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EUROPEAN COOPERATION

ECSS

FOR SPACE STANDARDIZATION

Space product assurance

The determination of offgassing products from materials and assembled articles to be used in a manned space vehicle crew compartment

ECSS Secretariat
ESA-ESTEC
Requirements & Standards Division
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Foreword

This Standard is one of the series of ECSS Standards intended to be applied together for the management, engineering and product assurance in space projects and applications. ECSS is a cooperative effort of the European Space Agency, National Space Agencies and European industry associations for the purpose of developing and maintaining common standards.

Requirements in this Standard are defined in terms of what shall be accomplished, rather than in terms of how to organize and perform the necessary work. This allows existing organizational structures and methods to be applied where they are effective, and for the structures and methods to evolve as necessary without rewriting the standards.

The formulation of this Standard takes into account the existing ISO 9000 family of documents.

This Standard has been prepared by editing the ESA PSS-01-729, reviewed by the ECSS Technical Panel and approved by the ECSS Steering Board.

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Contents list

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Introduction

All non-metallic materials release trace contaminants into the surrounding environment; the extent to which this occurs is dependent on the nature of the material concerned. In the closed environment of a manned spacecraft contaminants within the atmosphere are potentially dangerous with respect to toxicity and its consequences for the safety of the crew.

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Scope

This Standard defines a test procedure for the determination of the trace contaminants release by non-metallic materials under a set of closely controlled conditions. The test procedure covers both individual materials and assembled articles.

This Standard describes a test to provide data for aid in the evaluation of the suitability of assembled articles and materials for use in a space vehicle crew compartment. The data obtained are in respect of the nature and quantity of organic and inorganic volatile contaminants evolved when subjected to the crew compartment environment.

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Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this ECSS Standard. For dated references, subsequent amendments to, or revisions of any of these publications do not apply. However, parties to agreements based on this ECSS Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

ECSS-P-001	Glossary of terms
ECSS-Q-20	Space product assurance – Quality assurance
ECSS-Q-20-09	Space product assurance – Nonconformance control system
ECSS-Q-70	Space product assurance – Materials, mechanical parts and processes
NASA-STD-6001	Flammability, Odor, Off-gassing and Compatibility Requirements & Test Procedures for Materials in Environments That Support Combustion

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Terms, definitions and abbreviated terms

3.1 Terms and definitions

The following terms and definitions are specific to this Standard in the sense that they are complementary or additional with respect to those contained in ECSS-P-001 and ECSS-Q-70.

3.1.1 Assembled article

Any component "black box" or assembly of components which represents the article to be used in a spacecraft.

3.1.2 Experiment

An item designed and built to accomplish a specific purpose which can be disassembled and retain its capabilities after re-assembly.

3.1.3 Rack

Structure where different experiments will take place during a manned mission.

3.1.4 Offgassing

The evolution of gaseous products for an assembled article subjected to slight radiant heat in the specified test atmosphere.

3.1.5 Offgassed product

An organic or inorganic compound evolved from a material or assembled article or experiment or rack.

3.1.6 SMAC (Spacecraft Maximum Allowable Concentration)

The maximum concentration of a volatile offgassed product that is allowed in the spacecraft atmosphere for a specified flight duration.

3.1.7 Toxic hazard index (T)

The T value is determined by calculating the 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.

3.2 Abbreviated terms

The following abbreviated terms are defined and used within this Standard.

Abbreviation	Meaning
C	Concentration expressed in $\mu g/m^3$
C_1	Concentration expressed in ppm
$CI^{\pm}$	Chemical Ionization
EI	Electron Ionization
eV	Electron volt
GC	Gas-Chromatograph
MS	Mass-Spectrometer
MWt	Molecular weight
m/z	Ratio Mass to electron
ppm	parts per million
SMAC	Spacecraft Maximum Allowable Concentration
T	Toxic hazard index

NOTE For $\mu g/m^3$, the conversion to ppm is done, using the formula given in annex A.

