

ISO/TC 20/SC 14

Date: 2000-11-14

ISO/CD 14624-3

ISO/TC 20/SC 14/WG 1

Secretariat: ANSI (AIAA)

Space Systems- Safety and compatibility of materials- Part 3 : Test method for determination of offgassed products from materials and assembled articles

—

Document type: International Standard
Document subtype: Not applicable
Document stage: (30) Committee
Document language: E

Contents

1 Scope.....1

2 Conformance.....1

3 Normative references.....1

4 Terms and definitions2

5 Principle.....3

6 Health and safety of test operators.....3

7 Test conditions.....3

8 Apparatus and materials4

9 Test samples.....4

10 Pretest procedure.....6

11 Test procedure.....6

12 Precision.....6

13 Reporting.....6

14 Good laboratory practices (GLP).....7

Table 1 Standard gas mixtures and recommended concentrations..... 7

Foreword

ISO (the International Organization for Standardization) is a world-wide 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 3.

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.

International Standard ISO 14624-3 was developed by Technical Committee ISO/TC 20.

Copyright notice

This ISO Document is a working draft or committee draft and is copyright protected by ISO. While the reproduction of working drafts or committee drafts in any form for use by participants in the ISO standards development process is permitted without prior permission from ISO, neither this document nor any extract from it may be reproduced, stored or transmitted in any form for any other purpose without prior written permission from ISO.

Requests for permission to reproduce this document for the purpose of selling it should be addressed as shown below or to ISO's member body in the country of requester:

Secretariat: AIAA, Mr. James E French, AIAA, 1801 Alexander Bell Drive, Reston, VA, TP: 1 703 264 7570, TF: 1 703 264 7551, e-mail: jimf@aiaa.org

Reproduction for sales purposes may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.

Introduction

Throughout this International Standard, the minimum essential criteria are identified by the use of 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 should occur only after careful consideration, extensive testing and thorough service evaluation have shown alternative methods to be satisfactory.

Space systems- Safety and compatibility of materials- Part 3 : Test method for the determination of offgassed products from materials and assembled articles

1 Scope

A test method for determining the identity and quantity of volatile offgassed products from materials and assembled articles utilized in manned, pressurized spacecraft environments is presented herein. These data are used for a toxicological assessment of the risks to personnel. Additional information may be gathered utilizing this test method, for example, taking samples at equal intervals may provide information on offgassing rates. These tests shall be performed by accredited test facilities (Annex A).

2 Conformance

The responsible procuring authority/user organization or hardware supplier should provide properly identified material for testing. Alternatively, accredited test facilities may be authorized by the test requester to procure the appropriate materials. Materials also shall be accompanied by the appropriate vendor-supplied Material Safety Data Sheet to comply with materials-handling requirements defined by the appropriate national occupational safety and/or health authority. 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 Normative references

The following normative documents contain provisions, which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, the publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO/IEC 17025:2000, General requirements for the competence of testing and calibration laboratories

4 Terms and definitions

For the purposes of this International Standard, the following definitions apply:

4.1

assembled article

an assembled article could be any component or assembly of components that is not a single material.

4.2

offgassed product

an organic or inorganic compound evolved as a gas from a material or assembled article.

4.3

offgassing

the evolution of gaseous products from a liquid or solid material into an atmosphere.

4.4

spacecraft maximum allowable concentration (SMAC)

the maximum concentration of an offgassed product that is allowed in the habitable area of the spacecraft for a specified flight duration. SMAC values for manned spacecraft are determined by the cognizant procuring authority/user toxicologist. NOTE: A current listing of SMAC values is maintained on the Internet at "map1.msfc.nasa.gov."

4.5

toxic hazard index (T)

the T value is the 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. The calculation of the T value is as follows:

$$T_{total} = C_1 / SMAC_1 + C_2 / SMAC_2 + \dots + C_n / SMAC_n$$

where C_1 = concentration of contaminant 1, and $SMAC_1$ = SMAC of contaminant 1. 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. For example, evaluating the maximum limit weight for a standard shuttle test, the total mass of material to be used is assumed to be 45 Kilograms and the habitable volume of the spacecraft is 65 cubic meters.

