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Space product assurance

Detection of organic contamination of surfaces by infrared spectroscopy

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Foreword

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

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

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

This Standard has been prepared by editing ESA PSS-01-705, reviewed by the ECSS Product Assurance Panel and approved by the ECSS Steering Board.

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Introduction

Spacecraft materials and hardware, or vacuum chambers can be contaminated by one or more of the following organic substances:

- Volatile condensable products of materials outgassing under vacuum.
- Volatile condensable products of off-gassing materials.
- Backstreaming products from pumping systems.
- Handling residues (e.g. human grease).
- Residues of cleaning agents.
- Non-filtered external pollution.
- Creep of certain substances (e.g. silicones).

There are several methods for identifying organic species, such as mass spectrometry, gas chromatography and infrared spectroscopy, or a combination of these methods. Infrared spectroscopy is the most widely used technique: It is a simple, versatile and rapid technique providing high resolution qualitative and quantitative analyses.

- Infrared qualitative analysis is carried out by functional group identification, or by comparison of the IR absorption spectra of unknown materials with those of known reference materials, or both. It is therefore possible to determine structural information about the molecules of contaminants. In some cases, the source of the contamination can be detected.
- Infrared quantitative analysis of levels of contaminants is based on the Lambert-Beer's (henceforth referred to as Beer's) law and requires calibration.

Infrared spectroscopy monitoring is used to verify that the stringent contamination and cleanliness controls applied to spacecraft materials and associated equipment are met. Different methods for measuring contamination are described:

- Direct methods

  IR-transparent windows used as witness plates (e.g. CaF_2, ZnSe, Ge) are placed in situ, for example, inside a vacuum facility, cleanroom or spacecraft. Contamination of the windows is then analysed (without further treatment) using an IR spectrophotometer.

- Indirect methods

  The contaminants on the surface to be tested are collected by means of a concentration technique, for example by washing or wiping a larger surface. Such a surface can also be a witness plate, which is removed after exposure and treated in the same way. The resultant contaminated liquid or tissue is then processed, and finally an IR-transparent or a reflective window containing the contaminants is analysed with the aid of an IR spectrophotometer.

  NOTE Whenever possible, the direct method is preferred.

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Scope

This Standard defines the test procedures for detecting organic contamination on surfaces using direct and indirect methods with the aid of infrared spectroscopy. The following test methods are described:

- Direct sampling of contaminants.
- Indirect sampling of contaminants by washing and wiping.

This Standard also provides further guidance on interpreting the spectral data obtained through:

- Qualitative interpretation of the spectral results.
- Quantitative interpretation of the spectral results.

The test methods described in this Standard apply to controlling and detecting organic contamination on all manned and unmanned spacecraft, launchers, payloads, experiments, terrestrial vacuum test facilities, and cleanrooms.

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Normative references

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

ECSS-P-001	Glossary of terms
ECSS-Q-20	Space product assurance — Quality assurance
ECSS-Q-20-09	Space product assurance — Nonconformance control system
ECSS-Q-70-01	Space product assurance — Contamination and cleanliness control
ECSS-Q-70-02	Space product assurance — Thermal vacuum outgassing test for the screening of space materials

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Terms and definitions

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ECSS-P-001 and the following apply.

3.1.1
absorbance
A
logarithm to the base 10 of the reciprocal of the transmittance, *T*

[ASTM-E-131]

NOTE The term absorbance is also widely used for the negative log of the ratio of the final to the incident intensities of processes other than transmission, such as attenuated total reflection and diffuse reflection.

3.1.2
absorption
transfer of infrared energy to the molecules present within the pathway of the radiation

3.1.3
absorptivity
absorbance *A* divided by the product of the concentration *C* of the substance and the sample path length *l*

NOTE 1 Absorptivity = A/(l C). The recommended unit for *l* is cm. The recommended unit for *C* is kg m^{-3}.

NOTE 2 The equivalent IUPAC term is "specific absorption coefficient".

[adapted from ASTM-E-131]

3.1.4
attenuated total reflection
reflection that occurs when an absorbing coupling mechanism acts in the process of total internal reflection to make the reflectance less than unity

[ASTM-E-131]

3.1.5
diffuse reflection
reflection in which the flux is scattered in many directions by diffusion at or below the surfaces

[ASTM-E-131]

3.1.6
Fourier transformation
mathematical process used to convert an amplitude-time spectrum to an amplitude-frequency spectrum or vice versa

[ASTM-E-131]

3.1.7
infrared spectroscopy
spectroscopy in the infrared region of the electromagnetic spectrum, i.e. with wavelength range from approximately 0,78 μm to 1 000 μm (wave number range 12 820 cm^{-1} to 10 cm^{-1})

[adapted from ASTM-E-131]

3.1.8
molar absorptivity
ε
product of the absorptivity and the molecular weight of the substance

NOTE The equivalent IUPAC term is "molar absorption coefficient".

[adapted from ASTM-E-131]

3.1.9
radiant power
P
amount of energy transmitted in the form of electromagnetic radiation per unit time

NOTE 1 Unit for radiant power is Watts

NOTE 2 Radiant power should not be confused with intensity (*I*), which is the radiant energy emitted within a time period per unit solid angle (measured in Watts per steradian).

3.1.10
reflectance
R
ratio of the radiant power reflected by the sample to the radiant power incident on the sample

[ASTM-E-131]

3.1.11
transmittance
T
ratio of the radiant power transmitted by the sample to the radiant power incident on the sample

[ASTM-E-131]

3.1.12
wave number
$\overline{\nu}$
number of waves per unit length

NOTE 1 The unit for wave number is cm^{-1}. In terms of this unit, the wave number is the reciprocal of the wavelength, λ (where l is expressed in cm).

NOTE 2 The wave number is normally used as the X-axis unit of an IR spectrum.

[adapted from ASTM-E-131]

3.2 Abbreviated terms

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

Abbreviation	Meaning
ASTM	American Society for Testing and Materials
ATR	attenuated total reflection
AU	absorbance unit
C	concentration
DOP	dioctylphthalate, synonym bis(2-ethylhexyl) phthalate
DRIFT	diffuse reflection infrared Fourier transform
DTGS	deuterated triglycine sulphate IR detector
ESD	electrostatic discharge
FTIR	Fourier transform infrared (spectrometry)
IES	Institute of Environmental Sciences
IPA	isopropyl alcohol
IR	infrared
IUPAC	International Union of Pure and Applied Chemistry
ISO	International Organization for Standardization
MCT	mercury cadmium telluride IR detector
NVR	non-volatile residue
PTFE	polytetrafluoroethylene
QCM	quartz crystal microbalance
RI	refractive index
S/N	signal to noise ratio
UV	ultraviolet
VCM	volatile condensable material

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Preparatory conditions

4.1 Hazard, health and safety precautions

The following health and safety precautions shall be taken into account:

a. Unavoidable hazards to personnel equipment and materials shall be controlled and kept to a minimum.

b. Hazardous substances, items and operations shall be isolated from other activities.

c. Items and controls shall be located so that personnel shall not be exposed to hazards such as electric shocks, cutting edges, sharp points, or toxic atmospheres.

d. Suitable warning and caution notes shall be included in the instructions for operation, storage, transport, testing, assembly, maintenance and repair. Hazardous items, equipment or facilities shall also be clearly marked to instruct personnel that they should take the necessary precautions.

e. Before starting any operation, any safety hazards shall be identified, and the necessary precautions taken to minimize risks (e.g. protection devices when chloroform is used).

f. Any operation requiring safety suits or protection devices shall only be initiated if all the personnel involved have the required protection, including any specific protection devices available at the work-place.

4.2 Facilities

4.2.1 Cleanliness

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

4.2.2 Environmental conditions

The ambient conditions for the test, process and work areas shall be (22 ± 3) °C with a relative humidity of (55 ± 10) %. Additional or other conditions can be imposed for critical operations.

4.3 Materials

Materials used in the process shall be stored in a cleanliness-controlled area as defined in subclause 4.2.1. Limited-life materials shall be labelled with their shelf lives and dates of manufacture, or delivery date if the date of manufacture is not known.

4.4 Handling

All operations shall be performed using tweezers and clean gloves (powder-free nylon, nitrile, latex, or lint-free cotton gloves). Compatibility between gloves and all chemicals used shall be evaluated.

4.5 Equipment

4.5.1 Infrared spectrophotometer

Spectral range: Typically 4 000 cm^{-1} – 600 cm^{-1} (2,5 μm – 6,6 μm).

Resolution: 4 cm^{-1}.

Sensitivity: defined by the applications and requirements.

For transmission methods, the detection limit of the IR spectrometer should have an absorbance resolution of 0,000 1. To achieve this level, adequate background measurements shall be performed. In addition, facilities should be available to reduce the interference of environmental components (such as H_2O and CO_2) in the region of interest by, for example, flushing with the proper gases or applying a vacuum.

Furthermore, plates of infrared-transparent material, such as NaCl, MgF_2, CaF_2, ZnSe, Ge, should be available.

For direct analysis of the surfaces of materials, an ATR-attachment to the spectrophotometer can be used.

It should be taken into account that the results of this technique are more dispersed and should therefore only be used for qualitative purposes.

4.5.2 Miscellaneous items

The following items shall be used for acquiring and preparing the samples:

- Pre-cleaned standard filter paper: e.g. 70 mm diameter (see C.2.3).
- Piece of pre-cleaned foam rubber, approximately 50 mm × 30 mm (see C.2.3).

  NOTE A PTFE film can be used to protect the foam rubber.
- Clean and lint-free gloves.
- Spectral grade solvents.
- Petri dishes ranging in diameter from 50 mm – 70 mm.
- Glass rod or micro-syringe.
- Glass syringe.
- Tweezers.
- Infrared lamp.

5

Procedure for sampling and analysis

5.1 General

In this Clause 5 the methods for contamination sampling for IR analysis are summarized (see also Figure 1).