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Test procedure

4.1 Preparatory conditions

4.1.1 Test specimen preparation

4.1.1.1 Materials

All the materials to be tested shall be classified into one of three categories: surface, volume or weight.

a. Samples based on surface

1. This category is defined as all those materials that are essentially two dimensional. This shall include films, fabrics, coatings, finishes, inks, primers, adhesives, thin film lubricants, tapes and electrical insulating materials.
2. The sample tested shall have a surface of (300 $\pm$ 10) cm^2 per litre of test chamber. For instance coatings and finishes shall be coated on a clean aluminium substrate. Material thickness, curing process and method of application shall be in accordance with the manufacturer's recommendations. Material may be coated on both sides of the aluminium panel. In all cases, only the outer surfaces of a material on the aluminium panel is counted in the surface area determinations. Films, fabrics and similar materials shall be cut to give (300 $\pm$ 10) cm^2 surface. Because these materials are two surfaced in use, both the top and bottom surface shall be counted in determining total surface area. Heat shrinkable tubing or boots shall be applied and shrunk to simulate actual use configuration.

b. Samples based on volume

1. This category is defined as all those materials having a definite volume but having a large real surface area due to surface convolutions or matting. These shall include foams and other blown or foamed materials and insulating padding.
2. Samples of these materials shall be cut to a thickness of (1,25 $\pm$ 0,2) cm unless the existing thickness is less than 1,25 cm. In this case, the existing thickness shall be used. The material shall be cut to such a size as to give (50 $\pm$ 5) cm^2 of total apparent surface per litre of test container volume. All surfaces, tops, bottoms and sides shall be used to compute total surface area. In case where the natural thickness is such that the material cut

would be too large to be placed into the container, two or more pieces may be cut as long as the total surface area requirement is met.

c. Samples based on weight

1. This category is defined as all those materials having a definite bulk and not falling into the volume classification. This shall include potting compounds, moulding compounds, cast of formed objects, solid wires and thick plastics.
2. The samples shall be used as much as possible in the supplied configuration and cut to give (5,0 ± 0,25) g per litre of chamber volume. Potted or moulded materials shall be prepared and cut to weight.

4.1.1.2 Assembled articles

Articles shall be tested in the "as received" condition. A full description of material identification and location shall be provided with each article.

The offgassing test shall be performed in a passive mode (without power).

4.1.1.3 Racks

Racks shall be tested in the context of their specific requirements, in relation to e.g. safety, non-degradation of equipments or vacuum resistance.

The offgassing test shall be performed in a passive mode (without power).

4.1.2 Cleaning

The cleaning and other treatment of the test article shall be the same as that to which the sample is submitted before integration into the spacecraft.

4.1.3 Identification

Materials submitted for testing shall be accompanied by a completed Materials Identification, with technical specifications, expected degradation during the test, cure and post-cure noted. For assembled articles and racks see 4.1.1.2 and 4.1.1.3 above.

4.2 Test facility

4.2.1 General

The basic equipment for the performance of this test shall consist of a sealed test chamber, a sampling capability and the analytical equipment.

4.2.2 Test chamber

The test chamber shall be of the hermetically sealable type and shall be of a sufficient size so that the material or assembled article can be adequately contained, by using the volume of the specimen equivalent to 1/3 of the test chamber. It should not be considerably larger than the article under test in order to avoid over-dilution of the offgassed products in the test chamber. It shall be constructed of materials that do not offgass under the most severe conditions.

The test chamber shall be easily accessible for any required cleaning operation and shall allow full visibility of the sample under test. In order to facilitate the process of chamber cleaning, the chamber should have the capability to be evacuated to a pressure of 1,3 Pa or less, otherwise a facility shall be included whereby the chamber can be purged with a suitable chamber heater, either internal or external, with an upper temperature limit of not less than 80 °C. Suitable feed-through connections shall be fitted to the chamber so that the temperature at various points on the article or rack may be measured. Heatable gas line connections shall be included for sampling of the test atmosphere.

The temperature control system shall be capable of maintaining the temperature in the test chamber uniformly within ± 2 °C of the designated test temperature. The test chamber instrumentation shall have the capability to continuously record the temperature.