4.6

good laboratory practices (GLP)

practices which involve the testing of standard materials to verify data accuracy and repeatability.

4.7**round robin testing**

testing of identical materials at different test facilities for the comparison of results.

4.8**average percent relative standard deviation.**

the average percent relative standard deviation is determined by dividing the standard deviations for each offgassed constituent of y replicate samples of a standard material by the total number of offgassed constituents. 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:

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

where \bar{x} is the mean for an individual offgassed constituent. Therefore, the calculation for the average percent relative standard deviation is:

$$A_s = \frac{s}{y}$$

where s is the summation of the standard deviations for each offgassed constituent, and y is the total number of offgassed constituents, for a standard material.

4.9**test chamber**

the apparatus into which the sample container is placed during thermal conditioning.

4.10**sample container**

the vessel which contains the test sample.

5 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.

6 Health and safety of test operators

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

7 Test conditions

7.1 The test atmosphere should be at least $20,9 \pm 2$ percent 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 concentration limits (in parts-per-million by volume) for impurities in the compressed gases are carbon monoxide (1), carbon dioxide (3,0), total hydrocarbons, as methane (0,1), halogenated compounds (0,5), and water (7,0).

7.2 The sample shall be subject to a thermal exposure for 72 ± 1 hours at $50 \pm 3^\circ\text{C}$. Samples tested at one oxygen concentration do not have to be retested at a different oxygen concentration.

8 Apparatus and materials

8.1 System

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

8.2 Sample container

The sample container should be 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.

8.3 Test chamber

The test chamber shall have the capability to maintain the test temperature to $\pm 3^\circ\text{C}$ for the duration of the test. The test chamber instrumentation shall have the capability to continuously record the temperature.

8.4 Analytical requirements

The analytical instrumentation is not specified; however, the instrumentation should allow separation, identification, and quantification of all offgassed products indicated in Annex C at or below their SMAC concentrations when tested at 5 g of sample per litre of container volume. 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, and special methods may be required to identify and quantify these compounds.

9 Test samples

9.1 Sample 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.

9.2 Sample preparation

9.2.1 When received, the test material shall be accompanied by proper identification, including appropriate Material Safety Data Sheets. Flaws and any residual contamination shall be noted. All materials shall meet the

requirement of $5,0 \pm 0,25$ grams sample weight per litre of sample container volume, and the approximate total sample surface area shall be recorded. Sample preparation for test materials based on weight shall be as follows:

9.2.1.1 Materials which are essentially two dimensional and require application to a substrate (e.g., coatings, primers, inks, paints, adhesives, tapes, thin film lubricants, etc.) shall be applied in their use thickness to clean aluminum substrates. Samples may be applied to both sides of the substrate. A sufficient number of substrates with sample material applied shall be prepared so as to provide a net sample weight of $5,0 \pm 0,25$ grams per litre of test chamber volume. The approximate total sample surface area shall be reported.

9.2.1.2 Materials which are essentially two dimensional and are not applied to a substrate (e.g., fabrics, photographic film plastic, plastic film, elastometrics, non-adhesive tape, etc.) shall be cut to convenient test dimensions. Heat shrinkable tubing shall be shrunk to simulate actual use configuration. A sufficient quantity of samples shall be prepared so as to provide a sample weight of $5,0 \pm 0,25$ grams per litre of test chamber volume. Liquids shall be placed in petri dishes with a diameter of $5,1 \pm 0,5$ cm.

9.2.1.3 It shall be recognized that some specialized items and materials may not meet the above requirements and shall require special handling. This most often will occur with non-homogeneous materials. These materials will be tested in the manner designated by the responsible procuring activity/user materials engineer. The manner of testing and sample preparation shall be reported. The desired ratio of test material to chamber volume is $5 \pm 0,25$ grams per litre.

9.3 Flight articles

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

9.4 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.

9.5 Inspection

The sample shall be inspected and any flaws shall be noted. (If the flaws result from sample preparation at the test facility, new samples should be prepared.) Samples shall be weighed and individually identified.