5.2 Direct method

Infrared-transparent windows shall be positioned at, or near, critical locations inside, for example, the compartment, chamber, or cleanroom to be monitored. For a representative measurement, the conditions that the witness plate is subjected to are crucial and shall be as close as possible to the conditions of the location to be monitored, for example, subject to the same temperature and pressure.

Before installation, the spectrum of the cleaned, non-exposed window shall be recorded and retained as a background measurement. After exposure, the infrared-transparent windows should be analysed with the IR spectrophotometer as soon as possible, otherwise creeping of some kinds of contaminants (e.g. silicones) can cause false results.

5.3 Indirect method

5.3.1 Introduction

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 (approximately 70 mm diameter) and processed in accordance with subclause 5.3.2.

When the surface to be analysed cannot be washed, it shall be wiped in accordance with subclause 5.3.3. For details see also Annex C.

Surfaces shall only be washed or wiped with solvents that are compatible with the surface to be analysed and that do not damage the surface in any way (e.g. solvation or swelling of any material not regarded as a contaminant, or scratching of the surface).

NOTE Chloroform ($CHCl_3$) is the most widely used solvent.

5.3.2 Overview of washing process

For details of the washing process, see Annex C.

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

b. The droplets shall then be transferred to a clean, IR-transparent, window using a clean glass rod or micro-syringe and positioned on the window in an area corresponding to the beam shape of the IR spectrophotometer.
High levels of contaminants or substances of low surface tension (e.g. silicones), even at low levels, tend to concentrate in small spots. This can lead to a local saturation of the IR signal and thus subsequent underestimation of the concentration. In these cases the contaminant should be carefully distributed over the respective area (covered by the IR beam) of the IR transparent disk using the glass rod or micro-syringe.

c. The window shall then be placed under the IR lamp, which causes the solvent to evaporate and a thin film of contaminant is left on the window

d. For quantitative transfer, the process should be repeated three times after washing the Petri dish with a small amount of spectral grade solvent.

e. Finally, the window shall be fitted to the IR spectrophotometer and aligned such that the beam of the IR spectrophotometer covers the contaminated area of the window (see A.3.1).

5.3.3 Overview of wiping process

For details of the wiping process see Annex C.

a. The lens tissue shall be pre-cleaned followed by a blank analysis performed according to steps b. and c.) and 5.3.2 until a background level of less than 5×10^{-7} g for any tissue size is obtained. For example, cleaning can be performed by Soxhlet extraction or immersion in chloroform. The tissue paper should then be stored in a clean glass bottle.

b. The surface to be analysed is wiped eight times, twice in each of four directions, with either a wet or dry wipe. During wiping, clean gloves shall be worn and the filter paper, with tissue attached, should be turned a little after each wipe.

   - Wet wipe: the pre-cleaned lens tissue (see a.) is folded with tweezers until it serves as a little 'sponge'. The folded tissue is held with curved point tweezers, and wetted with spectral grade IPA or chloroform. After wiping, the solvent should be evaporated before storage in the transport container.
   - Dry wipe: a foam or rubber tube is covered with standard filter paper and pre-cleaned lens tissue (see a.).

c. The contaminated tissue is immersed for 10 minutes to 15 minutes in a known quantity of spectral grade solvent in a Petri dish (70 mm diameter). During that time, it shall be covered by a larger Petri dish in order to avoid evaporation of the solvent. The tissue is then taken with tweezers and rinsed with 0,5 cm^3 of solvent on each side. The Petri dish containing the contaminated solvent is further processed in conformance with subclause 5.3.2.

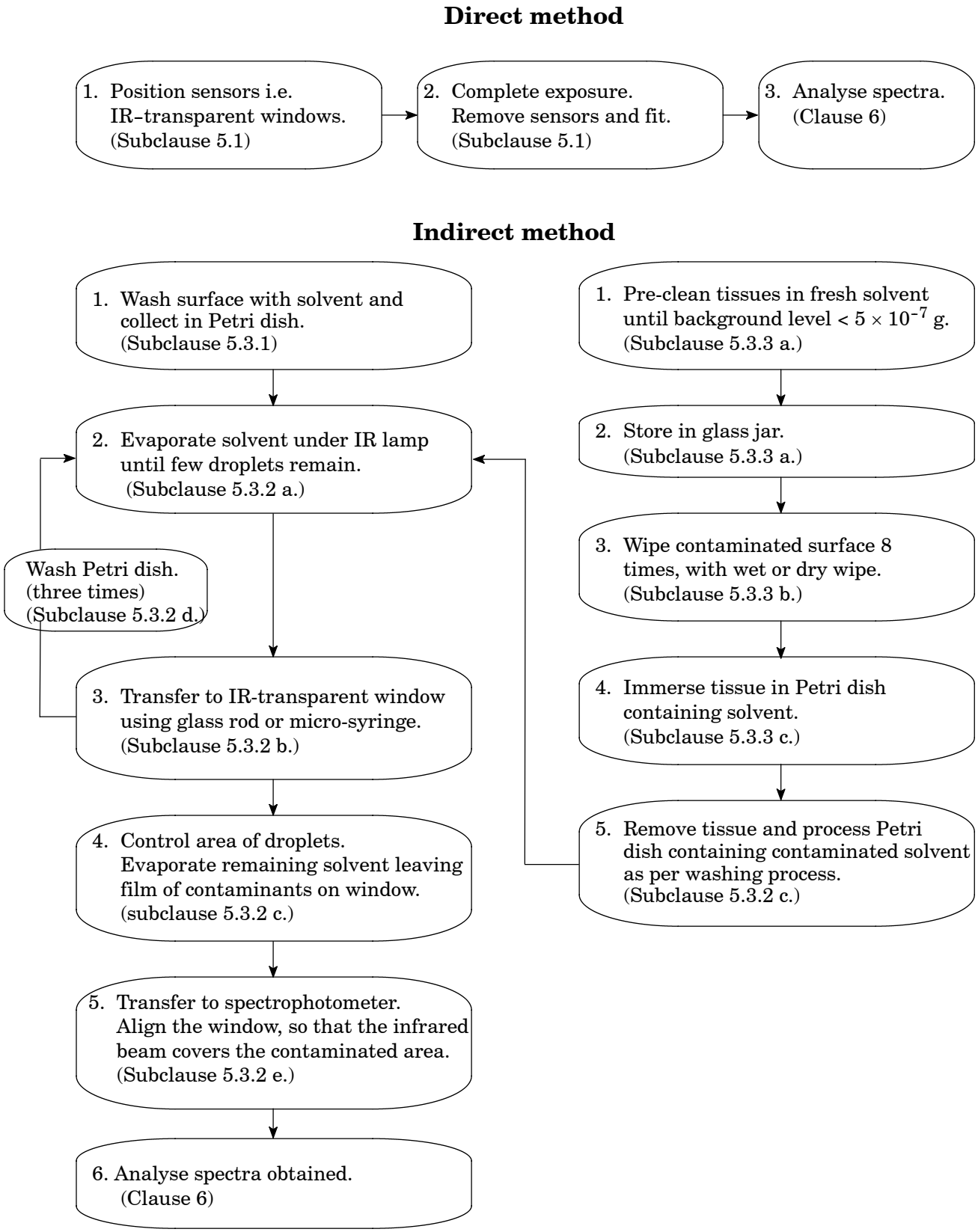

Figure 1: Sampling and analysis procedure flow chart

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Interpretation of infrared spectra

6.1 Qualitative interpretation of spectra

The different types of contamination present can be determined by examining the absorption bands of the spectra obtained from the analyses. Contamination in spacecraft and vacuum chambers commonly comprises mixtures of several contaminants. This makes it more difficult to identify the type and origin of the contamination. The "Micro-VCM" materials screening method (ECSS-Q-70-02) provides infrared spectra of the volatile condensable products released from the materials tested and these can be used as standards in contamination monitoring tests.

Past experience of numerous analyses has indicated that in general the contaminants can be divided into four main groups:

- hydrocarbons,
- esters,
- methyl silicones,
- phenyl silicones

See Figures 2 to 5 for example spectra for these four main groups. The main IR absorption bands for each group are attributed in the Table 1.

The ester band at about 1 735 cm^{-1} and the confirmatory bands between 1 300 cm^{-1} and 1 100 cm^{-1} indicate the type of ester (aryl or alkyl ester of aromatic or aliphatic acid). For a phthalate ester (mostly used as a plasticizer) the typical bands are the doublet at 1 600 cm^{-1} and 1 580 cm^{-1} with intensities of about 1:11 of the 1 735 cm^{-1} band. For human grease the ester or acid doublet at 1 735 cm^{-1} and 1 710 cm^{-1} are typical. Alkyl or aryl esters have also typical bands in the hydrocarbon region as indicated in Table 1.

Methyl and phenyl silicones have different IR spectra, but both have bands at about 805 cm^{-1}. From the ratio of the bands at 1 430 cm^{-1} and 790 cm^{-1}, the contribution of the phenyl silicones to the 805 cm^{-1} band can be calculated for defined compounds. Methyl and phenyl silicones generally do not have a band at 2 925 cm^{-1} or at 1 735 cm^{-1}.