The test chamber shall be equipped with an internal fan for the correct mixing of the test atmosphere.

A pressure gauge such as a Bourdon manometer shall be incorporated such that the test pressure can be measured to an accuracy of ± 1 300 Pa.

4.2.3 Sampling equipment

The sampling equipment shall consist of two basic types. The first of these is direct atmosphere sampling using suitable containers of accurately known volume (such as chromatograph gas sampling loops and evacuated glass chamber) for subsequent direct gas analysis. The second is dynamic atmosphere sampling performed by passing a known volume of the test atmosphere through an enrichment device (such as a pre-concentration adsorption trap and a cool empty loop). Additional sampling devices may be included such as specific gas monitoring equipment.

The sampling volume extracted should not exceed 25 % of the total volume of the facility.

4.2.4 Analytical equipment

The analytical equipment shall be such that it shall enable the objective of this test to be met. The basic equipment shall be:

a. A desorption or extraction device for the recovery and subsequent injection of the pre-concentrated contaminants into the following analytical instruments.

b. A gas-chromatograph with, as a minimum, a flame ionization detector, a temperature programming facility and the necessary recording equipment. In order to obtain maximum separation capability, a capillary GC column is required.

c. A second gas-chromatograph equipped with a gas sampling loop, pneumatic valves, a methanizer and a flame ionization detector, used for the determination of carbon monoxide, methane and total organics (screening test). Packed columns in this case are sufficient.

d. A mass-spectrometer, preferably connected to the gas chromatograph, for the identification of the offgassed products where this cannot be done by gas-chromatography alone.

   A typical mass-spectrometer has the following parameters:

   - Mass range: 1–700
   - Excitation: 70 eV
   - Mode: EI/CI$^{\pm}$
   - Resolution (Static)
     At the slit settings provided, resolution definition (10 % valley) shall be within the following values:

Size of the slit	Resolution	Organic reference equivalent	
4^{th} (large)	600	(1 666 ppm)	± 20 %
3^{rd}	2 000	(500 ppm)	± 20 %
2^{nd}	4 000	(250 ppm)	± 20 %
1^{st} (small)	10 000	(250 ppm)	± 10 %

- The GC/MS parameters shall be such that 100 picograms of Methyl Stearate injected on to the GC capillary column gives a signal to noise ratio of 50:1, for a MS source on the molecular ion at m/z 298.

e. The necessary calibration devices (external standards, such as a complex gas mixture).

NOTE An infrared spectrometer with a multipath gas cell, can be used as a complementary system of investigation, in particular with respect to the identification of inorganic contaminants.

4.2.5 Gas supplies

All carrier gases used (Helium, Hydrogen) shall be of a purity not less than 99,99 %.

4.3 Test chamber certification

Prior to testing the chamber shall be heated to at least 70 °C for a period of 24 hours, either under a clean vacuum of < 1,3 Pa or with a continuous purge of clean dry nitrogen or helium gas. The chamber temperature shall then be reduced to the test temperature and the chamber filled with the specified test atmosphere to the test pressure and conditioned for 72 hours. The chamber atmosphere shall be analysed for residual contamination (background). The chamber shall be certified as clean for use if the background level is sufficiently low to permit detection and quantification of offgassed products from the test specimen (a reasonable value for the background of larger facilities should be less than 20 ppm or 59 μg/l, in Pentane equivalent).

4.4 Test performance

4.4.1 Test conditions

The test conditions shall be (unless otherwise specified):

Test temperature: 50 °C ± 2 °C

Test atmosphere: at least 20,9 % oxygen, with the balance nitrogen or argon.

