such as the total pressure[7] or forced convective velocity thresholds, while maintaining other test conditions constant.

The intent of this Technical Specification is to highlight the importance of correlating laboratory test data with real-life space system applications. The method presented is just one of the possibilities believed to lead to a better understanding of the applicability of materials flammability test data. International feedback on improving the proposed method, as well as suggestions for correlating test data with space system applications, are being sought.

Space systems — Safety and compatibility of materials — Method to determine the flammability thresholds of materials

1 Scope

This Technical Specification provides a method to determine the oxygen concentration threshold when a material exposed to a standard ignition source self-extinguishes at a given constant total pressure, temperature, convective flow, and gravity-level conditions. The method can also be used to determine other flammability limits, such as the total pressure or forced convective velocity thresholds, while maintaining other test conditions constant. The flammability thresholds can also be determined with alternate pass/fail criteria, sample configurations and/or size, ignition mode, etc. to allow correlations with test data obtained, for example, under microgravity or reduced-gravity conditions which may have limited testing time in ground experiments.

NOTE The method described provides an assessment of the flammability of aerospace materials under laboratory conditions only. Nevertheless, the results may be used as elements of a fire risk assessment which take into account all the pertinent factors for an assessment of the fire hazard in space systems. Studies on parametric effects have been initiated to better understand the applicability of the data.[8]

2 Normative references

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

ISO 14624-1, *Space systems — Safety and compatibility of materials — Part 1: Determination of upward flammability of materials*

ISO 14624-2, *Space systems — Safety and compatibility of materials — Part 2: Determination of flammability of electrical-wire insulation and accessory materials*

3 Terms and definitions

For the purposes of this Technical Specification, the terms and definitions given in ISO 14624-1 and ISO 14624-2 and the following apply.

3.1
upward limiting oxygen index
ULOI
oxygen concentration where approximately 50 % of samples fail the test criteria described in Clause 4

3.2
maximum oxygen concentration
MOC
highest oxygen concentration where all samples tested (at least five) pass the test criteria described in Clause 4

4 Test criteria

This test method attempts to determine the self-extinguishment limits of materials. Individual samples are considered to self-extinguish when the burn length is less than 15,2 cm when tested in accordance with ISO 14624-1 or ISO 14624-2. The material is considered to self extinguish when all samples tested (at least five) pass the burn length criteria for individual samples.

5 Sample preparation

A minimum of 20 samples shall be prepared. Preparation of samples for testing involves the following tasks:

a) receiving and visually inspecting the material;

b) preparing samples to the proper dimensions;

c) cleaning the samples;

d) inspecting the samples (samples should be prepared exclusively for use in this test).

The sample length should be at least 15,2 cm. A standard sample length as indicated by ISO 14624-1 and ISO 14624-2 is recommended if a direct correlation with these standards is required. Otherwise, the samples shall be prepared as required in ISO 14624-1 or ISO 14624-2.

6 Test system

The test system shall comprise the components identified in ISO 14624-1 or ISO 14624-2.

7 Pre-test procedures

Before testing, all pertinent information (including sample identification, pressure, and oxygen concentration) shall be recorded on the data sheet (see Annex B). The system shall be visibly clean, and all measuring devices shall be in current calibration. The exposed centre section of standard-sized samples shall be 5,1 cm wide. Samples shall not be overly stretched or tightened, which would cause lines of horizontal stress; thin films shall be mounted with 1,3 cm of slack in the width to allow for shrinkage.

The igniter specified in ISO 14624-1 or ISO 14624-2 shall be placed parallel to the lower edge of the sample and centred along the plane of the front surface of the sample. The igniter shall be placed (0,6 ± 0,3) cm below the lower edge of the sample. Finally, the K-10 paper shall be mounted horizontally 20,3 cm and centred directly below the sample.

8 Test procedures

8.1 Determining the upward limiting oxygen index (ULOI)

Start testing at an oxygen concentration corresponding to the ULOI for similar materials. Conduct the testing in accordance with ISO 14624-1 or ISO 14624-2; if the sample burns less than 15,2 cm, record this as an “O” response, while if the sample burns at least 15,2 cm, record it as an “X” response on the data sheet. Observe and record the material's burning behaviour such as sparks, flame jets, burning/melting/dripping of material, and if ignition of K-10 paper occurs. Video record the tests if possible.

