

Space product assurance — Thermal vacuum outgassing test for the screening of space materials

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British Standard

ICS 49.025.01; 49.140

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National foreword

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This British Standard, having been prepared under the direction of the Engineering Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 18 March 2002

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 28, an inside back cover and a back cover.

The BSI copyright date displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

© BSI 18 March 2002

ISBN 0 580 39287 2

EUROPEAN STANDARD

EN 14091

NORME EUROPÉENNE

EUROPÄISCHE NORM

February 2002

ICS 49.025.01; 49.140

English version

Space product assurance - Thermal vacuum outgassing test for the screening of space materials

Assurance produit des projets spatiaux - Essai de dégazage sous vide thermique pour sélection des matériaux d'un projet spatial

Raumfahrtproduktsicherung - Thermal-Vakuum-Ausgastest für die Auswahl von Raumfahrtmaterialien

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Ref. No. EN 14091:2002 E

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Foreword

This document EN 14091:2002 has been prepared by CMC.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2002, and conflicting national standards shall be withdrawn at the latest by August 2002.

It is based on a previous version¹⁾ originally prepared by the ECSS product assurance working group, reviewed by the ECSS Technical Panel and approved by the ECSS Steering Board. The European Cooperation for Space Standardization (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.

This standard is one of the series of space standards intended to be applied together for the management, engineering and product assurance in space projects and applications.

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.

Annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

¹⁾ ECSS-Q-70-02A

Introduction

The acceleration of the outgassing process results from exposure to vacuum at an elevated temperature. The method described in this standard gives reliable data for the outgassing properties of materials at 125 °C. However, some materials have different kinetics at other temperatures. Nevertheless, comparisons are possible at other temperatures, provided that the kinetics of the outgassing phenomena are similar (defined activation energy of similar magnitude for the materials to be compared). Furthermore, the measurement of contamination potential is comparative and strictly valid only for collectors at 25 °C with similar sticking coefficients.

A basic screening test method is detailed in this standard. The data obtained are not intended to be used for contamination predictions; however, some worst-case analyses can be made with the test data, the masses of the relevant materials and the view factors with respect to contamination-sensitive elements.

1 Scope

This European Standard specifies a thermal vacuum test to determine the outgassing properties of materials proposed for use in the fabrication of spacecraft and associated equipment, for vacuum facilities used for flight hardware tests and for certain launcher hardware.

This standard covers the following:

- critical design parameters of the test system;
- critical test parameters such as temperature, time, pressure;
- material sample preparation;
- conditioning parameters for samples and collector plates;
- presentation of the test data;
- acceptance criteria;
- certification of test systems and their operators by audits and round robin tests.

The test described in this standard is applicable for all unmanned spacecraft, launchers, payloads, and experiments. The test is also valid for external hardware of inhabited space systems and for hardware to be used in terrestrial vacuum test facilities.

The acceptance criteria for a material, based upon the outgassing test data, depends upon the application and location of the material and can be more severe than the standard requirements as given in 7.2.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 13701, *Space systems - Glossary of terms*.

EN 14097, *Space product assurance — Nonconformance control system*.

ECSS-Q-20, *Space product assurance — Quality assurance*.

ECSS-Q-70, *Space product assurance — Materials, mechanical parts and processes*.

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in EN 13701 and ECSS-Q-70 and the following apply.

3.1.1

bakeout

activity of increasing the temperature of hardware to accelerate its outgassing rates with the intent of reducing the content of molecular contaminants within the hardware

NOTE Bakeout is usually performed in a vacuum environment but may be done in a controlled atmosphere.

3.1.2

cleanroom

room in which the concentration of airborne particles is controlled, and which is constructed and used in a manner to minimize the introduction, generation, and retention of particles inside the room, and in which other relevant parameters, e.g. temperature, humidity, and pressure, are controlled as necessary

[ISO 14644-1:1999]

3.1.3

collected volatile condensable material (CVCM)

quantity of outgassed matter from a test specimen that condenses on a collector maintained at a specific temperature for a specific time

NOTE CVCM is expressed as a percentage of the initial specimen mass and is calculated from the condensate mass determined from the difference in mass of the collector plate before and after the test.

3.1.4

outgassing

release of gaseous species from a specimen under high vacuum conditions

3.1.5

quartz crystal microbalance (QCM)

device for measuring small quantities of mass deposited on a quartz crystal using the properties of a crystal oscillator

3.1.6

recovered mass loss (RML)

total mass loss of the specimen itself without the absorbed water ($RML = TML - WVR$)

NOTE The RML is introduced because water is not always seen as a critical contaminant in spacecraft materials.

3.1.7

sticking coefficient

probability that a molecule, colliding with a surface, stays on that surface before thermal re-evaporation of that molecule occurs

3.1.8

total mass loss (TML)

total mass loss of material outgassed from a specimen that is maintained at a specific constant temperature and operating pressure for a specified time

NOTE TML is calculated from the mass of the specimen as measured before and after the test and is expressed as a percentage of the initial specimen mass.

3.1.9

water vapour regained (WVR)

mass of the water vapour regained by the specimen after the optional reconditioning step

NOTE WVR is calculated from the differences in the specimen mass determined after the test for TML and CVCM and again after exposure to atmospheric conditions and 65 % relative humidity at room temperature (22 ± 3 °C).

3.2 Abbreviated terms

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

Abbreviation	Meaning
ATOX	atomic oxygen
CVCM	collected volatile condensable material
DML	declared material list
PTFE	polytetrafluorethylene
QCM	quartz crystal microbalance
RH	relative humidity
RML	recovered mass loss
RT	room temperature
TML	total mass loss
VCM	volatile condensable material
WVR	water vapour regained

4 Preparatory conditions

4.1 Hazards, health and safety precautions

Particular attention shall be paid to health and safety precautions. A safety check-list is produced below.

