ESA PSS-01-705 Issue 1

European Space Agency

PRODUCT ASSURANCE DIVISION, ESTEC

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October 1982
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ABSTRACT

This specification details the test procedures for the detection of organic contamination of surfaces by direct and indirect means with the aid of infrared spectroscopy.

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TABLE OF CONTENTS

TABLE OF CONTENTS (CONT'D)

THE DETECTION OF ORGANIC CONTAMINATION OF SURFACES BY INFRARED SPECTROSCOPY

1. SCOPE

This specification details the test procedures for the detection of organic contamination of surfaces by direct and indirect means with the aid of infrared spectroscopy.

2. GENERAL

2.1 INTRODUCTION

2.1.1

Spacecraft materials and hardware or vacuum chambers may be contaminated by one or all of the following organic substances:

(a) Volatile condensable products of materials outgassing under vacuum;

(b) Backstreaming products from pumping systems;

(c) Handling residues (e.g. human grease);

(d) Residue of cleaning agents;

(e) Creep of certain substances (e.g. silicones).

2.1.2

Infrared (IR) spectroscopy monitoring will verify that the stringent contamination and cleanliness controls applied to ESA Spacecraft materials and associated equipment have been met. Moreover, the source of any contamination can be detected and the resolution is higher than most other techniques:

(a) **Direct Methods.** (see para 4.1). IR-transparent discs are placed in situ, i.e. inside vacuum chamber, clean room or spacecraft etc. Contamination of the discs is then analysed (without further treatment) by means of an IR spectrophotometer.

(b) **Indirect Methods** (see para 4.2). The contaminants on the surface to be tested are collected by means of washing or wiping. This surface can also be a witness plate which is removed after exposure and treated according to 4.2.1. The resultant contaminated liquid/tissue is then processed and finally a disc containing the contaminants is analysed with the aid of an IR spectrophotometer.

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 specifications is defined in the contract.

PSS-01-20 Quality Assurance of ESA Spacecraft and Associated Equipment

PSS-01-70 Material and Process Selection and Quality Control for ESA Spacecraft and Associated Equipment

PSS-01-702 A Thermal Vacuum Test for the Screening of Space Materials

2.3 DEFINITIONS

The definitions listed in Annex A shall apply.

3. PREPARATORY CONDITIONS

3.1 HAZARDS/SAFETY PRECAUTIONS

Particular attention should be made to health and safety precautions listed below (only $CHCl_3$ is used):

(a) unavoidable hazards to personnel equipment and materials must be controlled and kept to a minimum;

(b) hazardous substances, items and operations must be isolated from other activities;

(c) items and controls must be so located that personnel are not exposed to hazards such as electric shocks, cutting edges, sharp points, or toxic atmospheres;

(d) suitable warning and caution notes must be provided in operations, storage, transport, testing, assembly, maintenance and repair instructions and distinctive markings on hazardous items, equipment or facilities for personnel protection.

3.2 MATERIALS

Materials used in the process shall be stored in a cleanliness-controlled area, ambient temperature of 20 ± 3°C and relative humidity of 55 ± 10%. 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.

3.3 HANDLING

Samples shall only be handled with clean nylon or lint-free gloves.

3.4 FACILITIES

3.4.1 Cleanliness

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

3.4.2 Environmental Conditions

Unless otherwise stated, the ambient condition for the test process and work areas shall be 22 ± 3°C with a relative humidity of 55 ± 10%.

3.5 EQUIPMENT

3.5.1 Test Equipment

Suitable measuring equipment shall be available to fulfil the monitoring requirements of the processes (temperature 15-30°C, humidity RH 40-70%).

3.5.2 Special Apparatus

(a) **Infrared Spectrophotometer.** Preferably an infrared spectrophotometer with a wavelength range of 2.5 to 15 micrometres, double-beam and scale expansion ranges should be used. Furthermore, plates of infrared-transparent material, such as sodium chloride, should be available. For direct analysis of metal foils, an ATR-attachment to the spectrophotometer is required.