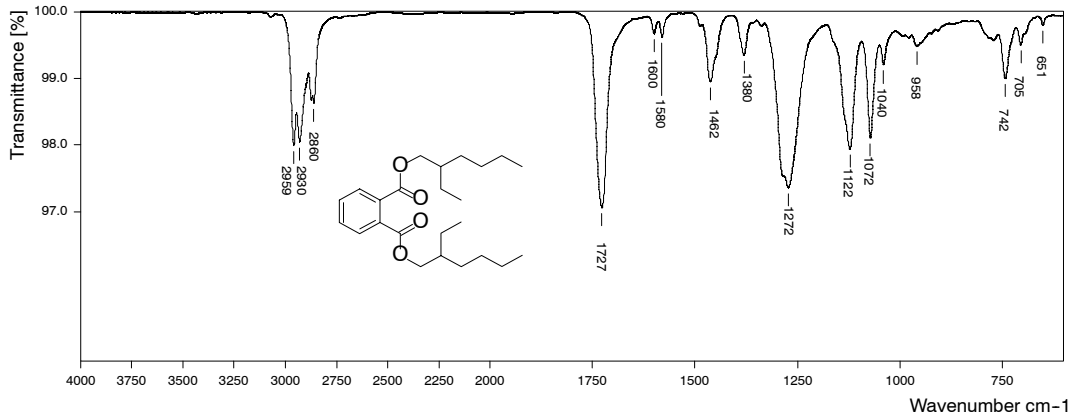

Figure 2: Characteristic spectrum of bis(2-ethylhexyl)phthalate

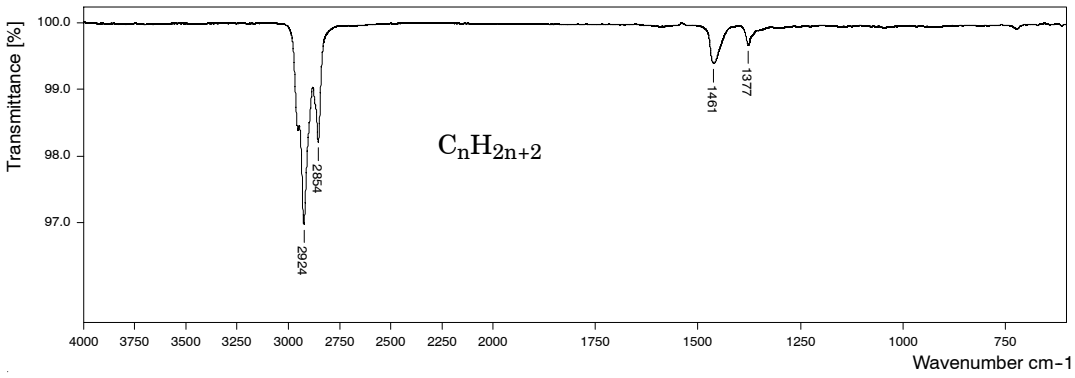

Figure 3: Characteristic spectrum of a long chain aliphatic hydrocarbon

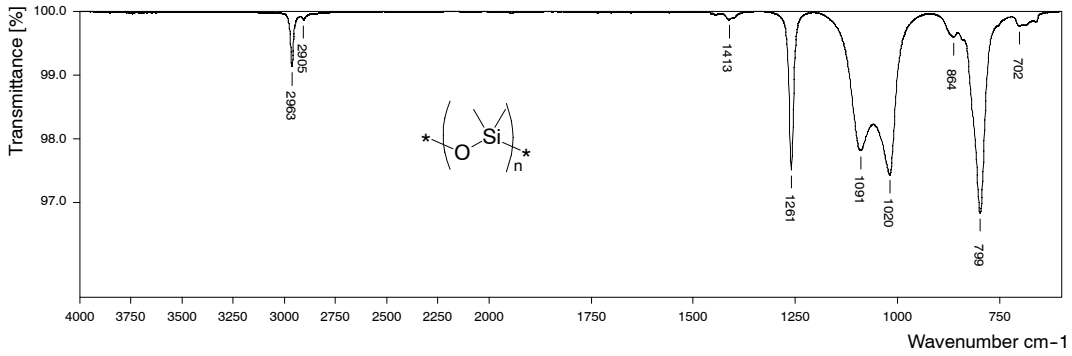

Figure 4: Characteristic spectrum of poly(dimethylsiloxane)

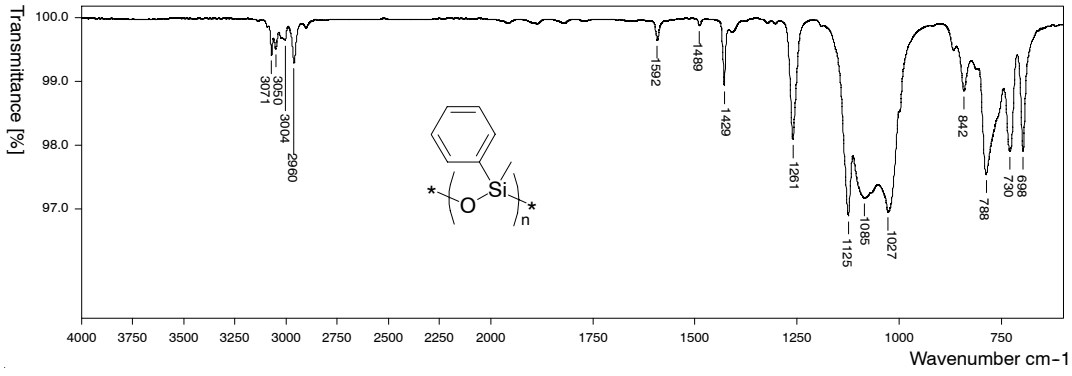

Figure 5: Characteristic spectrum of poly(methylphenylsiloxane)

Table 1: Assignment of infrared absorption bands for the four main groups of contaminants

Type of contaminant	Characteristic wave number (cm^{-1})	Functional group	Signal strength [a]	Remarks
Hydrocarbons	3 000 - 2 850	Alkanes (CH, CH_2, CH_3)	s	2 or 3 bands, stretching
	3 100 - 3 020	Alkenes	m	Stretching
	1 470 - 1 440	-CH_3	ms	Asymmetric deformation
	1 390 - 1 370	-CH_3	m	Symmetric deformation
Esters	1 750 - 1 735	C=O	s	Stretching (saturated ester)
	1 300 - 1 050	C-O	s	Stretching
Methyl silicones	1 280 - 1 255	Si-CH_3	vs	CH_3 deformation
	1 130 - 1 000	Si-O-Si	s	Asymmetric stretching
	860 - 760	Si-CH_3	vs	Si-C stretching or CH_3 rocking [b]
Methyl phenyl silicones	1 280 - 1 255	Si-CH_3	vs	CH_3 deformation
	1 130 - 1 000	Si-O-Si	s	Asymmetric stretching
	1 125 - 1 100	Si-Aryl	vs	
	860 - 760	Si-CH_3	vs	Si-C stretching or CH_3 rocking [b]

[a] Strength of signal: vs = very strong, s = strong, ms = medium to strong, m = medium.

[b] One methyl: 765 cm^{-1}; two methyls: 855 cm^{-1} and 800 cm^{-1}; three methyls: 840 cm^{-1} and 765 cm^{-1}.

6.2 Quantitative interpretation of spectra

The quantitative interpretation of IR spectra is not always simple. In some cases, the exact type of contamination is unknown, and insufficient material is available to make a calibration curve.

The quantification in infrared spectroscopy is based on the Lambert-Beer's law, in which a relationship is made between the absorbance and the concentration of a compound at a specific wavelength (equation 1).

$$Absorbance = \log\left(\frac{1}{T}\right) = \log\left(\frac{I_0}{I}\right) = \varepsilon_\lambda l C \qquad (1)$$

where:

T is the transmittance;

I_0 is the intensity of incident light;

I is the intensity of transmitted light;

ε_λ is the molar absorption coefficient at a given wavelength (l mol^{-1} cm^{-1});

l is the path length (cm);

C is the molar concentration (mol l^{-1}).

To quantify organic contamination, the absorbance is expressed as the mass of a standard material per surface area unit. The trend line (ideally linear) is calculated from the calibration points.

$$Absorbance = f(Mass) \approx Constant \times Mass \qquad (2)$$

$$Surface\ contamination = \frac{Absorbance}{Surface\ area} \qquad (3)$$

Calibration curves shall be derived from pure standard materials characteristic of the four main groups of contaminants (for examples see Figures 2 to 5). Unless the contaminant matches the calibration standard, quantification is always relative to the reference material and thus semi-quantitative.

Contamination levels shall be expressed in terms of the presence of the four main groups: hydrocarbons, esters, methyl silicones, and phenyl silicones. Calculations are performed using their characteristic group frequencies (see the detailed procedure in Annex A), whereas the peak maximum of the same vibration mode is selected as used for deriving the calibration curve.

NOTE A different chemical environment from a functional group (e.g. substitution, or neighbouring group effects) can lead to shifts in the frequency of the respective vibration modes.

The selected absorbance yields the mass of the contaminant via the calibration curve (equation 2) in units of corresponding grams of the standard material. This is subsequently expressed in terms of mass per surface area unit (equation 3) for the analysed region (see Clause 7).

If a new contaminant is encountered, it can be quantified by performing an individual calibration curve (if a standard material is available).

A different spectrophotometer or attachment requires new calibration curves.

For highly outgassing materials, quantitative information can also be obtained from the “Micro-VCM” infrared spectra since the accuracy of the weight of the contamination can be measured to about 10 μg.

6.3 Acceptance criteria

The acceptance criteria are normally defined by the customer (project). General guidelines for cleanliness and contamination control are given in ECSS-Q-70-01.

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Reporting of test data

The calibration procedure shall be outlined in terms of:

- method, type and purity of standard materials,
- frequency used for quantification,
- the correlation coefficients of the calibration curves.

The results obtained for each experiment shall be reported in terms of mass per surface area in units of g cm^{-2} for the four main groups:

- hydrocarbons,
- esters,
- methyl silicones,
- phenyl silicones.

For each of the four main groups, the mass always corresponds to the type of standard material used for obtaining the calibration curve.

The surface area is for the direct methods defined by the diameter of the IR beam diameter at the position of the sample window.

For indirect methods (Annex B and Annex C) the surface area corresponds to the surface area washed or wiped.

For contact or immersion tests (Annex D and Annex E) the surface area is defined by the size of the sample.

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8

Quality assurance

8.1 General

The quality assurance requirements are defined in ECSS-Q-20. Specific requirements are given in the subclauses 8.2 to 8.6.

8.2 Data

The records of the quality assurance (e.g. log sheets) shall be retained for at least ten years or in conformance with project contractual requirements, and include as a minimum the following:

a. Trade names and batch numbers of the materials under test.

b. Name of the manufacturer or supplier through whom the purchase was made.

c. Summary of the preparation and conditioning schedule (e.g. mixing proportions, coating thickness, cure time and temperature, post-cure, cleaning procedure).

d. Any noticeable incident observed during the measurement shall be recorded.

e. The deduced results.