10 Pretest procedure

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

10.1.1 The sample containers shall be cleaned by heating to drive off residual container contamination and then purged with clean air or nitrogen before each use. Solvent cleaning should be avoided.

10.1.2 Before loading the sample into the container, the container shall be filled with the test atmosphere or nitrogen and then conditioned for at least 72 ± 1 hours at 50 ± 3 °C. Alternatively, the sample container can be conditioned for at least 24 hours at 70 ± 3 °C. The sample container atmosphere shall be analyzed for residual contamination. The sample container can be certified 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.

10.1.3 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.

11 Test procedure

11.1 The sample shall be weighed and placed in the sample container. The room atmosphere in the sample container shall be replaced 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 minutes. The sample container, with the test atmosphere, shall be at the requested test pressure when the test temperature is achieved.

11.2 The sample container shall be placed in the test chamber and heated to the test temperature of 50 ± 3 °C, unless otherwise specified, and held for 72 ± 1 hours. The sample container shall then be cooled to 23 ± 3 °C, the pressure recorded, and the offgassed products sampled and analyzed. The sampling and analysis of the offgassed products shall be performed while the container temperature is in the range specified, or in cases where this temperature cannot be achieved, within 24 hours, of the time the sample container cools to room temperature. The identity and quantity of each analyzable offgassed product shall be determined and recorded, with emphasis given to those items shown in Annex C.

12 Precision

Measurements shall be made to the following precision:

- Absolute pressure, ± 1 percent of reading;
- Temperature, ± 3 °C;
- Oxygen concentration, $\pm 0,5$ % of reading;
- Mass, $\pm 0,01$ grams.

13 Reporting

13.1 The test report shall include sample identification, test chamber free volume (L), sample weight (g) and/or apparent surface area (cm²), test conditions, and observations from the test. For each offgassed product, the quantity shall be reported as $\mu\text{g/g}$ of material of material or $\mu\text{g/}$ assembled article. Trace constituents shall be reported but not used in calculation of the Toxic Hazard Index (T), which is indicated in the test report. The test report shall be submitted to the authority having jurisdiction and/or test requester. See Annex B for rating system.

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

14 Good laboratory practices (GLP)

14.1 The quantitative analytical instrumentation shall be calibrated before use. Replicate samples should be analyzed periodically to insure quality of test results. Precision is determined using average percent relative

standard deviation. The following standard gas mixtures (Table 2) shall be analyzed at least every three months, and the measured concentrations shall be within 25 % of the specified concentrations.

14.2 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 will determine the frequency of testing these materials for its test facilities. These materials shall include highly toxic materials as well as materials of low toxicity.

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

Components	Concentration (ppm by volume)
Mixture 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
Mixture 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

Annex A (normative)

Competency/accreditation of test facilities

A.1 Laboratories shall be qualified in the general fields of gas detection and identification and shall be accredited in the performance of test methods contained within this document. Accreditation is necessary because data from this testing will be presented for aerospace flight materials selection approval. As an alternative to accreditation, the test facility shall meet the requirements of ISO/IEC 17025:2000.

A.2 Test facility accreditation may be based on the following guidelines, as approved by the authority having jurisdiction. Accreditation is the responsibility of the authority having jurisdiction. This authority may appoint a panel, or perform the accreditation itself.

A.3 The test facility applying for accreditation shall prepare and submit detailed documentation of procedures utilized in the performance of all activities outlined in this document to the authority having jurisdiction or its representative. This documentation shall include provisions for instrument calibration, sample preparation, test chamber certification, test atmosphere analysis or verification, data analysis, recording and archiving of test data, materials control, and control of flight articles (if applicable). The test facility also shall include in its procedures the specification of the minimum acceptable personnel qualifications, training requirements, and personnel certification processes. The test facility also shall supply documentation demonstrating that operations performed utilizing the subject test methods present no hazards to personnel or flight hardware.

