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Space systems — Safety and compatibility of materials —

Part 3: Determination of offgassed products from materials and assembled articles

Systèmes spatiaux — Sécurité et compatibilité des matériaux —

Partie 3: Détermination des produits issus du dégazage sous atmosphère des matériaux et des articles assemblés

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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

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

ISO 14624-3 was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

ISO 14624 consists of the following parts, under the general title *Space systems — Safety and compatibility of materials*:

— *Part 1: Determination of upward flammability of materials*

— *Part 2: Determination of flammability of electrical-wire insulation and accessory materials*

— *Part 3: Determination of offgassed products from materials and assembled articles*

— *Part 4: Determination of upward flammability of materials in pressurized gaseous oxygen or oxygen-enriched environments*

— *Part 5: Determination of reactivity of system/component materials with aerospace propellants*

— *Part 6: Determination of reactivity of processing materials with aerospace fluids*

— *Part 7: Determination of permeability rate and penetration resistance of materials to aerospace fluids*

Introduction

Throughout this part of ISO 14624, the minimum essential criteria are identified by the use of the imperative or the key word “shall”. Recommended criteria are identified by the use of the key word “should” and, while not mandatory, are considered to be of primary importance in providing serviceable, economical and practical designs. Deviations from the recommended criteria may be made only after careful consideration, extensive testing and thorough service evaluation have shown an alternative method to be satisfactory.

The data obtained from this test are used for a toxicological assessment of the risks to personnel. Additional information can be gathered utilizing this test method, for example, taking samples at equal intervals can provide information on offgassing rates.

Space systems — Safety and compatibility of materials —

Part 3: Determination of offgassed products from materials and assembled articles

1 Scope

This part of ISO 14624 specifies a method for determining the identity and quantity of volatile offgassed products from materials and assembled articles utilized in manned, pressurized spacecraft. This test method is not intended to model or simulate spacecraft atmospheres.

2 Conformance

The test shall be performed in an accredited test facility (see Annex A for guidelines).

The authority having jurisdiction, or test requester, shall provide properly identified material(s) for testing. Alternatively, accredited test facilities may be authorized by the test requester to procure the appropriate material(s). Materials also shall be accompanied by the appropriate vendor-supplied Material Safety Data Sheets to comply with materials-handling requirements defined by the appropriate country's Occupational Safety and/or Health Administration. Materials and configured system characteristics can be significantly compromised by sources of contamination, such as exposure to solvents, cleaning agents, abnormal temperatures, variations in humidity, environmental pollutants, particulate, and handling. It is important that exposure of the material to these and other contamination sources be sufficiently controlled to minimize variation in test results.

As a minimum, all fluids used for testing shall meet or exceed user specifications.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
assembled article
any component or assembly of components that is not a single material

3.2
offgassed product
organic or inorganic compound evolved as a gas from a material or assembled article

3.3
offgassing
evolution of gaseous products from a liquid or solid material into an atmosphere

3.4
spacecraft maximum allowable concentration
SMAC
maximum concentration of an offgassed product that is allowed in the habitable area of the spacecraft for a specified flight duration

NOTE SMAC values for manned spacecraft are determined by the cognizant procuring authority/user toxicologist. A current listing of SMAC values is maintained on the Internet at http://www.jsc.nasa.gov/toxicology/Guidelines.

3.5
toxic hazard index
T
dimensionless ratio of the projected concentration of each offgassed product to its SMAC value and summing the ratios for all offgassed products without separation into toxicological categories, and the calculation of the T value is as follows:

$$T_{\text{total}} = C_1/I_{\text{SMAC1}} + C_2/I_{\text{SMAC2}} + \ldots + C_n/I_{\text{SMAC}n} \quad (1)$$

where

$C_1, C_2, \ldots, C_n$ are the concentrations of contaminants 1, 2 and n, respectively;

$I_{\text{SMAC1}}, I_{\text{SMAC2}}, \ldots, I_{\text{SMAC}n}$ are the SMAC values for contaminants 1, 2 and n, respectively

NOTE For assembled articles, concentration is calculated by dividing the total quantity of each contaminant offgassed during a test by the habitable volume of the spacecraft. For materials, the concentration is calculated by multiplying the total quantity of each contaminant offgassed per gram of material by the total mass of the material to be used in the spacecraft.

EXAMPLE Evaluating the maximum limit mass for a standard shuttle test, the total mass of material to be used is assumed to be 45 kg and the habitable volume of the spacecraft is 65 m^3.