Select the oxygen concentration to be used for the next test as follows:

a) decrease the oxygen concentration if the burning behaviour of the preceding specimen gave an “X” response;

b) increase the oxygen concentration if the burning behaviour of the preceding specimen gave an “O” response.

Repeat the test procedures using oxygen concentration changes of any convenient step size until two oxygen concentrations, in percent volume, have been found that differ by 1 % oxygen and of which one gave an “O” response and the other an “X” response. The two results at oxygen concentrations 1 % apart which gave opposite responses do not have to be from successive specimens. The ULOI is the lower oxygen concentration when the specimen fails the 15,2 cm burn length criteria (“X” response).

NOTE 1 The oxygen concentration change of 1 % has been determined to be adequate for most conditions. Using a lower oxygen concentration change may yield higher precision but could require more tests. Using a step size of 2 % will yield quicker results and could be employed when lower precision is acceptable. This step size is especially suitable when higher oxygen concentration thresholds than approximately 30 % by volume are expected.

NOTE 2 The procedure outlined in Annex A can be used to determine the ULOI when a higher precision is needed. See also ISO 4589[9] for additional details.

8.2 Determining the maximum oxygen concentration (MOC)

From the set of data obtained, determine the highest oxygen concentration where the specimen passed the burn length criteria ("O" response) and which is 1 % below the lowest oxygen concentration where the specimen failed ("X" response). Conduct additional tests at this oxygen concentration until a total of five tests have been completed without failure. If a failure is obtained, keep lowering the oxygen concentration by 1 % by volume until five samples pass.

NOTE If the step size in 8.1, Note 1, is selected, determine the highest oxygen concentration at which the material passed the burn length criteria, and which is 2 % below the lowest oxygen concentration where the material failed.

9 Recording and reporting

The following observations should be made and recorded:

a) material identification (composition, trade name, manufacturer, etc);

b) test sample description (average sample dimensions, weight, other pertinent information);

c) pertinent information relating to test sample preparation;

d) test total pressure;

e) test-chamber internal volume;

f) type of mounting fixture employed;

g) pre-test oxygen concentration, burn length, sparks, flame jets; melting material falling for each test;

h) at least for the MOC series of tests, use of K-10 paper; the use of K-10 paper can be discontinued at a given test condition, once an ignition has been observed;

i) at least in the MOC series of tests, post-test well-mixed chamber oxygen concentration.

The test report shall include sample identification, configuration, test conditions, and observations from the test. Proper reporting of the test observations, such as length of burn, ignition of the K-10 test paper, ULOI and MOC values, and other observations (especially of unusual behaviour) are critical.

When there is a deviation from standard test parameters, such as non-standard sample preparation, sample dimensions, sample orientation, or ignition source, the test shall be identified as non-standard.

10 Precision

Measurements shall be carried out to the precision specified in ISO 14624-1 or ISO 14624-2.

Annex A
(informative)

Alternate determination with increased precision of upward limiting oxygen index (ULOI)

Start testing at an oxygen concentration corresponding to the ULOI for similar materials. Conduct the test described in ISO 14624-1 or ISO 14624-2; if the sample burns less than 15,2 cm, record this as an "O" response, while if the sample burns at least 15,2 cm, record it as an "X" response on the data sheet. Observe the material's burning behaviour and record sparks, flame jets, burning/melting/dripping of material, and if ignition of K-10 paper occurs.

NOTE The ULOI is calculated following the "up-and-down method for small samples".[10]

Select the oxygen concentration to be used for the next test as follows:

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

b) increase the oxygen concentration if the burning behaviour of the preceding specimen gave an "O" response.

Repeat the test procedures using oxygen concentration changes of any convenient step size until two oxygen concentrations, in percent volume, have been found that differ by 1 % oxygen and of which one gave an "O" response and the other an "X" response. The two results at oxygen concentrations 1 % apart which gave opposite responses do not have to be from successive specimens. From this pair of oxygen concentrations, note which one gave the "O" response as the preliminary oxygen concentration.