8.3 Nonconformance

Any nonconformance that is observed during the measurement procedure shall be dispositioned in accordance with the quality assurance requirements; see ECSS-Q-20-09.

8.4 Calibration

a. All reference standards and measuring equipment shall be calibrated.

b. Any suspected or actual equipment failure shall be recorded in a project nonconformance report so that previous results can be examined to ascertain whether or not re-inspection and retesting is required.

c. The customer shall be notified of the details of nonconformance.

8.5 Traceability

Traceability shall be maintained throughout the process from incoming inspection to final measurements and calculations, and include details of the test equipment and personnel employed in performing the tasks.

8.6 Training

a. Trained and competent personnel shall be employed for all calibration and analysis operations.

b. A training programme shall be developed, maintained and implemented to provide for excellence of workmanship and personnel skills and a thorough knowledge of the requirements detailed in this Standard.

c. Trained personnel performing calibration and analysis shall be certified. This certification shall be based upon objective evidence of reproducibility and accuracy, resulting from the procedure outlined in Annex A.6.

d. Personnel shall be retrained or re-assessed annually to maintain the required skills.

e. Records shall be maintained of the training and certification status of personnel performing calibration and analysis.

9

Validation of measurement equipment

9.1 General

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

The infrared spectra from test houses that are obtained using methods conforming to the requirements in this Standard, shall be only accepted for customer projects if the test house is certified to perform the applicable procedures.

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

9.2 Initial audit of the system (acceptance)

a. Once a system is built or purchased, it shall be audited by the customer's product assurance department before it can be accepted for running qualification tests or quality control tests on materials intended for use in customer projects.

b. This initial audit shall, as a minimum, consist of (but not necessarily be restricted to) the following:

- an inspection of apparatus and associated equipment,
- calibration,
- performance of a test on a defined set of materials,
- reporting of the nonconformances and the audit findings.

9.3 Annual review (maintenance) of the system

The following reviews of the system shall be performed annually:

a. Apparatus and associated equipment shall be inspected.

b. Mutual comparability shall be evaluated (tested).

c. Equipment shall be calibrated.

d. If the system shows a non-conformance for any of the annual inspections with customer specifications or the acceptable limits of the test results, actions shall be undertaken by the test house to determine the reasons for the nonconformance and corrective actions implemented.

e. A written detailed report of the results of the regular review shall be delivered to the customer within six weeks after the end of the regular review or evaluation testing.

9.4 Special review

a. All modifications of the apparatus or associated equipment shall be reported and, if applicable, be audited by the customer before the modified system is used for a customer project.

b. Major modifications shall result in the retesting of apparatus as described in subclause 9.2.

Annex A (normative)

Calibration of infrared equipment and training of operators

A.1 Introduction

In this Annex A, the procedures for calibrating the infrared equipment and training operators are described.

A.2 Theory

A.2.1 Lambert-Beer's law

Lambert's law states that for parallel, monochromatic radiation that passes through an absorber of constant concentration, the radiant power decreases logarithmically as the path length increases arithmetically.

Beer's law states that the transmittance of a stable solution is an exponential function of the concentration of the absorbing solute. If both concentration and thickness are variable, the combined Lambert-Beer's law is expressed by equation (4):

$$A(\bar{\nu}) = \varepsilon(\bar{\nu}) l C \qquad (4)$$

where:

$A(\bar{\nu})$ is the absorbance at wave number $\bar{\nu}$,

$\varepsilon(\bar{\nu})$ is the molar absorptivity at wave number $\bar{\nu}$,

l is the path length,

C is the molar concentration.

To quantify organic contamination, the absorbance is expressed as the mass of a standard material per surface area unit. The trend line is calculated from the calibration curve (see subclause 6.2).

Four materials (see Table A-1) shall be used as a standard for the quantification. These materials are characteristic of the most common contaminants (hydrocarbons, esters, methyl silicones and phenyl silicones).

For contaminants that are unknown but are similar to the standard materials, the relation between the mass and the absorption at a specific wavelength of a standard is used for the quantification. As a result, this method provides the mass of the contaminant in terms of an equivalent amount of the standard material. If the contaminant matches the standard materials, this method is quantitative, otherwise it is semi-quantitative.

A.2.2 Dependency of equipment and operator

When an infrared-transparent window is used as a witness plate, the measurement is done directly on the window. This method is called the direct method. The amount of organic contamination measured depends on the area analysed, which corresponds to the diameter of the infrared beam.

For the indirect method the operator shall:

a. transfer the washed contamination from the Petri dish to the infrared-transparent window;

b. position the solvent containing the contaminants in the area of the infrared beam.

The efficiency of transfer and deposition is dependant on the operator. Operators shall be trained before performing analysis (see A.6).

The equipment shall be calibrated for obtaining quantitative information.

A.3 Optimization of equipment

A.3.1 Alignment of the sample holder

The sample holder in the sample compartment of the infrared spectrometer shall be aligned so that the infrared beam is positioned in the centre of the infrared transparent window as follows:

a. A mask plate is made with an aperture of 1 mm – 2 mm diameter.

b. This mask is placed in the window holder and positioned in the sample compartment of the spectrometer.

c. The aperture of the instrument is set to 1 mm.

d. By adjusting the position of the sample holder across the IR beam, the optimum position is determined.

e. The sample holder is fixed at this position along the line of the IR-beam and should remain at this position because, in most equipment, the focal point of the IR-beam is set to be in the sample compartment. This means that the beam diameter can be different if this position is changed.

f. Once the sample holder is aligned, the diameter of the beam shall be measured. This can be done by masking the window holder, using tape, from the top until the tape absorbs IR light. This is repeated from the bottom, left and right. A square is formed on the holder which marks the area where the IR beam passes through without touching the tape.

g. The size of the square is measured and used in further calculations.

A.3.2 Noise reduction

A.3.2.1 Dispersive infrared

The signal to noise (S/N) ratio for a dispersive instrument is given as a function of the wavelength resolution. Low signal to noise means low resolution. This is due to the use of slits. The resolution is not the most important factor of the analysis and can be set for this type of equipment between 8 cm^{-1} and 16 cm^{-1}.

A.3.2.2 Fourier transform infrared

For FTIR equipment there are several aspects that can influence the S/N ratio. The signal to noise ratio given by the manufacturer is commonly determined at 2 100 cm^{-1}. This is because the highest energy from the source is in this range and there is no interference of peaks from water vapour.

In most cases, a DTGS detector is favourable for high energy measurements and it also has a wider dynamic range compared to an MCT.

The S/N is measured over three ranges:

- 3 000 cm^{-1} - 2 800 cm^{-1},
- 1 800 cm^{-1} - 1 500 cm^{-1},
- 900 cm^{-1} - 700 cm^{-1}.

These three ranges correspond to lower energy levels. However, the range 3 000 cm^{-1} - 2 800 cm^{-1} contains peaks from water vapour which results in lower S/N levels than those defined by the manufacturer, but are in this case, more relevant for calculating the detection limits.

For an FTIR spectrometer, a resolution of 4 cm^{-1} is adequate; higher resolution results in more noise. The spectrum is derived from the ratio between a number of sample scans and a number of background scans. The number of sample scans is usually equal to the number of background scans, but the S/N ratio of the background shall not be lower than the one from the signal. The collected spectrum shall not be smoothed to get a better S/N ratio.

When optimizing the S/N ratio the following applies:

- The optimum mirror speed and zero filling on.
- The optimum number of scans. The S/N ratio is improved by a factor of $\sqrt{number\ of\ scans}$. The limit is the stability of the equipment.
- The best apodization function and phase correction.
- The amount of energy to the detector shall be kept below saturation point.
- If the energy to the detector is too high, the beam should not be made smaller by adjusting the aperture. This makes the spot on the sample smaller and thus makes it more difficult to position the contamination in the analysing area. Therefore, for example, germanium windows or maze filters should be used to receive the optimum energy on the detector.

The manufacturer should be consulted for the optimum settings of the infrared spectrometer. The optimum protocols should be stored and used for the actual measurements.

A.4 Calibration

A.4.1 General

The equipment is calibrated after alignment. The standard materials used for the IR analysis (see Table A-1) are used typically in a laboratory. If different types of contaminants are frequently found, individual calibration curves for each type of contaminant should be made.

The calibration curve that is produced using the direct method can be used for the indirect method, taking into account the transfer efficiency factor. This factor is determined by measuring the loss of signal due to the transfer step from the Petri dish to the window. For experienced operators, this factor is almost 1, but for less experienced operators it can be significantly less and shall be quantified. Operators should be evaluated annually.

A.4.2 Preparation of calibration standards

The standards used for the IR-analysis are summarized in Table A-1. A typical process for the preparation of the standard is summarized below:

a. A high purity reference material is chosen, see Table A-1.

b. For the preparation of the stock solution, chloroform of spectroscopic grade, having a non-volatile residue (NVR) < 5 µg/g, shall be used. Before preparing the stock solution, the spectrum of the NVR from 10 ml of chloroform shall be recorded; the absorbance level shall be below 0,000 1 AU.

c. A stock solution is prepared from the reference standard with the appropriate concentration in chloroform (e.g. 25 mg in 250 ml for C = 0,1 g l^{-1}). If a wider concentration range should be covered, more than one stock solution can be prepared (e.g. solution A: 12,5 mg in 250 ml for C = 0,05 g l^{-1}, and solution B: 25 mg in 50 ml for C = 0,5 g l^{-1}).

d. The standards shall be conserved in a cool and dark area and the evaporation of the chloroform limited by sealing the measuring flask.