3.6
good laboratory practice
GLP
practice that involves the testing of standard materials to verify data accuracy and repeatability

3.7
round robin testing
testing of identical materials at different test facilities for the comparison of results

3.8
average percent relative standard deviation
quotient of the standard deviations for each offgassed constituent of y replicate samples of a standard material and the total number of offgassed constituents

NOTE For actual samples, the expected test results and average relative standard deviations for the quantities of offgassed products are near 50 %. The calculations for standard deviation and average percent relative standard deviation are as follows:

The standard deviation, s, is given by:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

where $\bar{x}$ is the mean for an individual offgassed constituent.

Therefore, the calculation for the average percent relative standard deviation A_s, is given by:

$$A_s = \frac{\sum s}{y} \times 100\ \%$$

where

$\sum s$ is the summation of the standard deviations for each offgassed constituent;

y is the total number of offgassed constituents, for a standard material.

3.9
test chamber
apparatus into which the sample container is placed during thermal conditioning

3.10
sample container
vessel which contains the test sample

3.11
room temperature
room temperature is equal to (23 ± 3) °C

4 Principle

When this method is utilized for a toxicological assessment for a component or a material, the total toxic hazard index (T) values for all volatile offgassed products shall be less than 0,5.

5 Health and safety of test operators

Testing outlined in this part of ISO 14624 may generate toxic substances in either the gas or condensed phase. Care shall be taken to protect test operators from such substances.

6 Test conditions

6.1 The test atmosphere should be at least a volume fraction of (20,9 ± 2) % for oxygen with the balance nitrogen or argon, and the test pressure should be < 15 kPa of the ambient pressure of the test facility. The maximum volume fraction limits [expressed as a volume fraction in µl/l[1)]] for impurities in the compressed gases are:

— carbon monoxide 1;
— carbon dioxide 3,0;
— total hydrocarbons, as methane 0,1;
— halogenated compounds 0,5;
— water 7,0.

6.2 The sample shall be subject to a thermal exposure for (72 ± 1) h at (50 ± 3) °C. Samples tested at one oxygen concentration do not have to be retested at a different oxygen concentration.

1) 1 µl/l = 1 ppm. The use of “ppm” is deprecated.

7 Apparatus and materials

The test system shall comprise the following major components: sample container, test chamber with controlled temperature, and analytical instrumentation.

7.1 Sample container, being easy to clean and constructed so that gas samples can be collected easily.

The sample container, including any soft goods, shall not significantly affect the concentration of products offgassed from the samples.

7.2 Test chamber, having the capability to maintain the test temperature to within ± 3 °C for the duration of the test.

The test chamber instrumentation shall have the capability to continuously record the temperature.

7.3 Analytical instrumentation, not specified; however, capable of the separation, identification, and quantification of all offgassed products indicated in Annex B at, or below, their SMAC concentrations when tested at a sample-mass-to-container-volume ratio of (5,0 ± 0,25) g/l.

If the instrumentation cannot achieve this sensitivity, the minimum reportable concentration for those offgassed products shall be reported. The recommended analytical instruments include a gas chromatograph using primarily a flame ionization detector, gas chromatograph/mass spectrometer, and infrared spectrophotometer. Some analytical compounds may be more difficult to determine, therefore, special methods may be required to identify and quantify these compounds. For example, the determination of formaldehyde may be performed using the proposed method of trapping on 2,4-dinitrophenylhydrazine (DNPH) cartridges for derivation and subsequent analysis by HPLC.

8 Test samples

8.1 Handling/receipt

Handling of test articles shall be in a manner that preserves the integrity of the sample surface without adding contaminants. Test samples shall be prepared from either materials or assembled articles. Preparation of samples for testing involves the following tasks:

— receiving and inspecting the material;

— preparing samples to the proper dimensions, if required;

— cleaning the samples, if specified by the requester;

— inspecting the samples.

8.2 Preparation

8.2.1 When received, the test material shall be accompanied by proper identification, including appropriate Material Safety Data Sheets. Note flaws and any residual contamination. All materials shall meet the requirement of sample-mass-to-container-volume ratio of (5,0 ± 0,25) g/l, and record the approximate total sample surface area. Sample preparation for test materials based on mass shall be as specified in 8.2.2 to 8.2.4.