Using the preliminary oxygen concentration, conduct the test described in ISO 14624-1 or ISO 14624-2 and record the data in the data sheet (see Table B.1 in Annex B), in the first row under the heading "Determination of N_L series". Change the oxygen concentration for the next tests by 1 % by volume and conduct testing until a difference in response is obtained. Transpose the last measurement of the N_L series to the first row under the heading "Determination of C_F" of the data sheet. Conduct four additional tests; note the final oxygen concentration obtained as C_F.

NOTE 1 N_L is a series of tests conducted until two results at oxygen concentrations 1 % apart give opposite responses from successive measurements. C_F is the oxygen concentration obtained in the final test upon conducting four additional tests following the data point obtained in the N_L series.

NOTE 2 Do not skip any 1 % increment/decrement for the N_L and C_F series of measurements.

Considering the responses of the N_L series and the last series of five measurements resulting in C_F (see Table B.1), determine the k value from Table A.1 as follows:

— Use Column 1 if the first N_L determinations are O, OO, OOO, or OOOO.

— Use Column 6 if the first N_L determinations are X, XX, XXX, or XXXX, but with the sign for k in Columns 2 to 5 reversed.

The ULOI shall be calculated as follows:

$$\text{ULOI} = C_F + k$$

Table A.1 — Determination of k for first N_L series of measurements

1	2	3	4	5	6
Responses for the last five measurements	**Values of k for which the first N_L determinations are:** [a]				**Responses for the last five measurements**
	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

[a] Determine the k value from this table as follows:

— Use Column 1 if the first N_L determinations are O, OO, OOO, or OOOO.

— Use Column 6 if the first N_L determinations are X, XX, XXX, or XXXX, but with the sign for k in Columns 2 to 5 reversed.

Annex B
(informative)

Data sheet for upward flame-propagation oxygen-concentration flammability threshold test

The data sheets below are examples of data obtained. First, determine the upward limiting oxygen index (ULOI) according to 8.1 and the example given in Table B.1. Secondly, determine the maximum oxygen concentration where all samples tested (at least five) pass the test criteria described in Clause 4 (determine the MOC according to 8.2 and the example given in Table B.2).

Material: epoxy coating **Date:**

Test pressure: 70 kPa

Table B.1 — Upward limiting oxygen index (ULOI)

Test No.	Pre-test O_2 concentration vol. %	Pre-test sample length cm	Burn length cm	Pass/ fail burn length criteria O/X	Sparks[a]	Flame jets[a]	Burning/ melting/ dripping material[a]	K-10 paper ignition Y/N	Post-test O_2 concentration[b] vol. %
Determination of preliminary oxygen concentration									
1	22,2	15,2	0,3	O	N	N	N	N	ND
2	28,1	15,2	15,2	X	N	N	N	N	ND
3	27,0	15,2	0,8	O	N	N	N	N	ND
Determination of N_L series									
4	27,2	15,2	0,5	O	N	N	N	N	ND
5	28,2	15,2	15,2	X	N	N	N	N	ND
Determination of C_F[c]									
5	28,2	15,2	15,2	X	N	N	N	N	ND
6	27,3	15,2	15,2	X	N	N	N	N	ND
7	26,1	15,2	15,2	X	N	N	N	N	ND
8	25,1	15,2	0,5	O	N	N	N	N	ND
9	26,0	15,2	1,0	O	N	N	N	N	ND

[a] S = small; M = medium; L = large; N = none.

[b] ND = not determined.

[c] $C_F = 26,0$.

The N_L series and the last five tests for the determination of C_F (tests 5 to 9) are used to determine k in Table A.1. In Table B.1, for the O/X obtained for the N_L series, transpose the last set of measurements to the first row under “Determination of C_F”. In Table A.1, for the remaining O column in the N_L series (see Column 2 of Table A.1), choose row XXXOO in Column 1, obtained in the last five measurements. This gives:

$k = 0,50$

$\text{ULOI} = C_F + k = 26,5$