- a. Control and minimize hazards to personnel, equipment and materials.
- b. Locate items and controls in such a way that personnel are not exposed to hazards such as burns, electric shock, cutting edges, sharp points or toxic atmospheres.
- c. Provide suitable warning and caution notes in operations, storage, transport, testing, assembly, maintenance and repair instructions and distinctive markings on hazardous items, equipment or facilities for personal protection.

4.2 Material samples

4.2.1 Configuration

4.2.1.1

If the material is made up of several items, it shall be prepared according to the relevant process specification or manufacturer's data in such a quantity as to provide representative samples (a minimum of 12 g, 10 g for the initial test and 2 g for subsequent retest, if proved necessary). The material sample supplied shall be made according to the same process parameters (e.g. curing and baking) as the relevant material to be applied for spacecraft use.

4.2.1.2

The material cuttings are in general made by the test house concerned. Three test specimens of each material shall be prepared as follows:

- a. potting materials and bulky adhesives shall be cast on a PTFE sheet so that a sample a few millimetres thick (preferably 2 mm) can be separated from the PTFE after curing. The sample shall be cut into cubes (1,5 mm to 2 mm per side) before testing;
- b. thin films, coatings, adhesives and adhesive tapes shall be applied to a degreased, dried metal foil of known thickness, e.g. aluminium foil of typical $16 \mu\text{m}$ ($4 \times 10^{-3} \text{g/cm}^2$), and then cut into strips approximately 10 mm wide and rolled up to fit the specimen cup;
- c. non-curing adhesives shall be applied between thin metal foils and shall be prepared as b. above;
- d. if the substrate is non-metallic, a sample of that substrate shall be submitted for separate testing;
- e. when materials are prepared on substrates, a substrate sample shall be submitted with the material sample;
- f. when primers are applied, the complete system should be tested;
- g. materials such as wires, cables or sleeves, the smallest dimension of which is less than 1,5 mm, shall be cut into pieces about 10 mm long;
- h. materials containing metal parts (such as electrical wires or connectors) shall, if possible, be tested without the metal parts. If not, the ratio of metal mass to total mass shall be stated;
- i. liquids and greases shall be placed in a specimen cup; in some cases it can be more practical to mix the liquid with a neutral filler powder such as silica before placing it in a cup. In the latter case, the ratio of filler mass to total mass shall be stated;
- j. for low density foams a sample mass of 100 mg can be obtained by
 - 1. choosing a sample cup of bigger size, or
 - 2. compressing the foam into the sample cup.

4.2.2 Cleaning

The cleaning and other treatment of the samples shall be the same as that applied to the flight hardware, which the sample is intended to represent, prior to integration into the spacecraft. Further cleaning or other treatments shall not be done. The test house shall test the materials as received without any further cleaning or treatment, unless so requested.

4.2.3 Handling and storage

Samples shall only be handled with clean nylon or lint-free gloves and shall be stored in a controlled area, with an ambient temperature of $(22 \pm 3) ^\circ\text{C}$ and relative humidity of $(55 \pm 10) \%$. Coated surfaces shall be shielded from contact by using polyethylene or polypropylene bags or sheets. Physical damage shall be avoided by packing the polyethylene or polypropylene-wrapped workpieces in clean, dust- and lint-free material. Limited-life materials shall be labelled with their shelf lives and dates of manufacture, or date of delivery if date of manufacture is not known.

4.2.4 Identification of materials

Materials submitted for testing shall be accompanied by a completed material identification data sheet. Table A.1 gives an example of such a data sheet, containing information that can be used, at a later stage, for entering the relevant data in a declared material list (DML) or outgassing database (see clause 6).

4.3 Facilities

4.3.1 Cleanliness

The work area shall be nominally clean with minimum dust, but not necessarily a cleanroom environment. Air used for ventilation shall be nominally filtered to prevent contamination of the sample.

4.3.2 Environmental conditions

During the conditioning of the prepared material samples, the ambient temperature shall be $(22 \pm 3) ^\circ\text{C}$ with a relative humidity of $(55 \pm 10) \%$.

4.4 Equipment

4.4.1 Test equipment

a. Suitable measuring instruments shall be used to monitor the requirements of the process as follows:

Temperature: $10 ^\circ\text{C}$ to $130 ^\circ\text{C}$, $\pm 1 ^\circ\text{C}$ accuracy

Humidity: 40 % to 80 % RH, $\pm 1 \%$ RH accuracy

Vacuum: 10^{-4} Pa, $\pm 10 \%$ accuracy

b. Infrared spectrometer (if applicable) of suitable sensitivity so that an infrared spectrum of the condensed contaminants in the range $2,5 \mu\text{m}$ to $16 \mu\text{m}$ can be obtained.

c. Microbalance: 1×10^{-6} g to 5×10^{-6} g

d. Vacuum oven:

- 1 Pa
- up to $150 ^\circ\text{C}$

4.4.2 Special apparatus

The apparatus consists of an insert located in a common-type vacuum system suitably dimensioned with respect to the insert, able to accommodate the necessary feed-throughs.

The insert consists of a bar (or bars) accommodating 24 regularly spaced specimen compartments ($16 \pm 0,1$) mm in diameter and ($9,6 \pm 0,8$) mm deep. The distance between two adjacent specimen compartments is ($50 \pm 0,8$) mm. The open ends ($6,3 \pm 0,1$) mm in diameter and ($12,7 \pm 0,3$) mm long) of the specimen compartments face the collector plates on the cooling plate(s), which is (are) provided with attachments ensuring a good thermal contact with the collector plates. The distance between the open ends of the specimen compartments and the cooling plate(s) is ($13,45 \pm 0,1$) mm.