Table A-1: Standard materials used for the IR-analysis

Standard[a]	Chemical nature	Characteristic peaks (cm^{-1})
Paraffin oil[b]	Long chain aliphatic hydrocarbon	2 920
Bis(2-ethylhexyl) phthalate (DOP)	Aromatic ester	1 735
Poly(dimethylsiloxane)	Methyl silicone	1 260, 805
Poly(methylphenylsiloxane)	Methyl phenyl silicone	1 260, 1 120, 805

a Standard materials shall be of highest grade available, examples are given in Annex F.3.

b The ratio of peak heights (peak to baseline) between CH_2 (2 925 cm^{-1}) and CH_3 (2 955 cm^{-1}) shall be between 0,60 – 0,65.

A.4.3 Calibration method

The calibration is performed by transferring a defined volume from the standard stock solution directly onto the IR-window.

a. The gas-tight syringe is filled with a defined volume from the standard stock solution as given in Table A-2. The concentration range can be extended if applicable.

b. The droplets from the syringe shall be positioned in the centre of the IR-window, within the area where the IR beam covers the window. The window should be put above a circular mask that corresponds to the size of the IR beam, and viewed from above the window using a magnification device.

c. Step b. shall not be repeated with clean chloroform. The syringe contains a dead volume, which shall not be added to the already transferred standard solution.

d. The IR-window is positioned in the sample compartment of the spectrometer.

e. The spectrum is recorded and the transmission loss for the respective standards is measured at the following wave numbers (see also Table A-1):

- 2 920 cm^{-1} for hydrocarbons,
- 1 735 cm^{-1} for esters,
- 1 260 cm^{-1} or 805 cm^{-1} for methyl silicone,
- 1 260 cm^{-1}, 1 120 cm^{-1} or 790 cm^{-1} for methyl phenyl silicones.

f. Each point should be measured at least three times, possibly with different windows in order to eliminate systematic errors.

Table A-2: Volumes to be applied from stock solutions and respective target amounts

Stock solution	Volume (µl)	Target amount (g)
A	1,0	$5{,}0 \times 10^{-8}$
A	2,5	$1{,}3 \times 10^{-7}$
A	5,0	$2{,}5 \times 10^{-7}$
B	1,0	$5{,}0 \times 10^{-7}$
B	2,5	$1{,}3 \times 10^{-6}$
B	5,0	$2{,}5 \times 10^{-6}$
B	10,0	$5{,}0 \times 10^{-6}$

Alternative calibration methods include the use of an evaporation vacuum chamber containing a quartz crystal microbalance (QCM). The standard material is put in an electrically heated cell and yields, through a small hole, a homogenous stream of contamination in direct view of a QCM and IR-window.

The QCM measures the contamination on the IR-windows with an accuracy of 10^{-9} g cm^{-2}. The IR-windows can be directly measured in the FTIR and be used for calibration.

This QCM method can have drawbacks due to the differences in the view factor and the differences in the temperatures between the QCM and the IR-transparent window. As the process is performed in a vacuum, re-evaporation can affect the values.

A.4.4 Calibration curve

A graph can be plotted of all the values measured, with the absorbance ($A = \mathrm{Log}(I_0/I)$) of the standard material versus mass. An example of a calibration curve for DOP, on a double logarithmic scale, is shown in Figure A-1.

The peak height can be measured using the method indicated in Figure A-2. An alternative method is to calculate the corresponding peak area.

It is important that the same method (e.g. peak height or peak area, or setting a base line) is used for the experiment and the calibration. The best fit (usually a linear line or power curve) through the average points constitutes the calibration curve and can be used for quantification analysis. The calibration curve should have a correlation coefficient higher than 0,98 for six sample points.

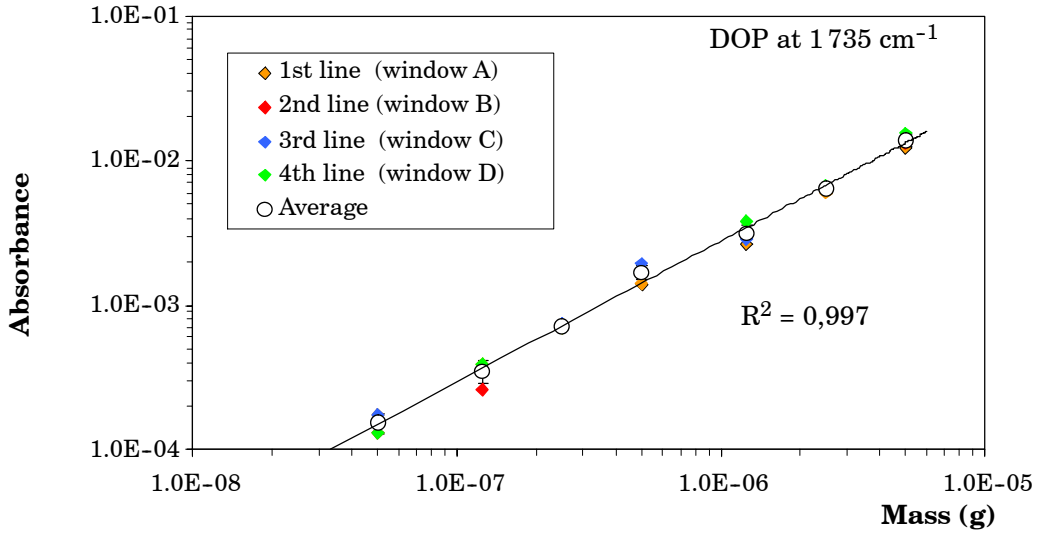

Figure A-1: Example for a calibration curve

The detection limit of the analysis can be calculated by using the S/N ratio at the specific wave number.

For the direct method, the detection limit is three times the S/N ratio. Mass at this absorbance level is divided by the analysed area (e.g. beam diameter/area: 7 mm/0,38 cm^2, 10 mm/0,79 cm^2, 12 mm/1,13 cm^2).

For the indirect method the detection limit is related to:

- The surface area of the witness plate or wiped area.
- The NVR of the solvents used.
- The extractable materials from the tissues used for wiping; this should be less than 5×10^{-7} g.
- The precision of the background correction for the NVR of the solvent and the tissue.

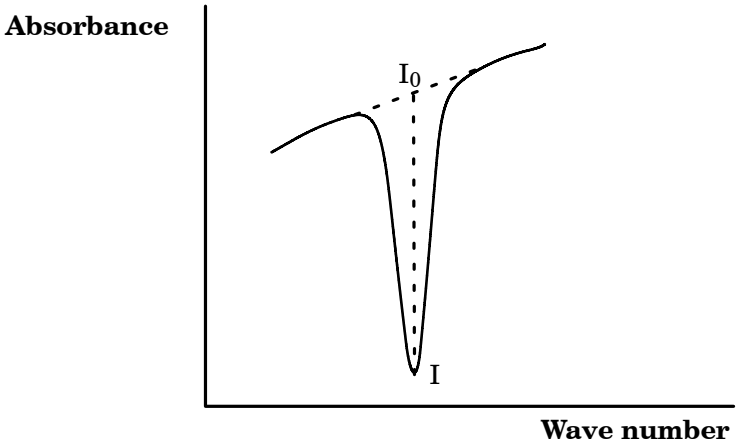

Figure A-2: Measurement of peak heights

A.4.5 Calibration results

Table A-3 shows the data obtained using the direct calibration method on a system with a beam diameter of 7 mm (0,38 cm^2). The peaks at 1 260 cm^{-1} and 1 120 cm^{-1} were selected, to be used with CaF_2 windows. These calibration lines are examples and are not generally applicable. Individual calibration lines shall be determined for each spectrometer, transfer process and operator.

Table A-3: Example results of the direct calibration method

Standard	Equation mass (g) =	Noise level (AU)	Detection limit (10^{-7}g)	Wave number (cm^{-1})
Paraffin	$5{,}55 \times 10^{-4} \times \text{absorbance}^{1{,}34}$	0,000 15	0,1	2 920
DOP	$7{,}72 \times 10^{-4} \times \text{absorbance}^{1{,}29}$	0,000 1	0,1	1 735
DC 200	$3{,}66 \times 10^{-4} \times \text{absorbance}^{1{,}14}$	0,000 1	0,2	1 260
DC 710	$5{,}84 \times 10^{-3} \times \text{absorbance}^{1{,}38}$	0,000 1	0,3	1 120

A.4.6 Obtaining quantitative information by experiment

Unless the contaminant matches the calibration standard, quantification is always relative to the reference material and thus semi-quantitative. Contamination levels shall be expressed in terms of the contribution of the four main groups: hydrocarbons, esters, methyl silicones, and phenyl silicones. The calculation is performed using their characteristic group frequencies (see Table A-1), whereas the peak maximum of the same vibration mode is used as for deriving the calibration curve.

A different chemical environment from a functionality (e.g. substitution, or neighbouring group effects) can lead to shifts in the frequency of the respective vibration mode. The selected absorbance yields the mass of the contaminant via the calibration curve in units of the corresponding grams of the standard material (see Table A-3).

A.5 Detection limits

A.5.1 Detection limits using the direct method

The direct method is performed with IR-transparent witness windows exposed for a specific period of time. The contamination is measured directly by placing it in the infrared beam.

An alternative method is to use highly reflecting witness plates placed on a reflection accessory. The limitation of this method is due to the beam diameter and the noise level of the equipment. The beam diameter is limited by the spectrometer and therefore fixed, e.g. the Bruker IFS-66v has a maximum aperture of 12 mm (1,13 cm^2), and the Mattson Cygnus 100 has a maximum aperture of 7 mm (0,38 cm^2). The S/N ratio of the spectrometer is also limited, but can be improved by increasing the number of scans.

The noise level of the equipment should be measured at the wave numbers of the calibration standards (2 920 cm^{-1}, 1 730 cm^{-1} and 800 cm^{-1}). The noise level should be at least three times less than the signal in order to recognize a signal.

For direct transmission measurements the detection limit of the IR spectrometer should be such that a signal of 0,000 1 AU can be measured.

A reflecting accessory can be used for the analysis of highly reflecting surfaces in order to obtain direct information about the contaminants.

The reliability of quantitative information, such as detection limits, depends on the accessories used and are not within the scope of this Standard.

