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A thermal vacuum test for the screening of space materials

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ABSTRACT

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

DOCUMENT CHANGE RECORD

Issue number and date	Sections affected	Remarks
Issue 1 March 1983		First release
Issue 2 October 1994	Section 5: Acceptance limits	- Method B deleted
	Section 5: Acceptance limits	- Water sensitive materials

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SECTION 1. SCOPE

This specification describes a thermal vacuum test to determine the outgassing properties of materials by a method proposed for use in the fabrication of ESA spacecraft and associated equipment and for vacuum facilities used for flight hardware tests and for certain launcher hardware.

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SECTION 2. GENERAL

2.1 INTRODUCTION

The acceleration of the outgassing process results from exposure to vacuum at an elevated temperature. This method gives reliable data for the outgassing properties of materials at 125°C. However, some materials may 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 attributes is comparative and strictly valid only for collectors at 25°C, with similar sticking coefficients.

The test method is detailed in this specification. 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 the contamination-sensitive element.

2.2 RELATED DOCUMENTS

Some or all of the content of the documents listed below is directly related to this specification. The applicability of these documents is defined in the contract.

ESA PSS-0l-20	Quality assurance requirements for ESA space systems
ESA PSS-0l-70	Material, mechanical-part and process selection and quality control for ESA space systems
ESA PSS-01-700	The technical reporting and approval procedure for materials, mechanical parts and processes.
ESA PSS-01-705	The detection of organic contamination of surfaces by infrared spectroscopy
ESA PSS-0l-7ll	Product assurance requirements for micro VCM - apparatus and associated equipment

ASTM-E595-90 Total mass loss and collected volatile condensable material from outgassing in a vacuum environment.
(The method described in this standard is based upon similar test equipment. The test method, e.g. as regards humidity control, is partly different and the acceptance limits are not included in this document.)

2.3 ABBREVIATIONS

The definitions listed in Annex A shall apply.

SECTION 3. PREPARATORY CONDITIONS

3.1 HAZARDS/SAFETY PRECAUTIONS

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

(a) control and minimise unavoidable 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 personnel protection.

3.2 MATERIAL SAMPLES

3.2.1 Configuration

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 (at least 10 g). The material sample supplied has to be made according to the same process parameters (e.g. curing and baking) as the relevant material to be applied for spacecraft use.

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 should 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 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 max. 16 μm ($4 \times 10^{-3} g/cm^2$), 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, the density of the substrate shall be stated in g/cm^2.

(f) When primers are applied, the density of the primer as well as that of the coating shall also be stated in g/cm^2.

(g) Materials such as wires, cables or sleeves, the smallest dimension of which is less that 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 may be considered 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 may be obtained by:
   1) Choosing a sample cup of bigger size;
   2) Compressing the foam into the sample cup.

3.2.2 Cleaning

The cleaning and other treatment of the samples shall be the same as that to which the sample will be submitted before incorporation in the spacecraft. Further cleaning or other treatments are not permitted. The Test House shall test the materials as received without any further cleaning or treatment, unless so requested.

3.2.3 Handling and storage

Supplied material samples shall be protected from dust, harmful vapours and damage by suitable packaging (e.g. Al-foil and clean polyethylene bags). Before the preparation the material samples shall be stored in a cleanliness-controlled area, with an ambient temperature of 20 ± 3° C and relative humidity of 55 ± 10%. Samples shall be handled only with clean lint-free gloves. Coated surfaces shall be shielded from contact by using polyethylene or polypropylene bags or sheets. Mechanical damage shall be avoided in the standard way by packing the polyethylene or polypropylene-wrapped workpieces in clean, dust- and lint-free material.

3.2.4 Identification of materials

Materials submitted for testing shall be accompanied by a completed Material Identification Card (see Annex B).

3.3 FACILITIES

3.3.1 Cleanliness

The work area shall be clean and free of dust. Air used for ventilation shall be filtered to prevent contamination of the workpieces by moisture, oil or dust.

3.3.2 Environmental conditions

Durinq the conditioning of the prepared material samples, the ambient temperature shall be 20 ± 1°C with a relative humidity of 65 ± 5%.