Cross contamination between different compartments is reduced by a separator plate(s) ($0,75 \pm 0,1$) mm thick and perforated with ($11,1 \pm 0,1$) mm diameter holes in front of each specimen compartment. The separator plate(s) is (are) situated between the heater bar(s) and the cooling plate(s) at a distance of ($9,65 \pm 0,1$) mm from the latter. Standard collectors are chromium-plated aluminium plates ($33,0 \pm 0,1$) mm in diameter and ($0,65 \pm 0,1$) mm thick. It should be possible to replace them by sodium-chloride or germanium collector plates so that infrared analysis of the condensed materials can be performed. Attention shall be paid to the alignment between the hot bar and the cooling plate(s) (see Figure 1).

A pressure of 10^{-4} Pa shall be reached within one hour with an unloaded system. The vacuum system shall be "oil free"; this shall be checked during each test with the aid of three blank collector plates placed at random. Provisions for maintaining the heater bars and the cooling plates at temperatures other than those mentioned further in this standard shall be available. It is advisable to make provision for a bakeout of the vacuum system as a means of cleaning it in the event of heavy contamination.

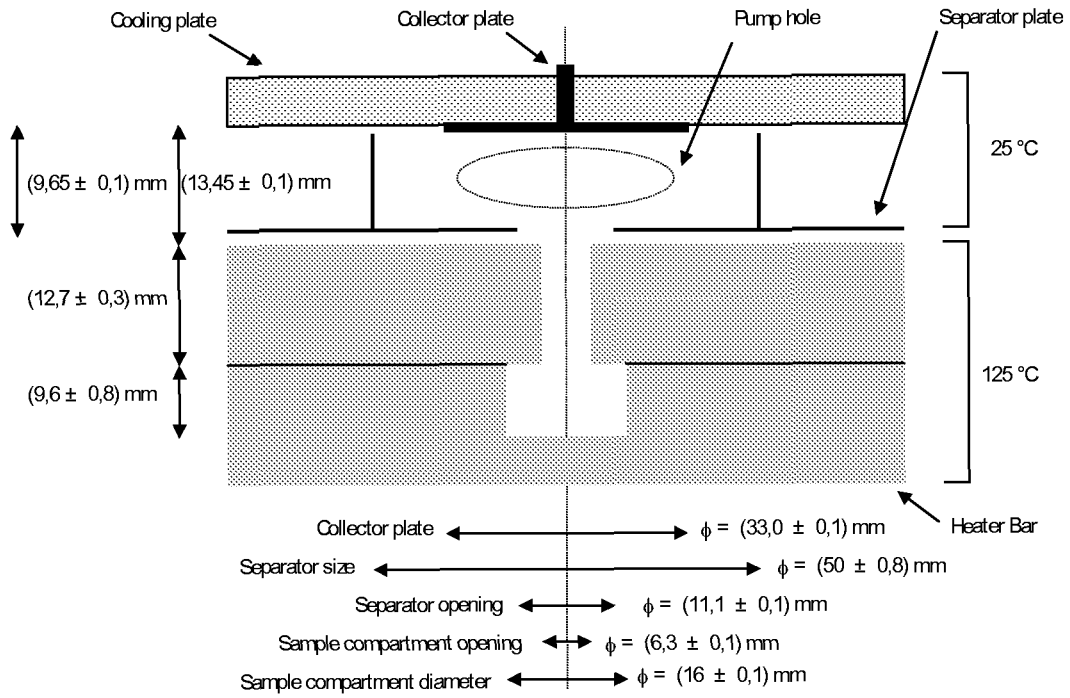


Figure 1 — Micro-VCM equipment

5 Test procedure

5.1 Introduction

Figures 2 and 3 are included as a guide to the test procedures. The sequence for the test is given in the flow chart (Figure 3) and described in more detail in 5.2 below.

5.2 Test process for general spacecraft application

5.2.1 Cleaning of cups and collector plates

Specimen cups and collector plates shall be cleaned with a suitable solvent.

5.2.2 Conditioning of cups and collector plates

The specimen cups shall be conditioned for at least 24 h in an environment of $(22 \pm 3)^\circ\text{C}$ and $(55 \pm 10)\%$ RH. The collector plates shall undergo a bakeout for at least 16 h in a vacuum oven at a pressure lower than 1 Pa, and at a minimum temperature of 125°C . After this bakeout they shall be conditioned for a minimum 24 h in a desiccator containing silica gel. Normal practice during a test shall be to expose three specimens of each material, three empty specimen cups and three collector plates. The three collector plates facing the empty cups shall act as blanks, so that the cleanliness of the equipment can be verified. Corrections based on the blanks shall be taken into account in the actual mass loss calculations.

5.2.3 Conditioning of samples

Material specimens shall be prepared in the manner laid down in 4.2 and shall be conditioned for at least 24 h at $(22 \pm 3)^\circ\text{C}$ and $(55 \pm 10)\%$ RH.

5.2.4 Weighing of samples

The pre-weighed specimen cups shall be filled with 100 mg to 300 mg of specimen (substrate not included) and weighing shall be performed on a microbalance (see 4.4.1 c.) located in a room conditioned at $(22 \pm 3)^\circ\text{C}$ and $(55 \pm 10)\%$ RH just before the loading of the test system.

5.2.5 Weighing of collector plates

The collector plates shall be weighed just before the loading of the test system and, for this purpose, they shall be taken from the desiccator one by one.

5.2.6 Loading of system

The test system shall be loaded with the specimen cups, blank cups, blank collectors, two chromium-plated and one infrared-transparent collector or three chromium-plated collectors per material.

5.2.7 Pump-down and heating

The pump-down of the test system shall be carried out as standard procedure; at 10^{-3} Pa the heater bar(s) shall be brought to 125°C within one hour and the cooling plate(s) shall be controlled at 25°C . These temperatures shall be maintained for a period of 24 h following the instant at which the heater bar(s) reach(es) 125°C .

