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Space product assurance

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

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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.

This Standard has been prepared by the ECSS Executive Secretariat, endorsed by the Document and Discipline Focal points, and approved by the ECSS Technical Authority.

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ECSS-Q-70-29A 30 July 1999	First issue Transforming ESA PSS-01-729 into an ECSS Standard
ECSS-Q-70-29B	Never issued
ECSS-Q-ST-70-29C 15 November 2008	Second issue Redrafting ECSS-Q-70-09A according to ECSS drafting rules and new template.

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Tables

1 Scope

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.

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.

In this Standard the supplier means the testing authority that is responsible for specifying and executing the offgassing tests.

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.

This standard may be tailored for the specific characteristics and constrains of a space project in conformance with ECSS-S-ST-00.

2 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 revision 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 more recent editions of the normative documents indicated below. For undated references, the latest edition of the publication referred to applies.

ECSS-S-ST-00-01	ECSS system – Glossary of terms
ECSS-Q-ST-10-09	Space product assurance – Nonconformance control system
ECSS-Q-ST-70	Space product assurance – Materials, mechanical parts and processes

3 Terms, definitions and abbreviated terms

3.1 Terms defined in other standards

For the purpose of this Standard, the terms and definitions from ECSS-S-ST-00-01 and ECSS-Q-ST-70 apply.

3.2 Terms specific to the present standard

3.2.1 assembled article

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

3.2.2 experiment

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

3.2.3 offgassing

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

3.2.4 offgassing product

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

3.2.5 rack

structure in which different experiments take place during a manned mission

3.2.6 SMAC (Spacecraft Maximum Allowable Concentration)

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

3.2.7 toxic hazard index (T)

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

NOTE Further details on the calculation of this T-value can also be obtained in NASA-STD-6001.

3.3 Abbreviated terms

For the purpose of this Standard, the abbreviated terms from ECSS-S-ST-00-01 and the following apply:

Abbreviation	**Meaning**
C	concentration expressed in µg/m³
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 charge
ppm	part per million
SMAC	Spacecraft Maximum Allowable Concentration
T	toxic hazard index

NOTE For µg/m3, the conversion to ppm is done, using the formula ((3-1) given in clause 3.4

3.4 Formula for conversion

Formula (3-1) shows the conversion from µg/m³ to ppm:

$$C = C_1 \times MW_t \times \frac{10^3}{24,47} \times \frac{P}{1013} \times \frac{298}{273 + t} \quad (3\text{-}1)$$

Where:

- *C* is the concentration (µg/m³)
- *C_1* is the concentration (ppm)
- *MWt* is the molecular weight (g)
- *P* is the pressure (hPa)
- *t* is the room temperature (°C) (End of test)
- 24,47 is the molecular volume at 25 °C (l)

4 Requirements

4.1 Preparatory conditions

4.1.1 Test specimen preparation

4.1.1.1 General preferences

a. The supplier shall classify all materials to be tested into one of the three categories "Surface", "Volume" and "Mass" described in 4.1.1.2, 4.1.1.3 and 4.1.1.4, respectively.

4.1.1.2 Samples of category "Surface"

a. The category "Surface" shall include films, fabrics, coatings, finishes, inks, primers, adhesives, thin film lubricants, tapes and electrical insulating materials.

NOTE This category is defined for materials that are essentially two dimensional.

b. The supplier shall select test samples with a surface of (300 ± 10) cm^2 per litre of test chamber.

c. Coatings and finishes shall be coated on a clean aluminium substrate.

d. Material thickness, curing process and method of application shall be in accordance with the manufacturer's recommendations.

NOTE Only the outer surfaces of a material on the aluminium panel are counted in surface area determinations.

e. The supplier shall cut films, fabrics and similar materials to ensure a surface of (300 ± 10) cm^2.

NOTE Because materials are often "two surfaced" in use, the total surface area is determined by counting, the top and the bottom surface.

f. Heat shrinkable tubing or boots shall be applied and shrunk to simulate actually used configuration.

4.1.1.3 Samples of category "Volume"

a. The category "Volume" shall include foams and other blown or foamed materials and insulating padding.