A.5.2 Detection limits using the indirect method

The methods are described in Annex B and Annex C. After transfer from the contaminated surface to an IR-transparent window using a suitable spectral grade solvent, the absorption of the residue shall be measured at the wave numbers of the calibration standards (2 920 cm^{-1}, 1 730 cm^{-1} and 800 cm^{-1}).

The detection limit for this method is determined by:

- the purity of the solvents,
- the cleanliness of wipes,
- the transfer efficiency of residue,
- the signal to noise ratio of the infrared spectrometer (should be such that a signal of 0,000 1 AU can be measured).

All solvents used should have a NVR of less than 5 μg/g and an infrared absorption of the NVR of less than 0,000 5 AU ml^{-1}.

The loss of efficiency during the transfer step can be compensated for by performing the same procedure with the calibration as with the analysis.

If the rinsing method is used (see Annex B and Annex C), a detection limit of approximately 5×10^{-9} g cm^{-2} can be obtained depending on the surface area analysed (e.g. for a 15 cm^2 witness plate).

For the wiping method (see Annex C), the amount of solvent used is higher, and the used tissue should have a contamination potential of less than 5×10^{-7} g. The wiped area shall be assessed (even when using a mask) and this introduces an additional error. The wiped area should thus be 100 cm^2 in order to reach a detection limit of about 2×10^{-8} g cm^{-2}.

A.6 Training operators

Operators shall be trained by performing the procedure in A.4.2 for preparing a hydrocarbon standard solution and also the following procedure:

a. A gas-tight syringe shall be filled with the standard solution containing an equivalent of 1×10^{-6} g analyte and put in the Petri dish.

b. The sample shall be transferred, drop-wise, with the glass rod or micro-syringe from the Petri dish onto the IR-window within the area of the IR-beam.

c. After all droplets are transferred to the window, the Petri dish shall be washed with a few droplets of fresh chloroform and transferred again according to step b..

d. Step c. shall be repeated at least twice.

e. The IR-transparent window shall be placed on the sample holder in the sample compartment of the pre-aligned spectrometer.

f. A spectrum shall be recorded and the transmission loss measured for hydrocarbons at about 2 920 cm^{-1}.

g. Steps a. to f. shall be repeated 10 times.

h. All 10 measurements should be within 20 % of the average value. Experienced operators should be able to perform this test within 10 % of the average value.

Once the positioning or transfer of the solution can be performed within the accepted limits, the trainee operator can start to produce the calibration curves as described in A.4.

Annex B (informative)

The use of molecular witness plates for contamination control

B.1 General

In this Annex, the handling and use of molecular witness plates is described. It is written as a practical guide. The method used to analyse the plates corresponds to the infrared method.

B.2 Design of the witness plates

Stainless steel polished plates can be used to verify the cleanliness level of satellite hardware by being exposed adjacent to it, or they can be used to monitor the deposition of contamination in a test area such as as cleanrooms and vacuum chambers.

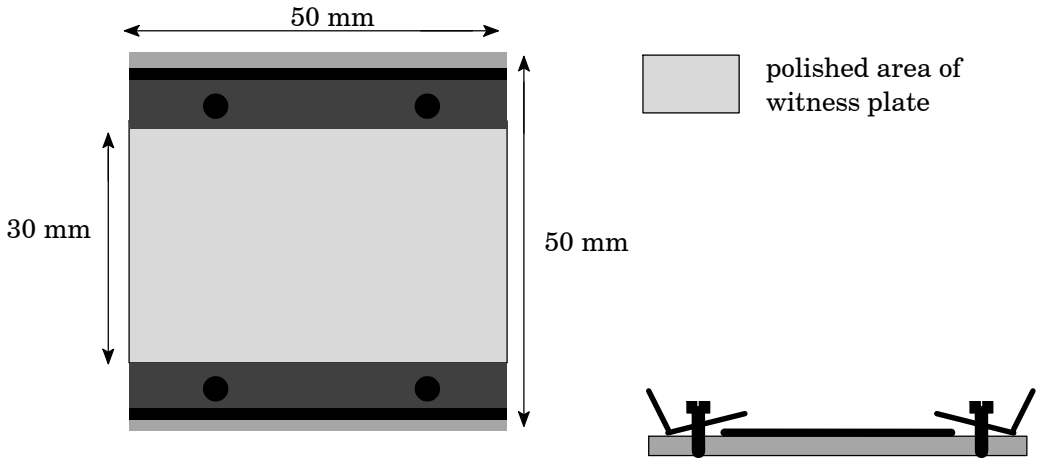

Figure B-1: Witness plate holder and witness plate used for organic contamination control

The size of the witness plate shall be 50 mm × 30 mm. For handling, it is fixed onto a stainless steel or aluminium holder of 50 mm × 50 mm with fixed upstanding bolts that are used for mounting the witness plate.

B.3 Cleaning the witness plates

B.3.1 General

Witness plates are cleaned by the provider of the witness plates.

B.3.2 Materials

- Chloroform of spectroscopic grade with NVR < 5 μg/g and stabilized with ethanol.
- Glass Syringe: 10 ml, plunger coated with PTFE.
- Transport container, preferablly metal.
- Tweezers.
- Solvent resistant clean gloves.
- Tissue, cotton, lint free.
- Ultrasonic bath.

B.3.3 Procedure

a. The polished stainless steel witness plate and holder shall be cleaned in an ultrasonic bath with a suitable solvent, to remove excessive contamination, and rinsed with demineralized water or spectroscopic grade solvents. For low levels of contamination UV-O_3 cleaning can be used as an alternative.

b. The witness plate shall be cleaned with a tissue and chloroform.

c. The witness plate shall be handled using tweezers and rinsed with chloroform three times using a syringe held at an angle of 60°.

d. The last droplet of chloroform at the bottom of the plate can be tapped off against the tissue.

e. The holder shall be rinsed with chloroform in the same way as the witness plate (using tweezers).

f. The holder and witness plate shall be reassembled without touching the surface of the polished plate.

B.4 Storage and transport of witness plates

a. After cleaning, the witness plates shall be stored in a pre-cleaned box (e.g. metal).

b. The box shall not cause any detectable contamination on the witness plates.

c. The box shall be packed in a clean ESD bag. The bag shall not contain any volatile organic processing aids, e.g. slipping agents, that can cause molecular contamination.

d. The following criteria shall apply for the packaging:
   - The box should not have an organic coating on the inside.
   - The box should not have open holes.
   - The lid should be tight.
   - If the lid has to be taped, an adhesive tape with low outgassing values (e.g. polyimide tape with acrylic adhesive) should be used.
   - The contact surface between the box and the lid should not be painted.
   - The clean bag in which the box is packed should be sealed or airtight.

e. The plates should be transported at a temperature between 10 °C and 30 °C.

f. The plates should not be stored in the vicinity of high outgassing materials or water.

g. The packaging shall only be opened in a clean environment by qualified personnel.

B.5 Handling of witness plates

a. The witness plate is fixed onto a holder. The surface of the witness plate should not be touched and not breathed upon.

b. The witness plate holder should be handled by the upstanding edges with tweezers or gloved hands (of cleanroom quality).

c. The witness plate shall not be used when it is stored, unused, for more than two months. After such a period the witness plate shall be sent back to the supplier.

B.6 Exposure of witness plates

a. Molecular contaminants consist of organic molecules that are condensable under an ambient environment. When molecules are adsorbed onto a surface, the surface temperature, the environmental pressure, as well as the vapour pressure of the contaminant, influence the time that the molecule is resident on the surface.

b. To obtain representative results during the exposure experiment, the witness plate shall be subject to the same conditions as the hardware.

c. Witness plates should be placed in, for example, vacuum systems or cleanrooms, at locations around the hardware and near potential sources of contamination, e.g. in the vicinity of soldering or other ‘dirty’ activities.

d. The cleanliness acceptance levels are defined in ECSS-Q-70-01. For vacuum systems the acceptance limits shall be given for a representative blank test over a period of at least 24 hours. The acceptance level for cleanrooms is defined after an exposure of one week. For a continuous verification in a cleanroom, one of the following exposure sequences can be applied:

1. Method 1
   (a) Two witness plates shall be placed adjacent to each other at the same location.
   (b) Plate 1, the (accumulated) witness plate, is the witness for the total exposure time. Plate 2, is replaced weekly (weekly requirements according to ECSS-Q-70-01), every two weeks, or monthly.
   (c) Plate 2 is analysed to verify the cleanliness for the exposed period (a week, two weeks, month).
   (d) If contamination is evident from plate 2, then plate 1, the accumulated witness plate, can be analysed to confirm the results of plate 2.
   (e) If there was no contamination problem during the total exposure time, plate 1 can be analysed to quantify the accumulated contamination levels.
2. Method 2
   (a) Two witness plates shall be placed adjacent to each other at the same location.
   (b) One of the witness plates, plate 1, is analysed after exposure for one week and replaced by a new one.
   (c) The second witness plate, plate 2, is exposed for two-weeks, then analysed and replaced by a new witness plate 2.
   (d) If there is a contamination problem, witness plate 2 can be analysed in order to confirm the results of witness plate 1.

e. After exposure, the witness plate shall be packed immediately and sent as soon as possible to the laboratory that performs the analysis. The NVR shall be analysed according to this Standard, not later than 4 weeks after the end of the exposure experiment.

f. When applying long exposure times to witness plates, there is a proportional accumulation of contaminants when the contamination rate is expressed in time units, which are different from the exposure times.

B.7 Witness plate information sheet

A witness plate information sheet should be filled in and a logbook kept for all witness plates that are used for contamination detection. This information sheet shall be sent with the packed witness plate to the laboratory for analyses. An example of a witness plate information sheet is given in Figure B-2.