5.2.8 End of test

After a 24-hour exposure, the heaters shall be switched off and the system shall be vented up to 1×10^4 Pa to 2×10^4 Pa with dry nitrogen or rare gas; cooling shall be continued until the end of the test.

5.2.9 Gas inlet

When the temperature of the heater bar(s) has fallen to 50 °C, which shall take 90 min - 120 min, dry nitrogen or rare gas shall be admitted up to atmospheric pressure.

5.2.10 Unloading of system

The system shall be unloaded as soon as possible; the specimen cups shall be kept in a desiccator for not more than 30 min and the collector plates for about one hour. The specimen cups and collector plates shall be weighed, being taken from the desiccator for this purpose one by one and returned immediately thereafter.

5.2.11 Storage of collector plates

The specimen cups with material samples shall then be stored in a room, at an ambient temperature of (22 ± 3) °C and a relative humidity of (55 ± 10) %, for a nominal 24 h, and then re-weighed.

5.2.12 Infrared analyses

The infrared-transparent collector plates shall be examined in the transmission mode, with the aid of an infrared spectrometer of suitable sensitivity so that an IR spectrum of the condensed contaminants can be obtained (see also 5.2.14).

In special cases the standard metal collector plates can be analysed by infrared reflection techniques, however, no quantitative information can be expected from this reflection method. The contaminants can also be washed from the metal collector plates and the washing liquid can be used for further analyses.

5.2.13 Cleaning of system

After each test, the heater bar(s), condenser plate(s) and screen(s) of the equipment shall be thoroughly cleaned with a suitable volatile solvent, and baking shall be performed at 150 °C if the blank collectors indicate a mass increase $> 30 \mu\text{g}$ during the previous test.

NOTE It is a good practice to bake the system once every four months.

5.2.14 Improved sensitivity of the CVCM measurements

In cases where the CVCM outgassing requirements (see 7.2) are more stringent than the standard detection limits, a quartz crystal microbalance may be installed as a fourth collector plate. The infrared transmission data (see 5.2.12) can also be used for equivalent mass determination as the intensities of the infrared absorption bands are related to the mass of the contaminants by the law of Lambert-Beer.