NOTE This category is defined for materials having a defined volume but a large real surface area due to surface convolutions or matting.

b. The supplier shall cut samples to a thickness of (1,25 ± 0,2) cm unless the existing thickness is less than 1,25 cm. In this case, the existing thickness shall be used.

c. The supplier shall cut the samples to a surface size of (50 ± 5) cm^2 per litre of test container volume.

d. The supplier shall use all surfaces, tops, bottoms and sides to calculate the total surface area.

e. In cases where the cut pieces of the sample do not fit into the container because of its size, the supplier shall cut two or more pieces, as long as 4.1.1.3d. is met.

4.1.1.4 Samples of category "Mass"

a. The category "Mass" shall include potting compounds, moulding compounds, cast of formed objects, solid wires and thick plastics.

NOTE This category is defined for materials having a defined bulk but not falling into the volume classification.

b. The supplier shall use the samples as much as possible in the supplied configuration and cut them to give (5,0 ± 0,25) g sample per litre of chamber volume.

c. The supplier shall prepare and cut potted or moulded materials to adequate weight.

4.1.1.5 Assembled articles

a. The supplier shall test assembled articles in the condition in which it was received.

b. The supplier shall perform the offgassing test in a passive mode.

NOTE Passive mode means that the test is performed without any power supply for the assembled article.

4.1.1.6 Racks

a. The supplier shall test racks in the context of their specific requirements, in relation to safety, non-degradation of equipments and vacuum resistance.

b. The supplier shall perform the offgassing test in a passive mode.

4.1.2 Cleaning

a. The customer shall perform the same cleaning and treatment of the test article as the sample undergoes before integration into the spacecraft.

4.1.3 Identification

a. The customer shall accompany materials submitted for testing by a completed Materials Identification, with technical specifications, expected degradation during the test, cure and post-cure noted.

b. A full description of each assembled article, material identification and location shall be provided with each article by the customer.

4.2 Test facility

4.2.1 General requirements

a. A basic facility for the performance of the tests shall include a sealed test chamber, a sampling capability and the analytical equipment.

4.2.2 Test chamber

a. The test chamber shall be hermetically sealable.

b. The test chamber shall be of a sufficient size so that the material or assembled article can be contained.

NOTE Best practice is to use the volume of the specimen equivalent to 1/3 of the test chamber. If the test chamber is considerably larger than the article under test, this can lead to over dilution of the offgassed products in the test chamber.

c. The test chamber shall be constructed out of materials that do not offgass beyond the background level.

NOTE Refer to the note in 4.3.

d. The test chamber shall be accessible for any cleaning operation and shall allow full visibility of the sample under test.

e. For cleaning purposes, the chamber shall have the capability to be evacuated to a pressure of 1,3 Pa or less, or to be purged with clean gas not below 80 °C.

f. Feed-through connections shall be fitted to the chamber.

NOTE This is done so that the temperature at various points on the article or rack may be measured.

g. Heatable gas line connections shall be included for sampling of the test atmosphere.

h. The temperature control system shall be capable of maintaining the temperature in the test chamber uniformly within ± 2 °C of the designated test temperature.

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

j. The test chamber shall be equipped with an internal fan for the convection of the test atmosphere.

k. A pressure gauge shall be installed such that the test pressure can be measured to an accuracy of ± 1 300 Pa.

4.2.3 Sampling equipment

a. The sampling equipment shall consist of the following two basic types:

   1. Direct atmosphere sampling using containers of accurately known volume for subsequent direct gas analysis.

      NOTE For example, chromatograph gas sampling loops and evacuated glass chambers.

   2. Dynamic atmosphere sampling performed by passing a known volume of the test atmosphere through an enrichment device.

      NOTE 1 For example, a pre-concentration adsorption trap and a cool empty loop.

      NOTE 2 Additional sampling devices can be included such as specific gas monitoring equipment.

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

4.2.4 Analytical equipment

a. A desorption or extraction device shall be used for the recovery and subsequent injection of the pre-concentrated contaminants into the analytical instruments specified in 4.2.4b, 4.2.4c., and 4.2.4d.

b. A gas chromatograph with at least one flame ionization detector, a temperature programming facility, the necessary data recording equipment and a capillary GC column shall be used.