3.4 EQUIPMENT

3.4.1 Test equipment

(a) *Suitable measuring instruments* to fulfil the monitoring requirements of the process:

Temperature	10 to 130°C, ± 1°C accuracy
Humidity	40 to 80% RH, ± 1% RH accuracy
Vacuum	10^{-4} Pa(~10^{-6} mbar), ±10% accuracy

(b) *Infrared spectrometer* (if applicable) of suitable sensitivity so that an infrared spectrum between 2.5-16 μm of the condensed contaminants may be obtained.

(c) *Microbalance* - accurate to within ± 1 x 10^{-6} g.

(d) *Vacuum oven* - 1 Pa (10^{-2} mbar) - to 150°C.

3.4.2. 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 feedthroughs.

The insert consists of a bar (or bars) accommodating 24 regularly spaced specimen compartments 16 ± 0.1 mm in diameter and 9.6 ± 0.8 mm deep. The distance between two adjacent specimen compartments is 50 ± 0.8 mm. The open ends (6.3 ± 0.1 mm in diameter and 12.7 ± 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 ± 0.1 mm.

Cross contamination between different compartments is reduced by a separator plate (or plates), 0.75 ± 0.1 mm thick and perforated with 11.1 ± 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 ± 0.1 mm from the latter. Standard collectors are chromium-plated aluminium plates 33.0 ± 0.1 mm in diameter and 0.65 ± 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 has to be paid to the alignment between the hot bar and the cooling plate(s) (see Figure 1).

A pressure of 10^{-4} Pa (1 x 10^{-6} mbar) shall be reached within one hour with an unloaded system. The vacuum system shall be 'oil free'; this is to 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 the specification shall be available. It is advisable to make provision for a bake out of the vacuum system as a means of cleaning it in the event of heavy contamination.