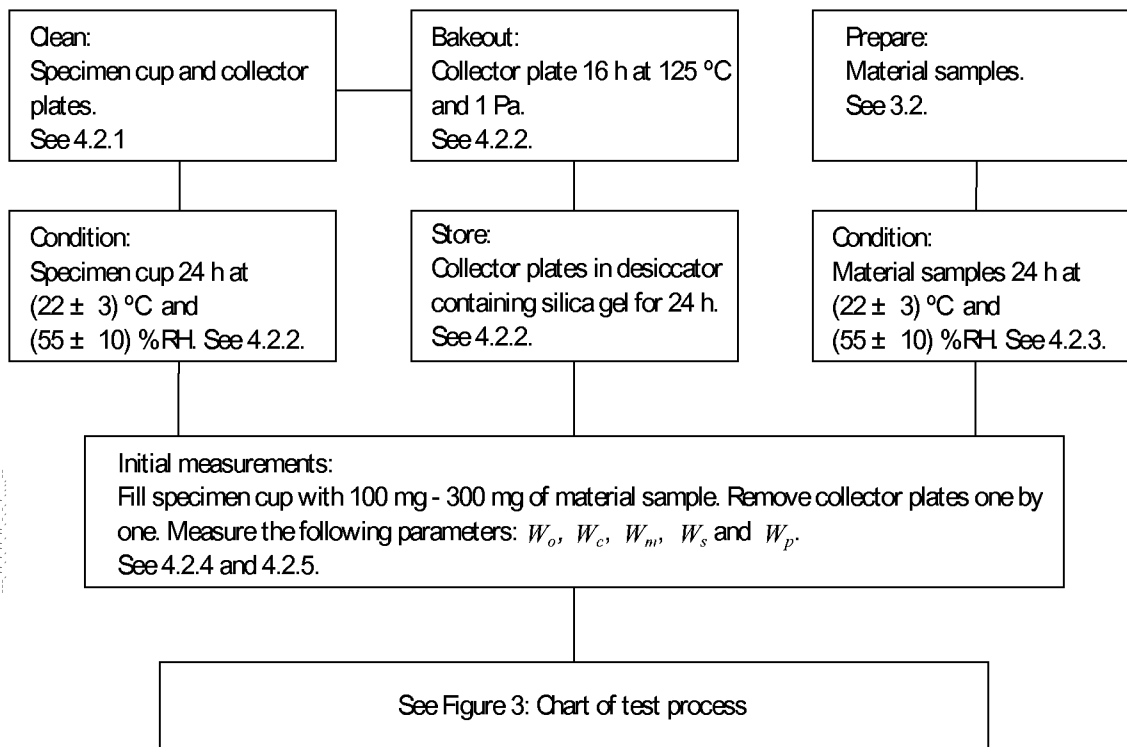


Figure 2 — Flow chart of preparation and initial measurements

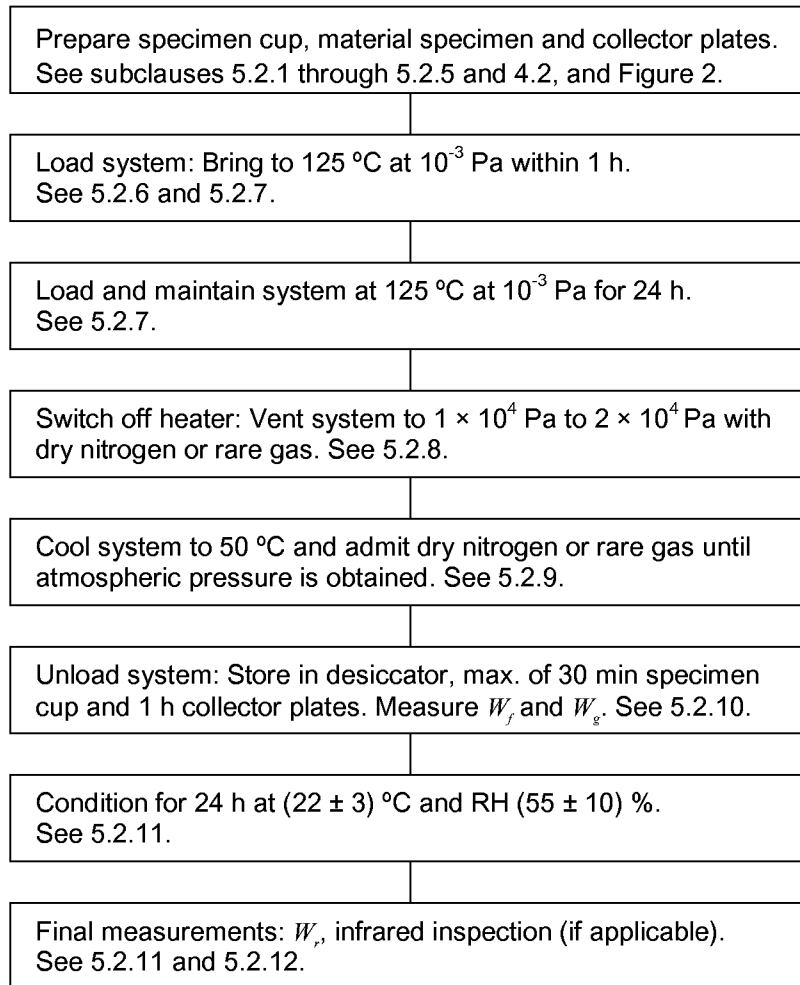


Figure 3 — Flow chart of test process

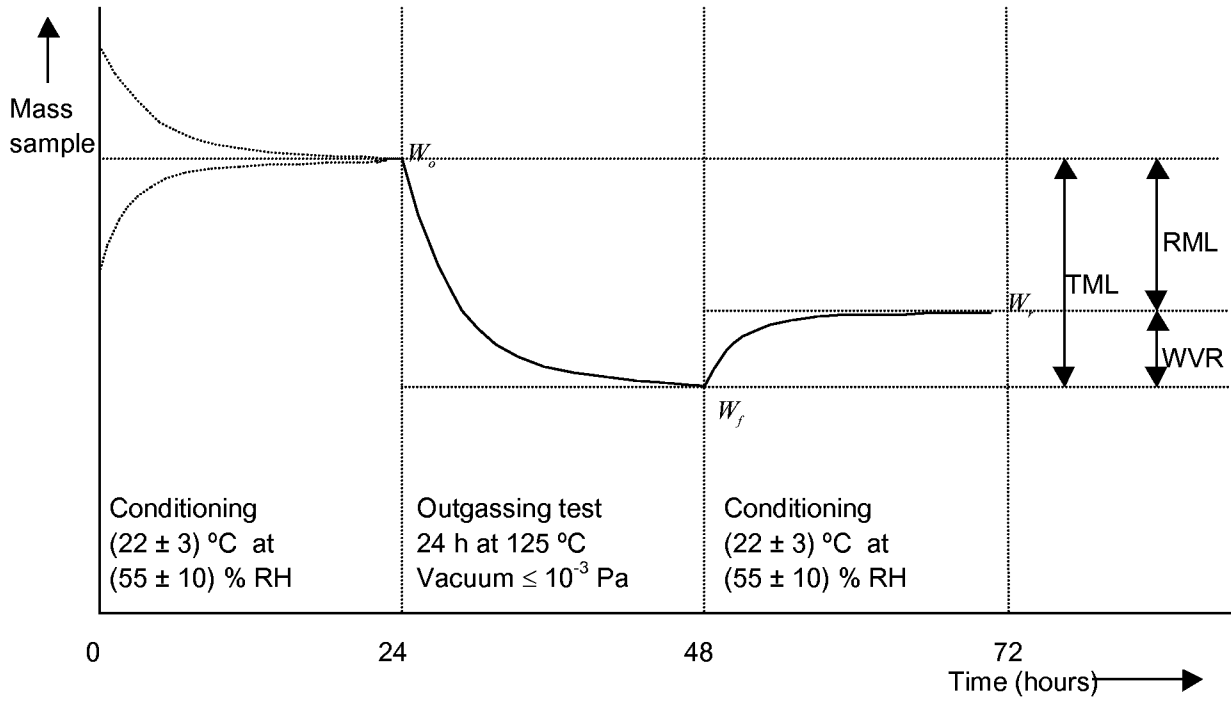


Figure 4 — Parameters for sample

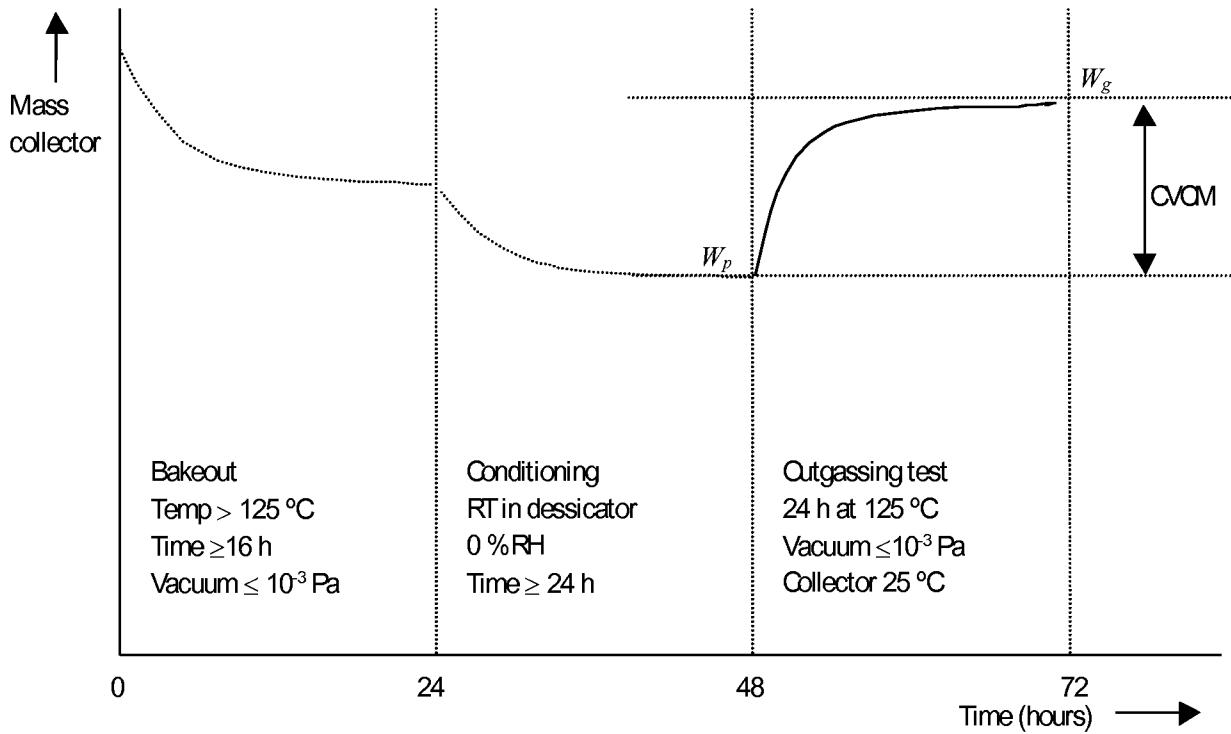


Figure 5 — Parameters for collector plate

6 Reporting of test data

All data obtained during an outgassing test shall be filled in by the test house on a form indicated as the Micro-VCM worksheet. Table A.2 gives an example of such sheet.

The results shall be reported by the test house on a form indicated as the Micro-VCM data sheet. An example of such sheet is given in Table A.3. As a minimum such sheet shall contain a unique sample identification, the material identification, manufacturer, summary process parameters and test results. It should also make reference to the relevant materials identification card (see Table A.1).

The outgassing data are given down to 0,01 % for normal tests, and for tests with increased sensitivity these figures can be one order of magnitude lower.

7 Acceptance limits

7.1 Acceptance limits for a retest of the material

A material is normally tested threefold and the average data of the three values shall be within the limits as given below. In case these limits are exceeded, a retest of the material shall be performed. The variation of the outgassing data can be caused by the material variance or by the variance of the test parameters. The maximum values for "one standard deviation" (S) with respect to the "mean" values derived from the three specimens of each material tested are as follows:

- $S < 1/10$ of the mean values of TML and RML, with a minimum S value of 0,05 %;
- $S < 1/5$ of the mean value of CVCM, with a minimum S value of 0,03 %.

It is advisable to have a material sample of > 1 g in order to be able to repeat a test (see 9.4.2).

7.2 Acceptance limits for application of a material

7.2.1 General

The generally accepted limits for outgassing of materials are listed below. It should be noted that for materials used in the fabrication of optical devices, or in their vicinity, the acceptance limits can be more stringent than those stated below. It is nowadays becoming standard practice to bake critical hardware (such as structural parts, harness, electronic boxes and thermal blankets) to the highest permissible temperature for a few days in order to remove residual contaminants, process contaminants and handling contaminants.

In this respect it is of interest to test materials after such baking as is expected for the hardware. Also, infrared inspection may be invoked if considered necessary (see 5.2.12).

The general outgassing requirements for a material selection are:

RML $< 1,0$ %, CVCM $< 0,10$ %

7.2.2 Corrective actions for high outgassing materials

In case the material outgassing is higher or different from the above general requirements, corrective actions should be taken for typical application as for example:

- if very small mass is used;
- if the location of the material is far away from the sensitive items;
- if the high outgassing material is shielded;

- if the outgassing species (e.g. water) are not seen as a critical containment.

7.2.3 Water absorption of materials

Water absorption of materials is included in the measured TML. The TML data for water absorbing materials such as polyamides, polyimides and polyurethanes, is often above 1,0 %. Water absorption is in most cases reversible and can be controlled by purging of critical hardware with dry gases.

The measuring method as described in this standard takes water reabsorption into account, as both the TML and the RML are measured. (The RML is basically the TML value that does not include reabsorbed water or $RML = TML - WVR$).

In most cases the water absorption of materials is not harmful with respect to contamination and materials with a high TML outgassing and a RML outgassing of < 1 % may be accepted for the normal cases and the general acceptance criteria laid down in 7.2.1 are based upon the following conditions:

- when no equipment at a temperature below -100 °C is involved;
- when the water desorption is fast (e.g. in the case of polyimide films and polyurethane paints);
- when no high voltage equipment is involved;
- when dry gas purging controls the water reabsorption during ground life up to launch.

7.2.4 More stringent outgassing requirements

- The general acceptance limits defined in 7.2.1 should be made more stringent if the materials concerned are used in very critical areas. The use of materials that are deemed acceptable according to the limits stated above does not ensure that the spacecraft system or component remains uncontaminated. Consequently, subsequent functional, development and qualification test shall be used where appropriate to ensure that the material's performance continues to be satisfactory.
- In case water absorption (or desorption) of materials can result in contamination problems, for instance, cryogenic equipment, the outgassing requirements for materials shall be evaluated on a case by case basis.
- For cases (e.g. certain optics) where the CVCM requirements are lower than 0,1 % one shall consider that the CVCM detection limit of the standard Micro-VCM test is around 0,02 %, because of both material variances and measuring errors. For cases where e.g. CVCM < 0,05 % is required, other types of CVCM measurements shall be implemented (see 5.2.14).