   NOTE The capillary GC column is used in order to obtain maximum separation capability.

c. A second gas chromatograph equipped with a gas sampling loop, pneumatic valves, a methanizer and a flame ionization detector shall be used for the determination of carbon monoxide, methane and total organics (screening test).

   NOTE Packed columns are sufficient in this case.

d. A mass spectrometer, preferably connected to the gas chromatograph, and with the following parameters shall be used for the identification of the offgassed products where this cannot be done by gas chromatography alone:

1. Mass range: 1-700.
2. Excitation: 70 eV.
3. Mode: EI/CI$^{\pm}$.

4.2.5 Resolution (Static): at the slit settings provided, the resolution definition (10 % valley) is within the values listed in Gas supplies

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

NOTE For example Helium or Hydrogen.

1. Table 4-1.
2. The GC/MS parameters are 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.

b. Calibration devices shall be used.

NOTE 1 For example, external standards, such as a complex gas mixture.

NOTE 2 An infrared spectrometer with a multipath gas cell for use as a complementary system of investigation, in particular with respect to the identification of inorganic contaminants.

4.2.6 Gas supplies

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

NOTE For example Helium or Hydrogen.

Table 4-1: Slits setting

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

4.3 Test chamber certification procedure

a. The test chamber certification procedure shall be as follows:

1. Heat the chamber prior to testing 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.
2. Reduce the chamber temperature to the test temperature and fill the chamber with the specified test atmosphere to the test pressure and condition it for 72 hours.

3. Analyse the chamber atmosphere for residual contamination.

4. Certify the chamber as clean for use if the background level is sufficiently low to permit detection and quantification of offgassed products from the test specimen.

   NOTE Best practice is to have a background level less than 20 ppm or 59 mg/l, in Pentane equivalent.

4.4 Test procedure

4.4.1 Test conditions

a. The test conditions shall be as follows:

   1. Temperature: 50 °C ± 2 °C.
   2. Atmosphere: at least 20,9 % oxygen, with the balance nitrogen or argon.
   3. Pressure: 1 atmosphere pressure (1 013 hPa at 50 °C).
   4. Duration: 72 hours ± 1 hour.

4.4.2 Test execution

a. The test procedure for both materials and assembled articles shall be as follows:

   1. Place the clean, weighed test item into the test chamber and ensure, for assembled articles, experiments or racks, that all electrical connections are made.
   2. Place the various temperature sensors on the spots to be monitored and close the chamber.
   3. Expose the experiment to a vacuum of less than 100 hPa and less than 3 minutes.
   4. 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.
   5. After completion of the purge, adjust the test atmosphere to the test pressure.

      NOTE 940 hPa at room temperature provides a pressure close to 1 atmosphere at 50 °C.

   6. At the end of the defined duration, allow the chamber to cool down to room temperature by passive cooling.
   7. Connect the sampling equipment to the chamber and take samples of the atmosphere within 12 hours after room temperature has been reached.

8. Open the chamber and note any relevant information.

   NOTE For example, particular odour remaining in the chamber and condensation.

4.4.3 Analysis of samples

4.4.3.1 Materials screening test

a. A material screening test shall be applied only to individual materials.

b. The sample analysis shall identify the following:

   1. Quantity of carbon monoxide offgassed in µg/g of material tested.

   2. Quantity of total organic (expressed as pentane equivalents) offgassed in µg/g of material tested.

   3. 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

a. 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

a. For materials the following acceptance limits shall apply:

   1. Carbon Monoxide: 25 µg/g of material tested.

   2. Total Organics: 100 µg/g of material tested.

b. In cases where materials are added to an assembled article which has already been subjected to test, the material shall be tested and shall meet the same acceptance limits as the assembled article.

   NOTE For example, materials can be added as a result of repair or rework.

4.5.2 Assembled articles, experiments and racks

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

   NOTE When this condition is not met, consider the rejection and retest of the article concerned.

b. 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.

c. 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.

e. The supplier shall assess the possible toxic hazards.

NOTE Those toxic hazards due to the volatile contamination evolved from the assembled article under investigation and for impacts on the potential toxicity of the total quantity of offgassed products from all contaminant generating items for a given mission.