Test pressure: 1 atmosphere pressure (1 013 hPa at 50 °C)

Test duration: 72 hours ± 1 hour

4.4.2 Test procedure

The procedure detailed below shall be equally applicable for both materials and assembled articles.

a. Place the clean, weighed test item into the test chamber and ensure that, for assembled articles, experiments or racks, all necessary electrical connections are made. Place the various temperature sensors on the spots to be monitored and close the chamber. Expose the experiment to a vacuum of less than 100 hPa. The exposure at this pressure shall be less than 3 minutes.

b. In case the experiment is not resistant to vacuum, connect the chamber to the pressurisation system and purge it with the test atmosphere (through a molecular sieve trap) with a minimum volume of three times the chamber capacity. After completion of the purge, adjust the test atmosphere to a pressure such that the chamber will be at test pressure (940 hPa at room temperature shall provide a pressure close to 1 atmosphere at 50 °C).

c. At the end of the time duration defined allow the chamber to cool to room temperature, by passive cooling. Connect the sampling equipment to the chamber, then take samples of the atmosphere using the methods described previously. When the chamber is at room temperature, the samples shall be taken during a period which shall not exceed 12 hours.

d. After the sampling, open the chamber and note any relevant information (such as a particular odour remaining in the chamber and condensation).

4.4.3 Analysis of samples

4.4.3.1 Materials screening test

This test shall be applied only to individual materials.

The sample analysis shall identify the following:

a. quantity of carbon monoxide offgassed in μg/g of material tested;

b. quantity of total organic (expressed as pentane equivalents) offgassed in μg/g of material tested;

c. the identity and quantity of each contaminant offgassed in an amount in excess of 10 μg/g of material tested.

4.4.3.2 General test for materials and assembled articles

In specific cases for materials tested, and in general for assembled articles and racks, the sample analysis shall identify and quantify all contaminants present.

4.5 Acceptance limits

4.5.1 Materials

As a preliminary screening test a material shall show offgassing levels below the following limits:

Carbon Monoxide: 25 μg/g of material tested;

Total Organics: 100 μg/g of material tested.

In cases where materials are added (for instance as a result of repair or rework) to an assembled article which has already been subjected to test, the material shall be tested and shall meet the same offgassing limits as the assembled article.

4.5.2 Assembled articles, experiments and racks

No definitive and final acceptability is defined by this test. The data obtained shall be evaluated for the assessment of any possible toxic hazard due to the volatile contamination evolved from the assembled article under investigation and impacts on the potential toxicity of the total quantity of offgassed products from all contaminant generating items for a given mission.

However, the following primary acceptance criteria shall be met:

a. The quantity of each individual offgassed product shall result in a predicted spacecraft concentration less than the SMAC value for that product.

b. In the case where the conditions of a. above are not met this shall be considered as cause for rejection of the article concerned. Should this course of action be agreed then retest may be necessary afterwards.

c. The toxic hazard index T shall be determined by calculating the 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 T value shall not exceed 0,5.

d. Amount of each contaminant shall be expressed in $\mu g/m^3$ of the manned space vehicle crew compartment.

4.6 Quality assurance

4.6.1 General

The quality assurance requirements are defined in ECSS-Q-20. Particular attention shall be given to the following points.

4.6.2 Data

The quality records (e.g. logbooks) shall be retained for at least ten years or in accordance with project contract requirements, and contain as a minimum the following.

4.6.2.1 Materials

a. Material designation: Type of product
Chemical nature
Manufacturer
Procurements, standards specifications
Summary of processing parameters.

b. Test temperature duration and test atmosphere (normal air, enriched oxygen concentration, nitrogen). Volume of the test chamber. Test pressure at room temperature. Projected volume of the manned spacecraft.

c. Real mass (eventually surface) of the material tested.

d. Results will be expressed in μg/g or μg/cm^2.

4.6.2.2 Assembled articles, experiments and racks

a. Designation of the item. List of contents

b. Manufacturer

c. Reference and serial number

d. Application

e. Location

Any noticeable incident observed during the test on material, article, experiment or rack shall be recorded.

4.6.3 Nonconformance

Any nonconformance which is observed in respect of the test process shall be dispositioned in accordance with the quality assurance requirements, see ECSS-Q-20-09.

4.6.4 Calibration

Each reference standard and piece of measuring equipment shall be calibrated. Any suspected or actual equipment failure shall be recorded as a project nonconformance report so that previous results may be examined to ascertain whether or not re-inspection and retesting is requires. The customer shall be notified of the nonconformance details.