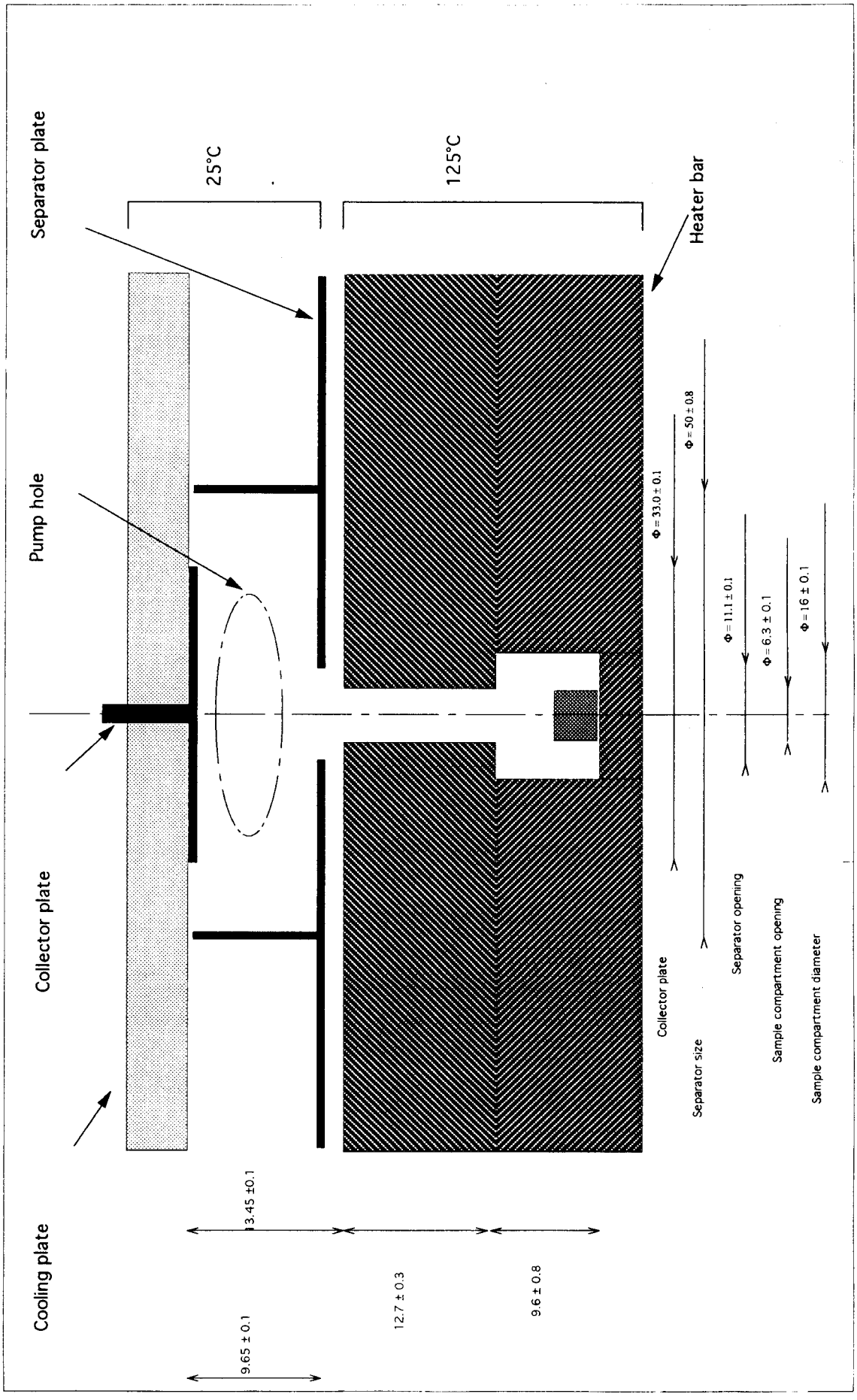

FIGURE 1 - MICRO VCM EQUIPMENT

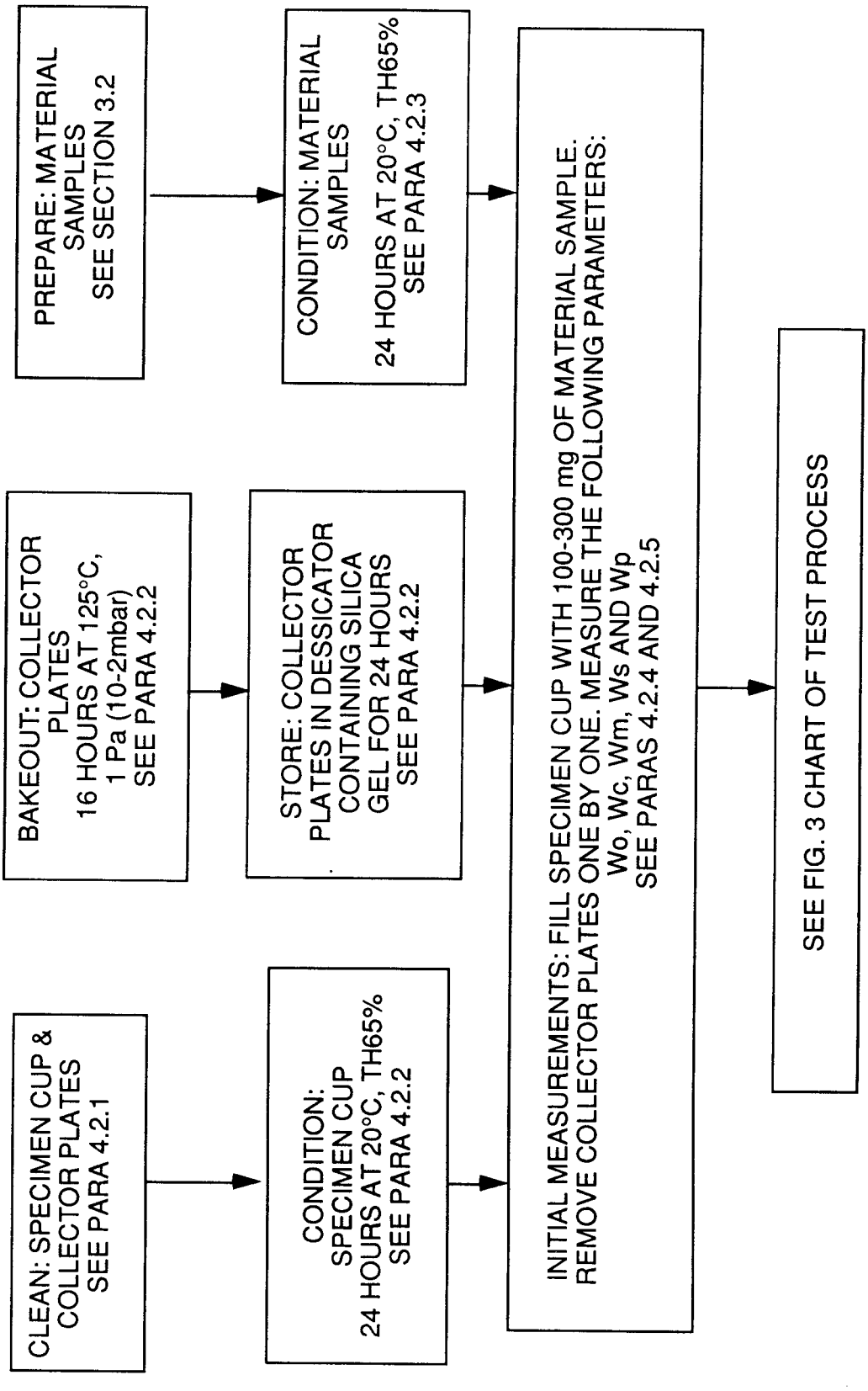

FIGURE 2 - FLOW CHART OF PREPARATION AND INITIAL MEASUREMENTS

SECTION 4. TEST PROCEDURE

4.1 INTRODUCTION

Figures 2 and 3 are included as a guide to the test processes. The sequence for the test is given in Section 4.2.

4.2 TEST PROCESS FOR GENERAL SPACECRAFT APPLICATION

4.2.1 Cleaning of cups and collector plates

Specimen cups and collector plates shall be cleaned with solvent.

4.2.2 Conditioning of cups and collector plates

The specimen cups shall be conditioned for at least 24 hours in an environment of 20°C and 65% RH. Before the collector plates are stored for 24 hours in a dessicator containing silicagel, they shall undergo a bakeout of contamination attributes for at least 16 hours in a vacuum oven at a pressure of 1 Pa (10^{-2} mbar) and at a minimum temperature of 125°C Normal practice during a test shall be to expose at the same time three specimens of each material, three empty specimen cups and three collector plates.The three collector plates facing the empty cups are to 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.

4.2.3 Conditioning of samples

Material specimens shall be prepared in the manner laid down in Section 3.2 and shall be conditioned for at least 24 hours in a 20°C/65% RH environment.

4.2.4 Weighing of samples