4.6 Quality assurance

4.6.1 Data

a. The supplier shall retain the quality records for at least ten years, or in accordance with project business agreement requirements.

NOTE For example, the logbooks.

b. The supplier shall provide the offgassing evaluation report in conformance with Annex A – DRD for customer approval.

4.6.2 Calibration

a. The supplier shall calibrate any measuring equipment to traceable reference standards.

b. The supplier shall record any suspected or actual equipment failure as a project nonconformance report according to ECSS-Q-ST-10-09.

NOTE This is to ensure that previous results are examined to ascertain whether or not re-inspection and retesting is necessary.

Annex A (normative) Offgassing evaluation report - DRD

A.1 DRD identification

A.1.1 Requirement identification and source document

This DRD is called from ECSS-Q-ST-70-29, requirement 4.6.1b.

A.1.2 Purpose and objective

The purpose of the document is to describe the contents of the offgassing evaluation report.

A.2 Expected response

A.2.1 Scope and content

<1> Materials, assembled articles and racks designation

a. The report shall contain the following information for the material:

1. ID
2. Type of product
3. Chemical nature
4. Manufacturer
5. Procurements
6. Standards specifications
7. Summary of processing parameters.

b. The report shall contain the following information for the assembled articles and racks:

1. Designation of the item
2. List of contents
3. Manufacturer
4. Reference and serial number

5. Application
6. Location.

c. The report shall contain the description of the samples.

<2> Test equipment

a. The report shall contain the volume of the test chamber.

<3> Test procedure

a. The report shall contain the following information:

1. Test temperature duration
2. Test atmosphere (normal air, enriched oxygen concentration, nitrogen)
3. Test pressure at room temperature.

<4> Test result

a. The report shall contain the following information:

1. Result of analysis
2. Projected spacecraft volume.

b. The report shall contain any observation during and after the test on material, assembled article or rack.

NOTE Results are expressed in µg/g or µg/cm^2.

A.2.2 Special remarks

None.

Annex B (informative) Example of test method procedure

B.1 Introduction

The data given in B.2 to B.4 is for information purposes and reflects possible experimental system surroundings that can be found in any laboratory. It is included here for the purpose of guidance and serves as an example assembly.

B.2 Test chamber

a. HERAEUS model VHT 85/110:

1. Volume of test chamber: 718 l.
2. Temperature rise: From 22 °C (room temperature) to 50 °C in 20 minutes.
3. Set the fan to ”on”.
4. Expose the experiment for 72 hours at 50 °C, and then stop the heater.
5. Recovering time to reach room temperature and 940 hPa: 12 hours.
6. Clean and dry synthetic air or pure nitrogen is used.

B.3 Sampling

B.3.1 Gas

5 litre glass bottle.

B.3.2 Ceramic Traps

a. Ceramic trap model 1-010 R filled with Carbopack B® from SUPELCO:

1. 60 mesh - 80 mesh: equivalent to 100 m^2;
2. Pneumatic pumping system, flow rate: 24,33 ml/min;
3. Gas volume passing through the trap: 243,3 ml.

B.3.3 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 two columns are packed with Molecular Sieve and Porapak N®. A backflush is used during the first period of the elution.

B.3.4 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}

B.4 Analytical equipment

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

  1. Dimensions: 60 m × 0,320 mm
  2. Film thickness: 0,25 µm

b. Temperature programme

  1. 20 minutes at -20 °C, then 1 °C/min to 150 °C
  2. 150 °C during 15 minutes then 2 °C/min to 200 °C

c. Detector

  1. Flame Ionization Detector (FID) at 180 °C
  2. Thermal Conductivity Detector (TCD) at 180 °C

d. Gas Carrier

  1. Helium: 99,999 %
  2. Flow rate: 2 ml/min

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

  1. Magnet system
  2. Mass range: 1-500
  3. Excitation (Electron beam): 70 eV
  4. Mode: EI
  5. Scan rate: 0,6 seconds per decade
  6. Resolution: 600

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

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

Bibliography

ECSS-S-ST-00	ECSS system – Description, implementation and general requirements
NASA-STD-6001	Flammability, Odor, Off-gassing and Compatibility Requirements & Test Procedures for Materials in Environments That Support Combustion