8.2.2 Materials that are essentially two-dimensional and require application to a substrate (e.g. coatings, primers, inks, paints, adhesives, tapes, thin film lubricants) shall be applied at their thickness of use to clean aluminium substrates. Samples may be applied to both sides of the substrate. Prepare a sufficient number of substrates with applied sample material so as to provide a net sample-mass-to-test-chamber-volume ratio of (5,0 ± 0,25) g/l. Report the approximate total sample surface area.

8.2.3 Materials that are essentially two-dimensional and are not applied to a substrate (e.g. fabrics, photographic film plastic, plastic film, elastometrics, non-adhesive tape) shall be cut to convenient test dimensions. Shrink heat-shrinkable tubing so as to simulate actual use configuration. Prepare a sufficient quantity of samples so as to provide a sample-mass-to-test-chamber-volume ratio of (5,0 ± 0,25) g/l. Place liquids in petri dishes of (5,1 ± 0,5) cm diameter.

8.2.4 It shall be recognized that some specialized items and materials may not meet the above requirements and shall require special handling, most often with non-homogeneous materials. Test these materials in the manner designated by the responsible procuring activity/user materials engineer. Report the manner of testing and sample preparation. The desired test-material-to-chamber-volume ratio is (5,0 ± 0,25) g/l.

8.3 Flight articles

If a sample is an assembled article, inspect it for parts that are not designated for flight, such as dust covers, tape, or test leads. Remove these items before testing. Record the absence of such items as batteries or photographic film included during flight but not included with the sample. The ratio of sample volume to sample container volume should be approximately 1:3.

8.4 Cleaning

Samples should be cleaned and dried to the end-use specifications by the requester prior to receipt at the test facility. The cleaning of assembled articles shall be the responsibility of the requester. If a sample received by the test facility is visibly contaminated, clear instructions shall be received from the requester as to proper procedures for continuing testing. For samples prepared by the test facility, all preparation and cleaning shall be in accordance with user/requester specifications. All cleaning procedures shall be first approved by the requester, and verified to have no influence on analytical results. As a minimum, particulate on sample surfaces should be removed with filtered, gaseous nitrogen.

8.5 Inspection

Inspect the sample and note any flaws. (If the flaws result from sample preparation at the test facility, new samples should be prepared.) Weigh the samples and individually identify them.

9 Pretest procedure

9.1 The pretest procedure includes cleaning of sample containers, certification of container cleanliness, and calibration of the quantitative analytical instruments.

9.2 Clean the sample containers by heating to drive off residual container contamination then purge them with clean air or nitrogen before each use. Solvent cleaning should be avoided.

9.3 Before loading the sample into the container, fill the container with the test atmosphere or nitrogen then condition it for at least (72 ± 1) h at (50 ± 3) °C. Alternatively, the sample container can be conditioned for at least 24 h at (70 ± 3) °C. Analyse the sample container atmosphere for residual contamination. Certify the sample container as clean for use if the concentrations of residual gases are sufficiently low that they will not interfere with interpretation of results of the offgas analysis.

9.4 The methods of quantitative analysis shall be traceable to primary gas standards. Any standards used to quantify specific compounds shall be traceable to the national or international authority having jurisdiction.

10 Procedure

10.1 Weigh the sample and place it in the sample container. Replace the room atmosphere in the sample container with the test atmosphere, either by purging or by evacuation. The requesting organization shall indicate if the sample can or cannot withstand a vacuum and exposure of any sample to vacuum shall be less than 3 min. The sample container, with the test atmosphere, shall be at the requested test pressure when the test temperature is achieved.

10.2 Place the sample container in the test chamber and heated to the test temperature of (50 ± 3) °C, unless otherwise specified and maintain this temperature for (72 ± 1) h. Then cool the sample container to (23 ± 3) °C, record the pressure, and sample and analyse the offgassed products. Perform the sampling and analysis of offgassed products as soon as ambient (room) temperature is achieved and no later than 24 h subsequent to reaching ambient (room) temperature. In cases where ambient (room) temperature is not within the specified range, perform the sampling and analysis within 24 h of the test facility's laboratory reaching its own ambient (room) temperature. Determine and record the identity and quantity of each analysable offgassed product with emphasis given to those items shown in Annex B.

10.3 A screening test for total organics may be used to determine if a full analysis is to be performed. However, it is necessary to evaluate all organics quantitated but not identified using a worst case scenario of SMAC value equal to 0,1. The screening test procedure may include specific methods to detect critical components of the target list, i.e. formaldehyde, carbon monoxide and ammonia.