Witness plate information sheet

Project: **Specimen verified:**

Cost code: **Date:**

Test centre: **Chamber/Cleanroom:**

Initiator: **Results to:**

Description of test (number/name, conditions, time, temperatures and pressure):

Witness plate no.	Location of witness plates	Exposed (date, days, hours)

Figure B-2: Example of a witness plate information sheet

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Annex C (informative)

Collecting molecular contamination from surfaces by wiping and rinsing

C.1 Introduction

C.1.1 General

Wiping and rinsing is the only method for verifying contamination levels on non-witnessed surfaces. In this Annex C, the methods for cleaning the necessary tools and performing the wiping and rinsing process are described.

C.1.2 Wiping methods

There are two wiping methods: a dry and a wet method. The dry wiping method can be used, in most cases, on painted surfaces and on plastic foils. The wet wiping method is only used on surfaces that are compatible with the solvents. Typical solvents are spectroscopic grade IPA or chloroform.

The wiping method can be used to indicate the level of contamination of a specific surface. When comparing the results of measuring contamination from wipes or using witness plates, the witness plates provide, in most cases, more reliable results. There are three reasons for this:

- The transfer of contaminants from the surface using the wiping method is never 100 %. This is especially critical if the contaminants have poor solubility or are cross-linked e.g. by UV-induced deposition.
- The wiping method has a higher background signal in FTIR than the witness plate analysis, therefore a surface of about 100 cm^2 should be wiped (if possible). However, for highly contaminated surfaces it should be taken into account that the large amount of material on the IR-transparent window can lead to a saturation of the signal.
- The results of wiping a coated or a plastic surface indicate contamination at that area, including the dissolved surface material.

The higher background signal of the wipes can be corrected by subtracting the spectrum of a blank wipe and from the solvent NVR.

C.1.3 Rinsing method

The rinsing method can only be used when the rinsing solvent can be collected, directly or by being absorbed in a clean tissue, and when the surface is compatible to the solvent used.

In most cases the rinsing method has a lower background signal compared to the wiping method. Another advantage of rinsing over wiping is that wiping can damage sensitive surfaces because the surface has been "touched" using some force.

C.2 Preparations

C.2.1 General

The tissues used for wiping are prepared by the tissue provider. The user should not perform any cleaning on the tissue.

C.2.2 Materials for wiping and rinsing

- Tweezers: 145 mm curved 45°.
- Tweezers: 145 mm straight.
- Glass Syringe: 10 ml, plunger coated with PTFE (for rinsing and wiping).

  NOTE Plastic syringes should not be used because the rubber plunger contains silicone.
- Lens tissue, cleaned, e.g. tissue paper for cleaning optical glasses, size 100 mm × 150 mm.
- Petri dish: 70 mm diameter (for rinsing).
- Glass bottle with lid, cleaned.

  NOTE Plastic lids often supplied with glass bottles can contain some mould release agent on the surface. They shall be properly cleaned to prevent cross-contamination.
- Chloroform of spectroscopic grade, NVR < 5 µg/g.
- Isopropyl alcohol (2-propanol) of spectroscopic grade, NVR < 5 µg/g.
- Acetone of spectroscopic grade, NVR < 5 µg/g.

C.2.3 Cleaning of filter papers, foam rubbers and tissues

The tissues shall be cleaned as follows:

a. The tissues shall be cut into the dimensions required for wiping. e.g. pieces of 100 mm × 50 mm.

b. The tissues shall be placed in a Soxhlet extraction unit.

c. Extraction shall be performed using acetone for four hours.

d. The solvent shall be replaced with chloroform, extracted for 12 h, replaced with fresh chloroform and extracted for another 12 h.

e. After extraction, a representative tissue shall be analysed according to 5.3.3.c.

f. If the tissue contains more than 5×10^{-7} g contamination (corrected for solvent background), extraction shall be continued until an acceptable background level is achieved.

g. The cleaned tissues shall be stored in a special container or directly in a clean glass bottle.

This procedure (from steps b. to g.) shall be used to clean filter papers and foam rubbers.

C.2.4 Cleaning bottles and Petri dish

Glass bottles shall be cleaned by rinsing the bottle with the appropriate solvents (the final solvent being chloroform) and dried by holding it upside down.

Petri dishes are cleaned in the same way as glass bottles. If the lid is made of polyethylene, the caps can contain a slipping agent used during production. This can be removed with clean isopropyl alcohol and chloroform.

C.2.5 Controlling the quality of the solvent

The quality of the solvent used for cleaning the materials and for the wiping procedure shall be evaluated. A known quantity of solvent (e.g. 10 ml) is evaporated and the residue weighed using a micro-balance. Furthermore, an infrared analysis is performed, conforming to this Standard, to establish the necessary data for spectral corrections.

A quick check of the purity of the solvents can be performed by dripping a few droplets from the filled syringe onto a clean witness plate and visually observing the residue on the surface after evaporation. If the residue is visible to the naked eye, the solvent cannot be used.

NOTE This visual method requires experience, and contamination levels below 10^{-6} g cm^{-2} are hardly visible to the naked eye.

C.3 Performing the wipe and rinse method

C.3.1 Wiping method

a. The syringe and two pairs of tweezers shall be cleaned with the relevant solvents and finally with chloroform before use.

b. A cleaned tissue shall be taken out of the transport container using the straight tweezers.

c. The tissue shall be folded a few times, using both tweezers, until it can be used as a little “sponge”.

d. The folded tissue shall be held with the curved tweezers and the surface wiped several times in four directions. When performing a wet wipe, the tissue is moistened with the solvent prior to wiping.

e. After wiping, the tissue shall be left until all the solvent has evaporated. The tissue is then placed in the glass bottle, the lid closed, the bottle numbered, and the NVR analysed according to this Standard.

f. The location wiped, the total area, the solvent used, and the type of surface wiped shall be recorded. See C.4 for a sample information form.

C.3.2 Rinsing method

a. The Petri dish that is used as the solvent collector and a syringe shall be cleaned with the relevant solvents (and finally chloroform).

b. The surface area to be cleaned can be rinsed gently using the syringe containing the solvent without wetting surrounding areas. The solvent is collected directly in the Petri dish.

c. The collected solvent shall be left in the Petri dish to evaporate and the NVR is analysed according to this Standard.

d. If necessary, to determine the NVR, a second Petri dish containing the residue of a known amount of clean solvent should also be analysed.

e. The amount of solvent used, the type of solvent, the location that has been rinsed, the type of surface and the area rinsed shall be recorded. See C.4 for a sample information form.

C.4 Sample information form

When the wiping and rinsing procedures are performed, a record should be kept of the sample identification and all the information relevant for the analysis. This information is sent to the laboratory that performs the analysis. An example of a sample information form is given in Figure C-1.

Sample information form

Project:	**Specimen verified:**
Cost code:	**Date:**
Test centre:	**Chamber/Cleanroom:**
Initiator:	**Results to:**

The reasoning for wiping and rinsing:

Type of wiping method: WET/DRY

Type of solvent used: Chloroform/isopropyl alcohol/other:

Volume of solvent used:

Sample no.	Location	Surface area (cm^2)

Figure C-1: Example of a sample information form

Annex D (informative)

Contact test: measuring the contamination transfer of materials which can come into contact with spacecraft hardware

D.1 Introduction

The contact test is performed in order to measure the contamination transfer of materials which can come into contact with spacecraft hardware. Examples of these materials include: packaging materials, shielding materials such as covers and gloves, or materials that are not intended to be used under vacuum. The use of the contact test for molecular contamination control is described.

The contact test is also used to verify the contamination transfer from materials which can come in contact with spacecraft hardware. The samples are placed in direct contact with aluminium foils and compressed with a force of about 100 N cm^{-2} for 1 h, which is comparable to manual pressure.

D.2 Contact test

D.2.1 Materials and equipment

- Chloroform of spectroscopic grade, NVR < 5 μg/g.
- Glass Syringe: 10 ml, plunger coated with PTFE.
- Petri dish: ranging in diameter from 50 mm to 70 mm.
- Tweezers.
- Aluminium foil: approximately 16 μm thick.
- Two aluminium plates of at least 100 mm × 100 mm surface area and 5 mm thickness.
- Hydraulic press capable of applying a force of 10 kN.

D.2.2 Procedure

a. The aluminium foil shall be cut into pieces that are the same size as the aluminium plates (about 100 mm × 100 mm).

b. The sample shall be cut into pieces of 100 mm × 100 mm. Gloves and bags have inner and outer sides, they shall be kept traceable. If the sample is not

large enough, smaller sizes can be used, but the force applied by the press shall be adjusted to ensure that the same pressure is applied.

c. The aluminium plates shall be cleaned with the syringe containing chloroform. The plates are marked as A and B.

d. The aluminium foils shall be cleaned with chloroform until no contamination can be measured using the infrared method. The foils should only be handled with tweezers.

e. The aluminium foil shall be placed with the glossy side up on the aluminium plate A. The glossy side shall be in contact with the sample.

f. The first sample shall be placed on the clean aluminium foil. The orientation of the sample to this first foil (inner or outer side) side shall be recorded.

g. On top of the sample, another clean aluminium foil shall be placed with the glossy side towards the sample. This results in one sample sandwiched between two aluminium foils.

h. The aluminium plate B shall be placed on top of the sandwiched sample.

i. The package with the two aluminium plates shall be placed between the hydraulic press and a force that corresponds to a pressure on the sample of 100 N cm^{-2} shall be applied for 1 h. For example, if the size of the sample is 100 mm × 100 mm, the force should be 10 kN.

j. After 1 h the pressure shall be released and the aluminium plate B removed.

k. The side of the aluminium foil that was in contact with the sample shall be is rinsed with chloroform.

l. The chloroform shall be collected in a Petri dish.

m. The NVR shall be analysed.

Annex E (informative)

Immersion test: measuring the extractable contamination potential of materials that can come in contact with spacecraft hardware

E.1 Introduction

This Annex explains the immersion test in detail. It is performed for measuring the extractable contamination potential of materials that can come into contact with spacecraft hardware. This includes, for example, packaging materials, gloves, shielding materials such as covers, wipes or other cleaning materials, which are not intended to be used under vacuum. The use of the immersion test for molecular contamination control is described.