(b) **Miscellaneous.** For obtaining and preparing samples, the following items are needed:
- standard filter paper 70 mm Ø,
- piece of pre-cleaned foam rubber, approximately 50 x 30 mm
- clean nylon gloves,
- spectral-grade solvent,

- petri dishes approximately 70 mm Ø,
- glass rod,
- glass syringe,
- tweezers,
- infrared lamp,
- chloroform spectral grade - WARNING TOXIC (see para 3.1).

4. TEST PROCEDURE (SEE CHART 1)

Two methods of contamination measurement based on infrared spectroscopy techniques are detailed (see para 2.1.2)

4.1 DIRECT METHOD

4.1.1 Infrared-transparent discs shall be positioned at or near critical places inside the compartment, chamber or room etc. to be monitored.

4.1.2 Upon completion of exposure, the infrared-transparent discs should be analysed with the IR spectrophotometer as soon as possible. Otherwise creep of certain kinds of contamination agents (e.g. silicones) may cause false results.

4.1.3 Interpretation of the infrared spectra obtained is given in Section 5.

4.2 INDIRECT METHOD

4.2.1 Introduction

If possible the surface to be analysed (which can be a witness plate) shall be washed with a known quantity of spectral-grade solvent which is collected in a Petri dish (approx. 70mm Ø) and processed in accordance with paragraph 4.2.2. However, washing is not usually possible and the surface then has to be wiped in accordance with paragraph 4.2.3.

4.2.2 Washing Process

(a) The Petri dish containing the contaminated solvent is placed in a slightly tilted position under an infrared lamp (in order to evaporate the solvent) until a few droplets remain.

The droplets are then transferred to a clean IR-transparent sodium chloride disc by means of a clean glass rod and positioned on the disc in an area corresponding to the beam shape of the IR spectrophotometer. (For the IR-12 instrument, this area is 3mm x 16mm, $0.5cm^2$.)
The disc is placed under the IR lamp, this causes the solvent to evaporate and a thin film of contaminants is left on the disc.

(b) The process can be repeated, if necessary, provided that the Petri dish is washed with a small amount of chloroform.

(c) Finally, the disc is fitted to the IR spectrophotometer and so aligned that the light beam of the IR spectrophotometer covers the contaminated area of the disc. In practice the disc is adjusted until maximum absorption is obtained in the 2925 cm^{-1} band.

(d) The infrared spectra are recorded and interpreted as explained in Section 5.

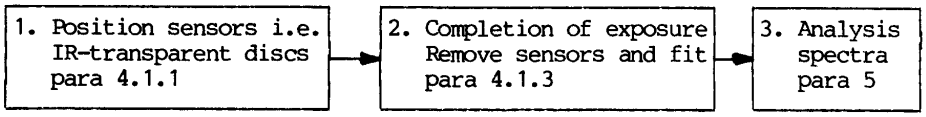
4.2.3 Wiping Process

(a) The lens tissue should be pre-cleaned by several immersions (each lasting at least one hour) in fresh solvent followed by a blank analysis performed to 4.2.3(b) and (c) and 4.2.2 until a background level of $5x10^{-7}$ g is obtained (normally 3 to 4 immersions are sufficient). The tissue paper should then be stored in a clean glass bottle.

(b) The surface to be analysed is wiped 8 times (twice in each of four directions) with a foam-rubber tube (50 x 33 mm dia) covered with standard filter paper (70 mm Ø) and pre-cleaned lens tissue (see para 4.2.3(a)). During wiping, clean nylon gloves shall be worn and ideally the filter paper with tissue attached should be turned a little after each wipe.

(c) The contaminated tissue is immersed for 10 to 15 min. in a known quantity of spectral-grade solvent contained within a Petri dish (70 mm Ø). The tissue is then taken with tweezers and rinsed with 0.2 cm^3 of solvent. The Petri dish containing the contaminated solvent is further processed in accordance with paragraph 4.2.2.