A.4 The authority having jurisdiction, or its representative, shall perform an on-site inspection of the test facility. The authority having jurisdiction, or its representative, will insure that the test facility is capable of performing all required activities specified in this document. The authority having jurisdiction, or its representative, shall recommend accreditation of the test facility on the basis of compliance with this annex. Accreditation may be maintained by meeting the following requirements:

- For required tests, the test facility shall have performed the test method at least once during the last eighteen months and participated in comparison of results with other accredited test facilities (round robin).
- All instrumentation used in the test shall be in current calibration and bear the appropriate documentation to validate traceability to appropriate national or international measurement standards.
- The test facility shall insure that all testing is accomplished in accordance with approved test plans and procedures, and that the data records and test results are complete and accurate.
- Complete test records shall be prepared by the test facility for each material or flight article tested. The test facility shall maintain a permanent record of test data for a minimum of ten years for historical purposes.
- The authority having jurisdiction, customer and/or user has the right to conduct at any time an on-site audit of any accredited test facility.

Annex B (normative)

Guidelines for evaluation

B.1 General

The total or summation Toxic Hazard Index (T) is determined by using the free volume of the spacecraft and the quantities of all volatile offgassed products that result from the 72 ± 1 hour test.

$$T_{total} = C_1 / SMAC_1 + C_2 / SMAC_2 + \dots + C_n / SMAC_n$$

B.2 Maximum limit weight (MLW) concept or maximum number of assemblies

The maximum limit weight for materials, as well as, the maximum number of units for assembled articles can be determined and is indicative of the quantity of material or the number of assembled articles that can be safely utilized inside the spacecraft without exceeding a total T of 0,5.

B.3 Suggested Ratings

Ratings may be assigned to materials based on the maximum limit weight as follows:

K = MLW of 45,36 kg or greater;

H = MLW of 22,68 kg to 45,359 kg

A = MLW of 4,536 kg to 22,679 kg

V = MLW of 2,268 kg to 4,5359 kg

X = MLW of less than 2,268 kg.

Ratings may be assigned to assemble articles based on the total T value as follows:

A = assembled articles with a T value ≤ 0.5

X = assembled articles with a T value > 0.5.

The maximum weight limit can be determined by calculating the total T value.

Annex C (normative)

Target compound list

Compound	CAS No.	Formula	Molecular Weight	7-day SMAC (ppm)
Formaldehyde	50-00-0	CH ₂ O	30,03	0,04
Methanol	67-56-1	CH ₄ O	32,04	7
Benzene	71-43-2	C ₆ H ₆	78,11	0,5
Vinyl chloride	75-01-4	C ₂ H ₃ Cl	62,50	1,0
Acetonitrile	75-05-8	C ₂ H ₃ N	41,05	4,0
Acetaldehyde	75-07-0	C ₂ H ₄ O	44,05	2,0
Dichloromethane	75-09-2	CH ₂ Cl ₂	84,93	15
Ethylene oxide	75-21-8	C ₂ H ₄ O	44,05	10
3-Buten-2-one	78-94-4	C ₄ H ₆ O	70,09	2,0
2-Methyl-2-propenal	78-85-3	C ₄ H ₆ O	70,09	0,6
Trichloroethene	79-01-6	C ₂ HCl ₃	131,39	9
1,3-Butadiene	106-99-0	C ₄ H ₆	54,09	0,3
Propenal	107-02-8	C ₃ H ₄ O	56,06	0,015
Chloropropene (as 3-Chloropropene)	107-05-1	C ₃ H ₅ Cl	76,53	0,2
1,2-Dichloroethane	107-06-2	C ₂ H ₄ Cl ₂	98,96	0,5
Acrylonitrile	107-13-1	C ₃ H ₃ N	53,06	2,9
Furan	110-00-9	C ₄ H ₄ O	68,08	0,04
Heptanal	111-71-7	C ₇ H ₁₄ O	114,19	1,2
2-Butenal	123-73-9	C ₄ H ₆ O	70,09	0,6
Methylfuran (as 2-Methylfuran)	534-22-5	C ₅ H ₆ O	82,10	0,04
2,5-Dimethylfuran	625-86-5	C ₆ H ₈ O	96,13	0,04
Carbon monoxide	630-08-0	CO	28,01	10
Ammonia	7664-41-7	NH ₃	17,03	7