The pre-weighed specimen cups shall be filled with 100 to 300 mg of specimen (substrate not included) and weighing shall be performed on a microbalance (accurate to $\pm$ 1 x 10^{-6} g) located in a room conditioned at 20°C/65% RH just before the loading of the test system.

4.2.5 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 dessicator one by one.

4.2.6 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 per material.

4.2.7 Pump-down and heating

The pump-down of the test system shall be carried out as standard procedure; at 10^{-3} Pa (10^{-5} mbar) 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 hours following the instant at which the heater bar(s) reach(es) 125°C.

4.2.8 End of test

After 24-hour exposure, the heaters shall be switched off and the system shall be vented up to 1 x 10^4 to 2 x 10^4 Pa (100-200 mbar) with dry nitrogen or rare gas; cooling shall be continued until the end of the test.

4.2.9 Gas inlet

When the temperature of the heater bar(s) has fallen to 50°C (which shall take 1 1/2 to 2 hours), dry nitrogen or rare gas shall be admitted up to atmospheric pressure.

4.2.10. Unloading of system

The system shall be unloaded as soon as possible; the specimen cups shall be kept in a dessicator for not more than 1/2 hour and the collector plates for about 1 hour.The specimen cups and collector plates shall be weighed, being taken from the dessicator for this purpose one by one and returned immediately thereafter.