DIRECT METHOD



INDIRECT METHOD

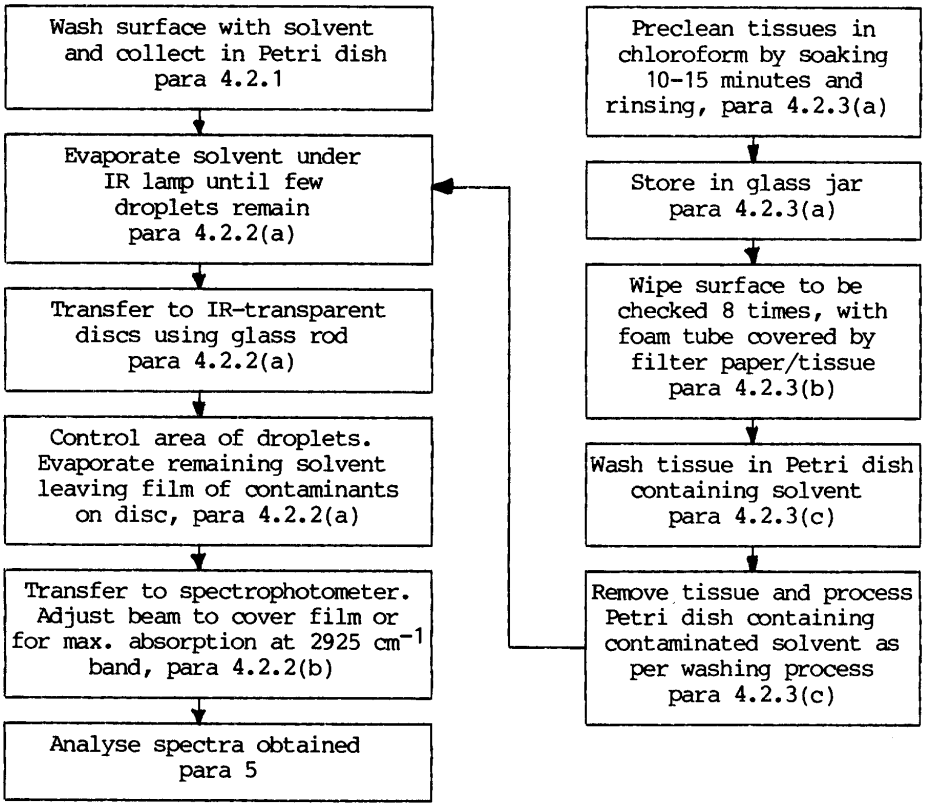


CHART 1

TEST PROCEDURE FLOW DIAGRAM

5. INTERPRETATION OF INFRARED SPECTRA

(a) Examination of the absorption bands enables the types of contamination to be determined. Contamination checks on spacecraft and in vacuum chambers usually indicate mixtures of several contaminants, and this makes it more difficult to find the type and origin of the contamination. The "Micro-VCM" materials screening method provides infrared spectra of the volatile condensable products released from the materials tested and these can be used as standards in contamination monitoring tests.

Experience with hundreds of analyses has indicated that the contaminants can be divided into four main groups:-

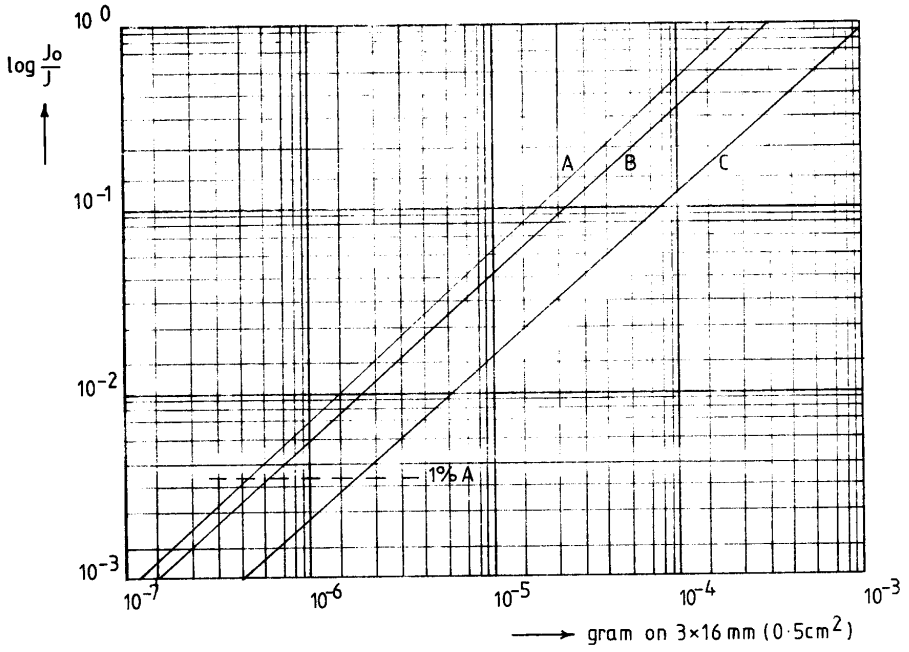
Hydrocarbons	with main bands at **2925** - 1465 - 1375 cm^{-1}
Esters	with main bands at **1735** - 1070 - 1120 - 1260 cm^{-1}
Methyl silicones	with main bands at **805** - 1020 - 1080 - 1260 cm^{-1}
Phenyl silicones	with main bands at **790** - 1050 - 1120 - 1260 - 1430 cm^{-1}