4.6.5 Traceability

Traceability shall be maintained throughout the process from incoming inspection to final test, including details of test equipment and personnel employed in performing the task.

Annex A (informative)

Formula

Conversion from μg/m^3 to ppm

$$C = C_1 \times MWt \times \frac{10^3}{24,47} \times \frac{P}{1\,013} \times \frac{298}{273 + t}$$

Where:

C	concentration (μg/m^3)
C_1	concentration (ppm)
MWt	molecular weight (g)
P	pressure (hPa)
t	room temperature (°C) (End of test)
24,47	molecular volume at 25 °C (l)

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Annex B (informative)

Example of analytical procedure

The data given below is for information purposes only and reflects the experimental system in use at one laboratory. It is included here for the purpose of guidance only.

- **Test Chamber**

  HERAEUS model VHT 85/110

  Volume: 718 l

  From 22 °C (room temperature) to 50 °C: 20 minutes

  Fan: ON

  Experiment exposed 72 hours at 50 °C, then STOP the heater

  Recovering time to reach room temperature and 940 hPa: 12 hours

  Clean and dry synthetic air is used, or pure nitrogen.

- **Sampling**
  - **Gas**
    5 litre glass bottle
  - **Ceramic Traps**
    Ceramic trap model 1-010 R filled with Carbopack B® from SUPELCO 60 mesh - 80 mesh: Equivalent to 100 m^2
  - Pneumatic pumping system, flow rate: 24,33 ml/min
  - Gas volume passing through the trap: 243,3 ml

- **Direct sample**

  This analysis is performed to establish the real value of carbon monoxide (CO) and methane (CH_4) produced by the experiment.

  The analytical equipment is a Gas Chromatograph equipped with a Methanizer and a Flame Ionization Detector (FID).

  The columns (2) are packed with Molecular Sieve and Porapak N®. A backflush is used during the first period of the elution.

- **Preconcentrated sample**

  The ceramic trap 1-01-R filled with 243,3 ml of contaminated air is desorbed by using a microwaves desorption system, model MW-1A from REKTORIK (CH).

  Desorption time: 30 seconds

  Energy: 1 425 Ah 10^{-6}

- **Analytical Equipment**

  A Gas Chromatograph equipped with a DB1701® column from J.W

  Dimensions: 60 m × 0,320 mm

  Film thickness: 0,25 μm

  Temperature programme

  20 minutes at -20 °C, then 1 °C/min to 150 °C

  150 °C during 15 minutes then 2 °C/min to 200 °C

  Detector

  Flame Ionization Detector (FID) at 180 °C

  Thermal Conductivity Detector (TCD) at 180 °C

  Gas Carrier

  Helium: 99,999 %

  Flow rate: 2 ml/min

  A Mass Spectrometer, model Profile®, from KRATOS (UK)

  Magnet system

  Mass range: 1-500

  Excitation (Electron beam): 70 eV

  Mode: EI

  Scan rate: 0,6 seconds per decade

  Resolution: 600

- A SUN® computer, using the MACH3® software, able to store simultaneously the MS and the GC signals (FID, TCD).

- The quantification is done by the GC signal, the identification, by the MS signal, compared with 80 000 spectrum in memory.

ECSS Document Improvement Proposal

1. Document I.D.	2. Document date	3. Document title
ECSS‐Q‐70‐29A	30 July 1999	The determination of offgassing products from materials and assembled articles to be used in a manned space vehicle crew compartment
4. Recommended improvement (identify clauses, subclauses and include modified text or graphic, attach pages as necessary)		
5. Reason for recommendation		
6. Originator of recommendation		
Name:	Organization:	
Address:	Phone: Fax: E‐Mail:	**7. Date of submission:**
8. Send to ECSS Secretariat		
Name: W. Kriedte ESA‐TOS/QR	Address: ESTEC, P.O. Box 299 2200 AG Noordwijk The Netherlands	Phone: +31‐71‐565‐3952 Fax: +31‐71‐565‐6839 E‐Mail: wkriedte@estec.esa.nl

Note: The originator of the submission should complete items 4, 5, 6 and 7.

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