11 Precision

Measurements shall be made to the following precision:

— absolute pressure ± 1 % of reading;

— temperature ± 3 °C;

— oxygen concentration ± 0,5 % of reading;

— mass ± 0,01 g.

12 Test report

The test report shall include the following:

a) reference to this part of ISO 14624 (ISO 14624-3:2004);

b) sample identification, test chamber free volume (l), sample mass (g) and/or apparent surface area (cm^2), test conditions and observations from the test;

c) for each offgassed product, the quantity as µg/g of material of material or µg/assembled article;

d) trace constituents [although not used in the calculation of the toxic hazard index (T)];

e) toxic hazard index (T).

The test report shall be submitted to the authority having jurisdiction and/or test requester. See Annex C for rating system.

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

13 Good laboratory practices (GLP)

13.1 Calibrate the quantitative analytical instrumentation before use. Analyse replicate samples periodically to insure the quality of test results. Determine precision using average percent relative standard deviation. Analyse the standard gas mixtures given in Table 1 at least every three months, and measure the concentrations to within 25 % of the specified concentrations.

13.2 In addition, the test facility shall successfully demonstrate the ability to obtain repeatable data when testing a selected material. The authority having jurisdiction shall choose appropriate GLP materials and shall determine the frequency of testing these materials for its test facilities. These materials shall include toxic materials as well as materials of low toxicity. Handle GLP materials in a manner that preserves the integrity of the sample surface without adding contaminants. Inspect all GLP materials prior to preparation. Prepare all GLP material to the proper dimensions and mass as determined by the authority having jurisdiction. Inspect all GLP material prior to loading into the sample container. Sample, store and transport all GLP materials in a manner that preserves the integrity of the material.

Table 1 — Standard gas mixtures and recommended concentrations (as gravimetric standards)

Mixture	Components	Volume fraction [a] µl/l
A	Acetonitrile	5,0
	Benzene	1,0
	1-Butene	10,0
	Dichloroethylene	1,0
	Ethyl alcohol	10,0
	Isopropyl alcohol	10,0
	Methyl alcohol	10,0
	Tetrachloroethylene	10,0
	Tetrachloromethane	5,0
	Toluene	10,0
	Trichloroethylene	1,0
	Vinyl chloride	1,0
B	Acetaldehyde	5,0
	Acetone	5,0
	Acrolein	1,0
	Acrylonitrile	5,0
	1,4-Dioxane	5,0
	Furan	1,0
	Furfural	5,0
	Methyl ethyl ketone	10,0
	Methyl isobutyl ketone	10,0
	Propionaldehyde	5,0

[a] 1 µl/l = 1 ppm. The use of "ppm" is deprecated.

Annex A
(informative)

Competency and accreditation of test facilities

A.1 Competency

Laboratories should be accredited to perform the flammability and/or combustion test methods contained within this part of ISO 14624. Accreditation is necessary because data from this testing is to be presented for aerospace flight materials selection approval. Accreditation should be based on ISO/IEC 17025 and the specific requirements described in this part of ISO 14624.

The accreditation program should include proficiency testing. Such a program should be consistent with ISO/IEC Guide 43-1.

A.2 Accreditation

Accreditation is the responsibility of the accreditation body recognized within its jurisdiction to administer laboratory accreditation. An acceptable laboratory accreditation body would be a signatory to the multi-lateral mutual recognition arrangement (MRA) of the International Laboratory Accreditation Cooperation (ILAC)[2)] or signatory to an ILAC equivalent regional/national MRA that requires accreditation bodies to conform to ISO/IEC 17011.

A.3 Guidelines

An accredited laboratory should meet the following guidelines.

a) For required tests, the test facility should have performed the test method at least once during the last eighteen months and participated in comparison of results with other accredited test facilities (interlaboratory testing).

b) All instrumentation used in the test should be in proper calibration and bear the appropriate documentation to validate traceability to appropriate national or international measurement standards.

c) The test facility should ensure that all testing is accomplished in accordance with approved test plans and procedures, and that the data records and test results be complete and accurate.

d) Complete test records should be prepared by the test facility for each material tested. The test facility should maintain a permanent record of test data for a minimum of fifteen years for historical purposes.

2) Full information is available at the web site http://www.ilac.org of ILAC or through the ILAC Secretariat, c/o NATA, 7 Leeds Street, Rhodes NSW 2138, Australia, tel. +61 2 9736 8374, fax +61 9736 8373, e-mail: ilac@nata.asn.au.