The ester band at about 1735 cm^{-1} and the confirmatory bands between 1100 and 1300 cm^{-1} may give an indication as to the type of ester (aryl/alkyl ester of aromatic/aliphatic acid). Typical for a phthalate ester (mostly used as a plasticiser) is the doublet at 1580/1600 cm^{-1} with intensities of about 1/11 of the 1735 cm^{-1} band. Typical for human grease is the ester/acid doublet 1735/1710 cm^{-1}.

(b) Quantitative interpretation of the infrared spectra is not always simple. Nominally, the exact type of contamination is unknown, and insufficient material is available to make a calibration curve.

From pure materials (e.g. pump oils) calibration curves must be made (see Figure 1). Quantitative information can also be obtained from the "Micro-VCM" infrared spectra as the weight of the contamination is known down to about 10 micrograms.

Methyl and phenyl silicones give different infrared spectra, but both have bands at about 805 cm^{-1}. From the ratio 1:1.28 for the bands at 1430 cm^{-1} and 790 cm^{-1} the contribution of the phenyl silicones to the 805 cm^{-1} band can be calculated. Methyl and phenyl silicones generally do not have a band at 2925 cm^{-1} or at 1735 cm^{-1}.
Most esters have a band at 2925 cm^{-1} (hydrogen chain) as well as those bands mentioned above. For calculations, however, it is simpler to see the hydrocarbon chain of the ester as hydrocarbons.
For the calculations of the contamination levels one must use the attached calibration curves (see Figure 1). Of course, if a new contaminant is encountered, one has to make a calibration curve for it (if sufficient material is available). Moreover, a different spectrophotometer or attachment requires new calibration curves.



Curve A: Methyl silicones 805 cm^{-1}
Curve B: Hydrocarbons 2925 cm^{-1}
Curve B: Esters 1735 cm^{-1}
Curve C: Phenyl silicones 790 cm^{-1}

FIGURE 1

CALIBRATION CURVES

(IR-12 SPECTROPHOTOMETER)

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 logbooks shall contain, as a minimum, the following:

(a) copy of final inspection documentation;

(b) index of limited-life articles and their use times;

(c) nonconformance reports and corrective actions;

(d) copy of the inspection and test results with reference to the relevant procedure and methods used for obtaining samples;

(e) the contamination levels of each substance in g cm^{-2} including their relative percentage compared with total contamination levels;

(f) details of failure mode (if applicable).

6.2 NONCONFORMANCE

Any nonconformance which is observed in respect of the process shall be dispositioned in accordance with the quality assurance requirements.

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 from incoming inspection to final test, including details of test equipment serial/batch numbers and personnel employed in performing the task.

ANNEX A

DEFINITIONS

NONCONFORMANCE
An apparent or proven condition of any item or documentation that does not conform to specified requirements or which could lead to incorrect operation or performance of the item or mission.
The term nonconformance is also used for failure, discrepancy, defect, anomaly, malfunction, deficiency, etc.

TRACEABILITY
The ability to trace the history, application, use and location of an item through the use of recorded identification numbers.