8 Quality assurance

8.1 General

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

8.2 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:

- specific mass of the finished product ($/\text{cm}^3$ for bulk solid, $/\text{cm}^2$ for coatings and thin layers, $/\text{cm}$ for wires and threads);
- density of substrate in g/cm^3 or ratio of material mass to total mass of material plus substrate;

- c. clear identification of size, area and mass of test specimen, together with an indication of whether the sample was of the substrate or sandwich type;
- d. the nature of the collector plates;
- e. if an infrared spectrum is obtained of the condensed material, the main wavelength peaks shall be marked with their wavelength value;
- f. any noticeable incident observed during the test shall be recorded;
- g. the masses of specimens and collectors before and after the test. For the definitions of the various masses refer to Table 1;
- h. the deduced outgassing properties (see Table 1, which lists the calculations needed to establish these values);
- i. details of failure mode (if applicable);
- j. a proper identification of the material as stated in 4.2.4.

8.3 Nonconformance

Any nonconformance which is observed in respect of the process or the audit shall be dispositioned in accordance with the quality assurance requirements, see EN 14097. However, retesting is allowed if material fails on the deviation limits (see 7.1).

8.4 Calibration

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

8.5 Traceability

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

Table 1 — Deduced outgassing properties

Term	Calculations	Remarks
TML %	$\frac{W_o - W_f}{W_m} \times 100$	$W_m = W_o - W_c - W_s$
CVCM %	$\frac{W_g - W_p}{W_m} \times 100$	
RML %	$\frac{W_o - W_r}{W_m} \times 100$	W_r is measured on completion of post conditioning
WVR %	$\frac{W_r - W_f}{W_m} \times 100$	
<p>where</p> <p>W_c is the mass of specimen cup;</p> <p>W_f is the total specimen mass just after test;</p> <p>W_g is the final mass of collector plates after test;</p> <p>W_m is the mass of material before test = $W_o - W_c - W_s$;</p> <p>W_o is total specimen mass (material + cup + substrate) before the test;</p> <p>W_p is the initial mass of collector plates before test;</p> <p>W_r is the total specimen mass after test and after 24 h final conditioning at $(22 \pm 3) ^\circ\text{C}$ and $(55 \pm 10) \% \text{RH}$ environment;</p> <p>W_s is the mass of substrate, determined by weighing or by calculation from density and surface area.</p>		

9 Audit of the Micro-VCM test apparatus

9.1 General

The main purpose of this audit is to ensure the validity of test results by comparison of the test data on identical materials by different test houses.

The material outgassing data from test houses for the projects of the customer, obtained in the manner laid down in this standard, are only accepted for the projects of the customer if the test house is certified to perform the Micro-VCM test.

The standard audit requirements are referred to in ECSS-Q-20.

9.2 Initial audit of the system (acceptance)

Once a system has been built it shall be audited by the product assurance department of the customer before it can be accepted for running qualification or quality control tests on materials for use in customer-projects. This initial audit shall at least consist of (but not necessarily be restricted to) the following.

9.2.1 Inspection of apparatus and associated equipment

a. Micro-VCM apparatus:

- internal dimensions, view factor (sample compartment to collector plates) and cross contamination protection;
- pumping system and associated monitoring equipment;

- temperature regulation of sample and collector plates and associated monitoring equipment;
- thermal contact of the temperature sensors.

b. Associated equipment:

- precision balance (reading to 1 µg);
- temperature and relative humidity in the balance room shall be controlled to obtain the required accuracy and reproducibility;
- room for sample pre- and post conditioning;
- vacuum oven for bakeout of collector plates;
- clean sample preparation room;
- infrared apparatus (optional).

9.2.2 Performing a blank test

The purpose of this test is to ensure the apparatus is performing correctly and no self-contamination occurs.

The conditions and procedures for the blank test are identical to the normal Micro-VCM test as described in 5.2, but is performed with empty sample cups.

The results for TML, RML and CVCM (theoretically all zero) are calculated as for a normal VCM-test.

The results shall be within the tolerance as specified for a normal Micro-VCM test, i.e. a mass variation of less than 30 µg on the collector plates.

9.2.3 Performing an actual test

- Seven samples for this test shall be selected and supplied by the customer.
- The conditions and procedure for this test are identical to the normal Micro-VCM-test as described in 5.2.
- The test is performed at the same time by all participants.
- The acceptance limits shall be within the following limits:
 - for CVCM/TML/RML values > 0,2 % within 20 % of the average value of all participants;
 - for CVCM/TML/RML values < 0,2 % within ± 0,05 % of the average value of all participants.

NOTE This test can be part of a "Round Robin Test" test on different apparatus of selected test houses.

9.2.4 Nonconformances

If the initial audit shows a nonconformance, all necessary modifications and corrective actions shall be made by the test house and a follow-up audit shall be made by the customer before a certificate of approval is granted.

9.2.5 Reporting of audit findings

A written report of the initial audit including the certificate of approval shall be delivered within six weeks after the end of the audit provided no nonconformances are detected. This certificate of conformance (example given in annex B) is valid for three years and can be renewed after a successful audit.

9.3 Annual regular review (maintenance) of the system

9.3.1 Inspection of apparatus and associated equipment

Based on the initial test, as described in 9.2.1, a review shall be done of any modifications made to the test facility or apparatus.

9.3.2 Mutual comparability evaluation (testing)

The seven samples for this test shall be selected and supplied by the customer who is also the coordinator for all results and final report. The conditions and procedures for this test are identical to the normal Micro-VCM test as described in 5.2. The test shall be run by all test houses involved in projects of the customer.

9.3.3 Nonconformance

If the inspection of the system or the "Round Robin Test" shows a nonconformance with the applicable audit specification of the customer or the acceptable limits of the test results, actions shall be undertaken by the test house in order to determine the reasons for the nonconformance and a further test shall be performed in accordance with 9.2.3 before a certificate of conformance is renewed.