4.2.11 Storage of collector plates

The specimens shall then be stored in a room conditioned to 20°C and 65% RH for 24 hours, and then reweighed.

4.2.12 Infrared analyses

The infrared-transparent collector plates shall be examined with the aid of an infrared spectrometer of suitable sensitivity so that an IR spectrum of the condensed contaminants may be obtained.

4.2.13 Cleaning of system

After each test, the heater bar(s), condensor plate(s) and screen(s) of the equipment shall be thoroughly cleaned with a suitable volatile solvent, and baking shall be performed if the blank collectors indicate a mass increase >30 μg during the previous test.

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

0. PREPARE SPECIMEN CUP, MATERIAL SPECIMEN AND COLLECTOR PLATES
SEE PARAS 4.2.1 through 4.2.5 and SECTION 3.2 (see Figure 2)

1. LOAD SYSTEM:
BRING TO 125°C AT 10^{-3} Pa (~10^{-5} mbar) WITHIN 1 HOUR
SEE PARAS 4.2.6 AND 4.2.7

2. MAINTAIN SYSTEM:
AT 125°C ≤ 10^{-3} Pa (≤ 10^{-5} mbar) FOR 24 HOURS
SEE PARA. 4.2.7

3. SWITCH OFF HEATER:
AND VENT SYSTEM TO 10^4 TO $2x10^4$ Pa (100-200 mbar) WITH DRY NITROGEN OR RARE GAS, SEE PARA. 4.2.8

4. COOL SYSTEM:
TO 50°C AND ADMIT DRY NITROGEN OR RARE GAS UNTIL ATMOSPHERIC PRESSURE IS OBTAINED. SEE PARA. 4.2.9

5. UNLOAD SYSTEM:
STORE IN DESSICATOR, MAX. OF 1/2 HOUR SPECIMEN CUP AND 1 HOUR COLLECTOR PLATES. MEASURE W_f AND W_g
SEE PARA. 4.2.10