The immersion test is developed to verify the potential extractable contamination from materials with solvents. The samples shall be submerged in a NVR solvent for 15 minutes and the extracted contaminants shall be analysed. The most common NVR solvent is chloroform, however some materials can be chemically attacked by it. The type of contaminants that are expected are, for example, organic antistatic additives, slipping agents, mould release agents, or residual monomers from polymerization processes.

E.2 Immersion test

E.2.1 Materials and equipment

- Spectroscopic grade solvent with NVR < 5 μg/g: Examples include chloroform, isopropyl alcohol (IPA), hexane, mixture of 1,1,1-trichloroethane : ethanol = 3:1 (ASTM E 1560).
- Glass syringe: 10 ml, plunger coated with PTFE.
- Petri dish: ranging in diameter from 50 mm – 70 mm.
- Tweezers.

E.2.2 Procedure

a. The sample shall be cut into small parts, for example, thin films to 30 mm × 30 mm, or wires to 30 mm length.

b. The sample shall be put into a Petri dish and immersed with 3 ml of NVR solvent.

c. The Petri dish shall be covered with a lid and left for 15 min.

d. The sample shall be taken out of the solvent and rinsed with 1 ml of NVR solvent on both sides.

e. The NVR is analysed.

f. Gravimetric determination of the NVR can be performed if applicable.

Annex F (informative)

Selection criteria for equipment and accessories for performing the infrared analysis of organic contamination

F.1 Infrared spectrometers

F.1.1 General

The different types of infrared spectrometers and accessories used for performing the analysis of organic contamination are described in this Annex F.

F.1.2 Dispersive infrared spectrometer

The dispersive infrared spectrometer uses one of the oldest principals in infrared spectroscopy. In dispersive infrared spectrometers, the light coming from the source, a black body emitter (e.g. a Globar), is dispersed by a grating and the energy per wavelength is measured by a detector using a slit.

The advantage of this type of spectrometer is that the sample and reference beam can be measured at the same time with almost no influence of the environment on the spectra.

The disadvantage is the use of a monochromator with slits.

The slit width defines the resolution and the noise on the signal. For a better resolution the slit width can be decreased, but because this means that less light goes through, the signal to noise ratio decreases.

Therefore, there is a trade-off between resolution and signal to noise ratio. Furthermore, the time to acquire a full spectrum can take several minutes (depending on wavelength interval), because each wavelength is measured individually.

This type of infrared spectrometer is now commonly replaced by the Fourier transform infrared spectrometer.

F.1.3 Fourier transform infrared (FTIR) spectrometer

The Fourier transform infrared spectrometer (FTIR) became more feasible with the availability of computers. It works using an interferometer (usually a Michelson interferometer) instead of a monochromator.

The principal is that the IR beam emitted from the source, a black body emitter (e.g. a Globar), is separated by a beam splitter into two paths. One path length is fixed and defined by a standing mirror, and the other is variable and defined by a moving mirror (moving forwards and backwards).

After reflection, the two beams recombine at the beam splitter by undergoing constructive and destructive interference. The resulting modulated signal is directed through the sample compartment to the detector.

The position of the moving mirror is measured by a He-Ne laser. The signal measured by the detector is correlated in time with the position of the mirror. This results in an interferogram with the highest signal intensity in the centre when both mirrors are at an equal distance from the beam splitter.

This interferogram is transformed into a spectrum by a computer using the fast Fourier transformation. One spectrum is produced by one full movement of the mirror. A computer is necessary to collect and transform the data online, and depending on the computational power, several spectra can be recorded per second.

The advantages of this type of spectrometer over the dispersive spectrometer are as follows:

- All wavelengths pass through the sample simultaneously, which means that a whole spectrum can be measured quickly in one go.
- The noise on the spectrum is reduced by acquiring a larger number of spectra.
- The amount of signal going through the sample is not limited by a slit, but is limited by the detector.
- The resolution of the spectrum is determined by the path length of the moving mirror.

The disadvantage of the FTIR is that the reference and the sample signal are collected separately. This means that the environment can have a significant influence on the results, e.g. in the region where there is water absorption.

F.1.4 Detectors

In the mid-IR range, two types of detectors are commonly used: the DTGS and the liquid nitrogen cooled MCT.

F.1.4.1 DTGS detector

The DTGS (deuterated triglycine sulphate) is a pyroelectrical detector that generates an electric charge on its surface when the temperature is changed. The scanning speed of this type of detector compared to the MCT detector is slower, however, it has a wider dynamic range. The spectral region depends on the material of the window used, and corresponds to 9 000 cm^{-1} - 400 cm^{-1} with KBr.

F.1.4.2 MCT detector

The MCT (mercury cadmium telluride) is a photo-conductive or photovoltaic detector and is based on the semi-conductivity of the materials used. Electrons are released when hit by photons (with energies higher than the respective band gap) and the changes in the conductivity are thus related to the intensity of the received infrared radiation. MCT detectors are cooled with liquid nitrogen.

MCT detectors have a very short response time, but the response is characterized by a gradual increase in response with increasing wavelength followed by a sudden sharp drop.

The other advantage of the MCT compared to the DTGS is the high response to lower light levels. This is the reason why MCT detectors are chosen with reflection units or accessories, because signals with low energy throughput can still be measured.

F.2 Accessories

F.2.1 Transmittance measurements

F.2.1.1 Window materials

For the mid-IR region there is no "perfect" material for windows, and several trade-offs are made in terms of transmittance performance, ease of use and price. The following is a short summary of window materials that are commonly used. Table F-1 summarizes the important properties.

- Alkali metal halides (except fluorides): generally water soluble, low RI, and soft. Most commonly NaCl, KBr and CsI.
- Metal fluorides: low water solubility, low RI, most commonly CaF_2, MgF_2.
- Heavy metal halides: silver salts (AgCl, AgBr) are water resistant, transparent over the entire mid-IR, but weak and tend to cold flow. Thallium salts such as KRS-5 have an excellent spectral range and are very robust and have become a commonly used optical material, especially for ATR. The drawback is their high toxicity.
- Metal oxides: in general they represent all hard materials with a limited spectral range, e.g. MgO, α-Al_2O_2, and ZrO_2.
- Group II-IV chalcogenides: the two workhorses, ZnS and ZnSe are mechanically and chemically robust and for many applications (transmittive, ATR) the preferred material.
- Groups IV and III-V (diamond family): generally extremely hard and brittle, excellent resistance towards thermal shock. Diamond has superior IR transmittance (except the phonon band around 5 μm) and is most suitable for high-pressure cells. Si and Ge have extremely high RI, making them interesting for ATR applications, however, because of free thermal electrons they become opaque at elevated temperatures.

Table F-1: Important properties of common window materials used for infrared spectroscopy

Material	RI $n_{5\mu m}$	Wavelength range (μm)	T_{max} (°C)	Incompatible with
NaCl	1,52	0,4 – 15	400	Water, glycols, high humidity
KBr	1,54	0,3 – 25	300	Water, alcohols, ether, humidity
CsI	1,74	0,3 – 70	200	Water, alcohols, humidity
CaF_2	1,40	0,15 – 8	600	Ammonium salts, some concentrated acids
MgF_2	1,34	0,15 - 8	500	Concentrated acids
AgCl	2,00	0,42 – 27	200	Oxidizers, chelators, concentrated chlorides
AgBr	~2,15	0,5 – 35	200	Oxidizers, chelators, concentrated chlorides
KRS-5	2,38	0,6 – 60	200	Methanol, chelators, strong bases
MgO	1,64	0,4 – 8	> 2 000	Concentrated acids, ammonium salts
α-Al_2O_3	1,62	0,15 – 5	1 700	Concentrated acids and bases
ZrO_2	2,13[a]	0,36 – 7	> 1 000	HF, H_2SO_4
ZnS	2,25	0,4 – 14	300	Strong oxidizers, some acids
ZnSe	2,43	0,5 – 20	300	Acids, strong concentrated bases
Diamond	2,39	0,22 – 4,3, > 5,4	> 700	Chromosulfuric acid
Si	3,42	1,06 – 6,7, > 30	300	HF + HNO_3
Ge	4,02	2,0 – 17	100	Hot H_2SO_4, aqua regia

a RI at 1 μm

F.2.1.2 Sampling techniques

There are several techniques for sampling gaseous, liquid, and solid materials. For further details refer to the Handbook of vibrational spectroscopy (see Bibliography).

F.2.2 Reflection accessories

There are several reflection techniques, e.g. attenuated total reflection (ATR), diffuse reflectance (DRIFT), grazing angle, integrating spheres, or microscopy. Some of these are also capable of yielding semi-quantitative information. These techniques are based on different theories and use procedures which are not within the scope of this Standard. For further details refer to the Handbook of vibrational spectroscopy (see Bibliography).

F.3 Examples of reference compounds for calibration

The compound references and suppliers given below are examples; equivalent or better grades from alternative suppliers can be used.

- Hydrocarbons

  Grade: paraffin liquid for spectroscopy, Ultrasolv®

  Supplier: Merck
- Esters

  Grade: bis(2-ethylhexyl)phthalate >98%

  Supplier: Merck
- Methyl silicones

  Grade: poly(dimethyl siloxane), DC 200® fluid, 1000 centistokes

  Supplier: Dow Corning
- Methylphenylsilicones

  Grade: poly(methylphenylsiloxane), DC 710® fluid, 500 centistokes

  Supplier: Dow Corning

Bibliography

Handbook of Vibrational Spectroscopy; Chalmers, J.M., Griffiths, P.R., Eds.; John Wiley & Sons Ltd., Chichester, UK, 2002

Analytical Chemistry Handbook, J.A. Dean; McGraw-Hill, New York, USA, 1995

ASTM E 131	Standard terminology relating to molecular spectroscopy
ASTM E 168	Standard practices for general techniques of infrared quantitative analysis
ASTM E 1252	Standard practice for general techniques for obtaining infrared spectra for qualitative analysis
ASTM E 1560	Standard test method for gravimetric determination of nonvolatile residue from cleanroom wipers

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