9.3.4 Reporting of audit findings

A written detailed report of the result of the regular review shall be delivered to all participants within six weeks after the end of the regular review or evaluation testing. The certificate of conformance (example given in annex B) shall be renewed every three years after a successful audit.

9.4 Special review

9.4.1 General

All modifications of the apparatus or associated equipment shall be reported and, if deemed necessary, be audited by the customer before utilization of the modified system for customer's project use. Major modifications shall result in retesting of apparatus as described in 9.2.

9.4.2 Preservation of samples

A quantity of untested material, at least sufficient for a second Micro-VCM test, shall be preserved by the test house for a period of not less than one year and be available for submittal to the customer in case of request by the customer's project product assurance representative or department.

Annex A (informative)

Examples of filled in data sheets

In this annex the following examples are presented:

- Table A.1: Example of filled in materials identification card
- Table A.2: Example of filled in Micro-VCM worksheet
- Table A.3: Example of filled in Micro-VCM datasheet

Table A.1 - Example of filled in materials identification card

Materials identification card	
Description and history of sample a. Trade name and number b. Manufacturer c. Type of product d. Chemical nature e. Processing details: e.g. - joining method - heat treatment - cure and postcure - cleaning method - relevant spec. no	a. Aeroglaze Z306 and Pyrolac P123 b. Lord Corporation/Akzo c. Paint, conductive, black, Primer yellow d. Polyurethane/Epoxy e. Mix ratio Primer P123:Hardener = 100:25 Paint S125:Z306:MEK is 50:100:60 Cure: 24 h at room temperature and 6 days at 65 °C Cleaning with IPA
Batch number Sample quantity Preparation date Prepared by	1108447 A4 sheet 26/03/99 TOS-QMC
Contractor/Experimenter Sample code (refer to the DML item number of the project) Application	Contractor Project/Cost code ESTEC PA manager or originator name and signature XMM - Project PA name
Test specification number	Quality control sample or evaluation sample Evaluation Report number: Results: TML = 1,55 % RML = 0,47 % CVM = 0,00 % <input type="checkbox"/> Accept <input type="checkbox"/> Reject
For materials and processes division use Date received: 12.10.1996 Responsible section: PXQ	Coating optical equipment prEN 14091:2001 Test date: 05.11.1996 Test number: ESTEC 448

Table A.2 - Example of filled in Micro-VCM worksheet

Item no.	Commercial identification or standard designation	W_c cup	W_o total before	W_s substr.	W_m material before	W_c total after	W_c total cond.	W_p collect before	W_g collect after	TML %	RML %	WCR %	CVCM %	Test reference no.	IR date	Observations
1	Honeycomb								average standard							
2	Aeroglaze Z306 and Pyrolac P123	174263	507690	89750	243677	503938	506522	367771	367783	1,54	0,48	1,06	0,00	E 448	None	Black paint with yellow primer
		181391	522828	89231	252206	519016	521730	157207	157213	1,51	0,44	1,08	0,00			
		179771	500914	96667	224476	497303	499780	191537	191569	1,61	0,51	1,10	0,01			
3	Aeroglaze Z306 and Pyrolac P123								average standard	1,55	0,47	1,08	0,01			
									average standard	0,05	0,04	0,02	0,01			
4	Eccosil 4952								average standard							
									average standard							
5	Araldite AV100/HV100								average standard							
									average standard							
6	Electrodag 501								average standard							
									average standard							
7	Solitane 113								average standard							
									average standard							

Table A.3 - Example of filled in Micro-VCM data sheet

Item no.	Commercial identification or standard designation	Chemical nature ISO-1043	Product type	Procurement information manufacturer/ procurement spec.	Summary of process parameters	Use and location	User code contractor project	TML %	RML %	WVR %	CVS M %	Test reference no.	IR- data results	Observations
1	Honeycomb	EP/Al	honeycomb	Casa	Spec Ca-423-95A	structure	Meris-C3							
2	Aeroglaze Z306 and Pyrolac P123	PUR/EP	paint black/ primer yellow	Lord/Akzo	Cure 24 h at RT and 6 d at 65 °C	optical equipment	XMM	1,55	0,47	1,08	0,00	E 448	None	
3	Aeroglaze Z306 and Pyrolac P123	PUR/EP	paint black/ primer yellow	Lord/Akzo	Cure 24 h at RT and 6 d at 65 °C	battery PF3/a12	Mipas-A5							
4	Eccosil-4952	SI	potting	Emerson & Curmin g	Cure 7 d at RT and 24 h at 45 °C	connector	EURECA-Columbus							
5	Araldite AV100/HV100	EP	adhesive	Ciba Geigy	Cure 4 h at 60 °C	insert	ISO-SS-Fok							
6	Electrodag 501	Fluoro Carbon	paint-cond. black	Acheson	as received		SOHO							
7	Solithane 113	PUR	potting	Thiokol	as received		Silex							

Annex B
(informative)

Example of a certificate of conformance

Certificate of conformance	
This certificate of conformance states that the customer declares that the supplier of Micro-VCM data complies with the requirements in EN 14091 and the audit requirements in ECSS-Q-20.	
Customer (incl. Auditor[s])	*
Supplier (incl. Operator[s])	*
Audit criteria according relevant clauses of EN 14091:2002	*
1. Data recording (8.2)	*
2. Nonconformance (8.3)	*
3. Calibration (8.4)	*
4. Traceability (8.5)	*
5. Inspection of apparatus and associated equipment (9.2.1)	*
6. Performing a blank test (9.2.2)	*
7. Performing an actual test (9.2.3)	*
The audit took place on * _____ and the certification is valid from * _____ to * _____	
This certificate is granted by: **	
Name:	Signature:
Function:	Date:

* fill in

** fill in customer and date

1

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