6. CONDITION:
FOR 24 HOURS AT 20°C, RH 65%. SEE PARA. 4.2.11

7. FINAL MEASUREMENTS:
W_r, INFRARED INSPECTION (IF APPLICABLE)
SEE PARAS 4.2.11 AND 4.2.12

FIGURE 3 - FLOW CHART OF TEST PROCESS

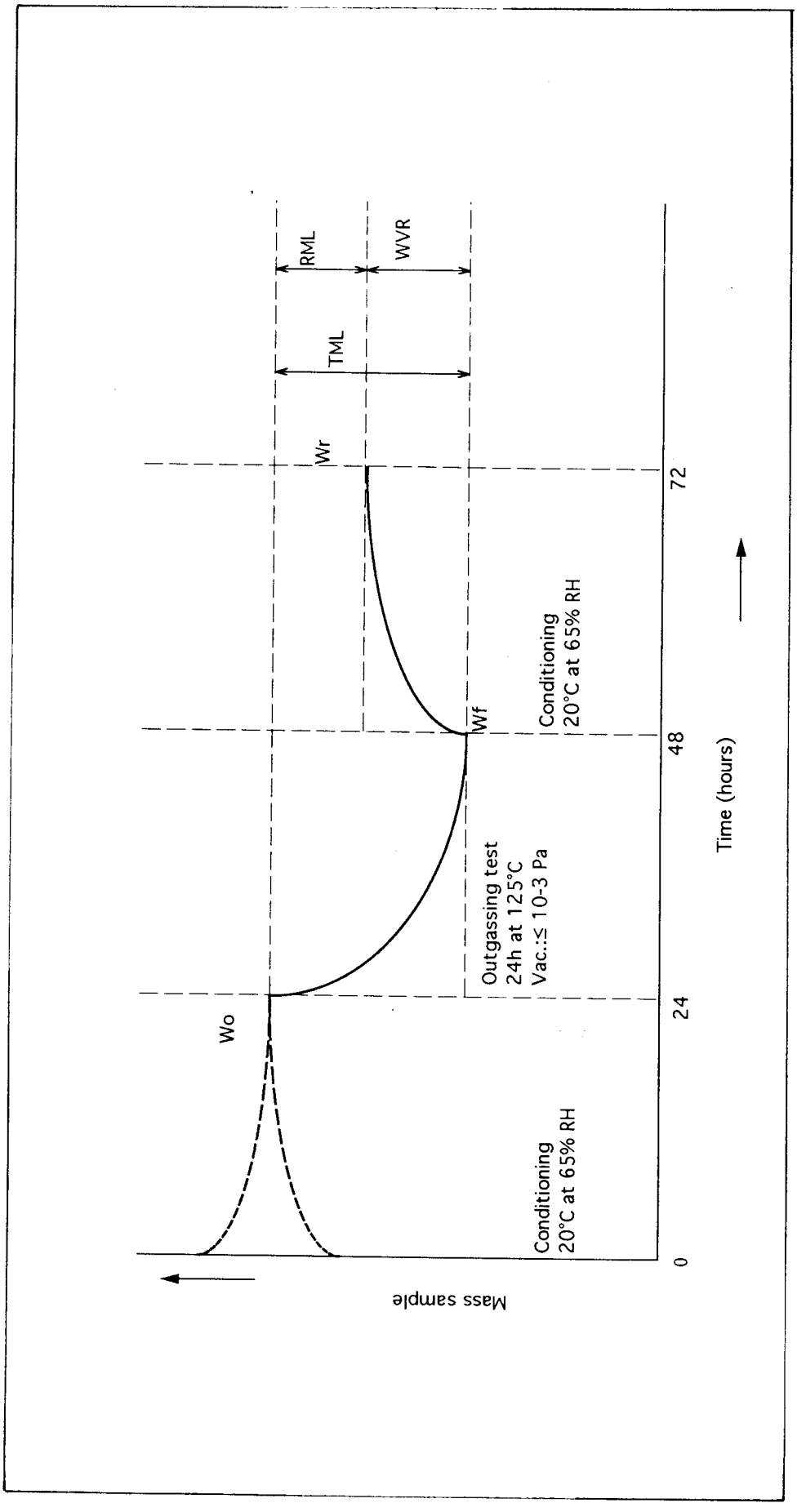

FIGURE 4 - PARAMETERS FOR SAMPLE

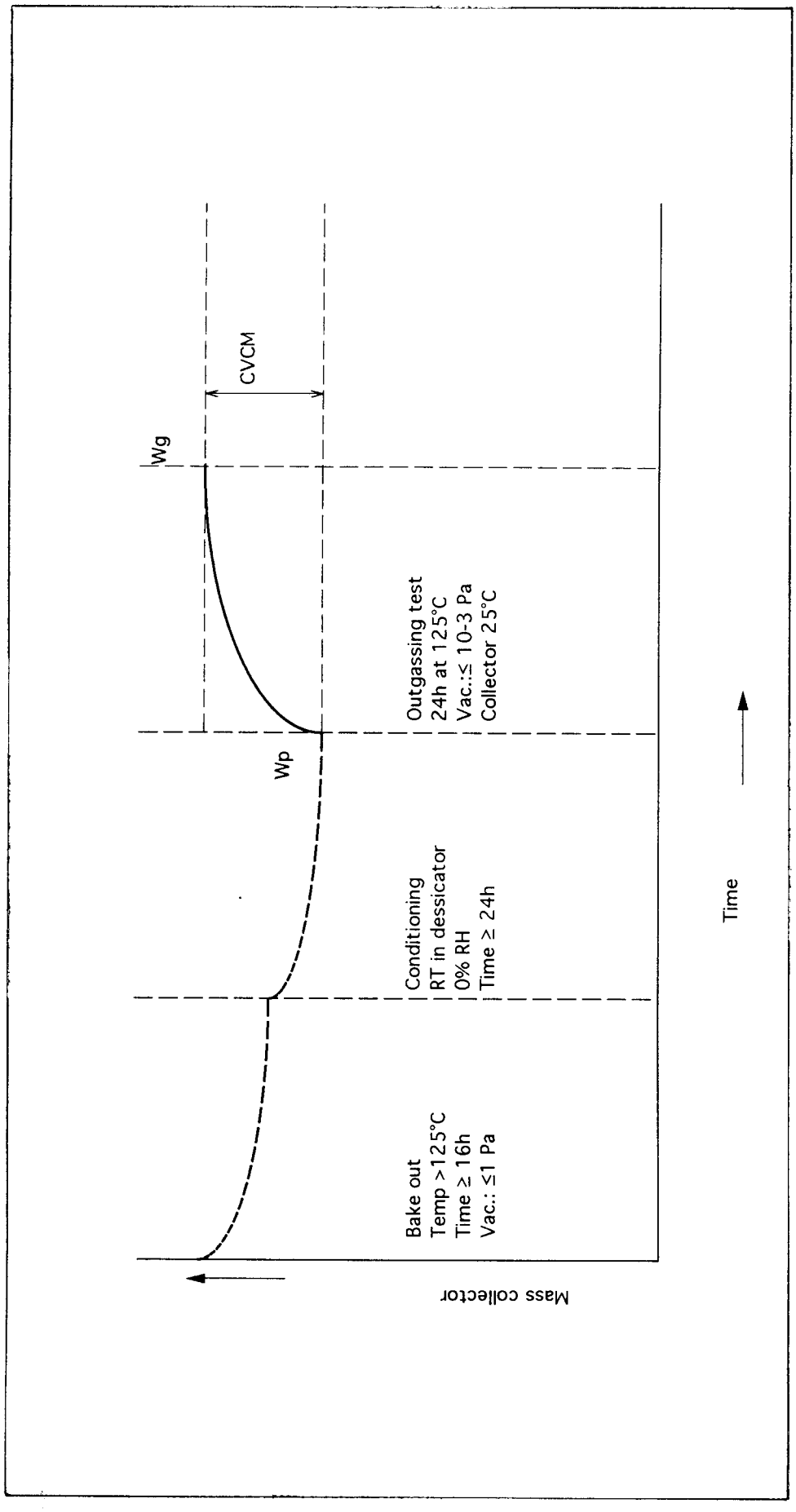

FIGURE 5 - PARAMETERS FOR COLLECTOR PLATE

SECTION 5. ACCEPTANCE LIMITS

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 may 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 foreseen for the hardware. Also, infrared inspection may be invoked if considered necessary (see Para. 4.2.12).

(a) TML <1.0 %, CVCM <0.10%

(b) In cases where water absorption is proven to be acceptable, the acceptance criteria for material outgassing can be based upon the following:

Water absorbed by materials is included in the measured TML, and the TML data for water-absorbing materials such as polyamides, polyimides and polyurethanes, is often above the acceptable limit of 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 earlier takes water-reabsorption into account, as the RML is also measured. (The RML is basically the TML value that does not include reabsorbed water.)

Under the following conditions an acceptance limit of 1.0% RML can be accepted and a waiver can be granted for not meeting the 1.0% TML:

- 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 purging controls the water-reabsorption during ground life up to launch.

(c) Material variance. The maximum values for "one standard deviation" (S) with respect to the "mean" values $\bar{x}$ 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%

(d) The limits defined under (a) and (b) above can be made more stringent if the materials concerned are to be used in 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 will remain uncontaminated. Consequently, subsequent functional, development and qualification tests must be used where appropriate to ensure that the material's performance continues to be satisfactory.

TERM	CALCULATIONS	REMARKS
Total mass loss % (TML %)	$\frac{Wo-Wf}{Wm} \times 100$	Wm = Wo - Wc - Ws
Collected volatile condensable material % (CVCM %)	$\frac{Wg-Wp}{Wm} \times 100$	
Recovered mass loss % (RML %)	$\frac{Wo-Wr}{Wm} \times 100$	Wr is measured on completion of post conditioning
Water vapour regained % (WVR %)	$\frac{Wr-Wf}{Wm} \times 100$	

NOTE: The definitions of the various masses are given in paragraph 6.1 (h)

FIGURE 6 - DEDUCED OUTGASSING PROPERTIES

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SECTION 6. QUALITY ASSURANCE

The quality assurance requirements are defined in ESA PSS-01-20. However, particular attention should be given to the following points.

6.1 DATA

The following properties shall be recorded in the test report:

(a) Specific mass of the finished product (per cm^3 for bulk solid, per cm^2 for coatings and thin layers, per cm for wires and threads).

(b) Density of substrate in g/cm^2 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 should be marked with their wavelength value.

(f) Any noticeable incident observed during the test should be recorded .

(g) Results of the blank test audit (see Section 6.5).

(h) The masses of specimens and collectors before and after the test:

W_o = total specimen mass (material+cup+substrate) before the test.

W_c = mass of specimen cup.

W_s = mass of substrate, determined by weighing or by calculation from density and surface area.

W_m = mass of material before test = $W_o - W_c - W_s$.

W_f = total specimen mass just after test.

W_r = total specimen mass after test and after 24 hours final conditioning at 20°C/65% RH environment.

W_p = initial mass of collector plates before test.

W_g = final mass of collector plates after test.

(i) The deduced outgassing properties (see Figure 6, which lists the calculations needed to establish these values).

(j) Details of failure mode (if applicable).

(k) A proper identification of the material as stated in Paragraph 3.2.4.

6.2 NONCONFORMANCE

Any nonconformance which is observed in respect of the process or the audit shall be dispositioned in accordance with the quality assurance requirements. However, retesting is allowed if material fails on the deviation limits (Paragraph 5(c) refers).

6.3 CALIBRATION

Each standard and piece of measuring equipment shall be calibrated. Any suspected or actual equipment failure must be notified to ESA so that previous results may be examined to ascertain whether or not re-inspection/re-testing is needed.

6.4 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.

6.5 AUDIT OF THE MICRO-VCM TEST APPARATUS

The main purpose of this audit is to ensure the validity of test results by comparison with those obtained by other laboratories/test houses for ESA projects (see also ESA PSS-01-711). The material-outgassing data obtained in the manner laid down in ESA PSS-01-702 are only accepted for ESA projects if the test house is certified to perform these tests. The audit shall consist of:

(a) A blank test, identical to the normal VCM test described in this document.

(b) An actual test, in accordance with this document. In parallel, ESTEC will perform an identical test and results shall be compared. The actual tests will be done as round robin tests between different test houses and the certification criteria are laid down in ESA PSS-01-711.

(c) A physical inspection of the complete apparatus including facilities and measuring equipment.

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ANNEX A
ABBREVIATIONS

ABBREVIATIONS:

CVCM - Collected Volatile Condensable Material

RML - Recovered Mass Loss

TML - Total Mass Loss

WVR - Water Vapour Regained

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ANNEX B
MATERIAL IDENTIFICATION CARD

MATERIAL IDENTIFICATION CARD - QM - ESTEC - NOORDWIJK - THE NETHERLANDS

DESCRIPTION AND HISTORY OF SAMPLE A) TRADE NAME + NUMBER B) MANUFACTURER C) TYPE OF PRODUCT D) CHEMICAL NATURE E) PROCESSING DETAILS: e.g. - joining method - heat treatment - cure + postcure - cleaning method - relevant spec. no.	a) c) e)	b) d)
BATCH NUMBER SAMPLE QUANTITY PREPARATION DATE PREPARED BY		DATA USEFUL FOR REQUIRED TEST e.g.: - material density - substrate density - substrate material
CONTRACTOR/EXPERIMENTER SAMPLE CODE (REFER TO THE DML ITEM NUMBER OF THE PROJECT)		PROJECT/COST CODE ESTEC P.A. MANAGER OR ORGINATOR NAME &SIGNATURE
APPLICATION		
REQUIRED TEST SPEC. NO.		QUALITY CONTROL SAMPLE OR EVALUATION SAMPLE
FOR MATERIALS & PROCESSES DIVISION USE DATE RECEIVED : RESPONSIBLE SECTION : TRACEABILITY CODE :	TEST DATE : TEST NUMBER :	REPORT NUMBER : ISSUE DATE : RESULTS : ACCEPT REJECT