

BS EN 16602-70-05:2014



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Space product assurance — Detection of organic contamination surfaces by infrared spectroscopy

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee ACE/68, Space systems and operations.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English version

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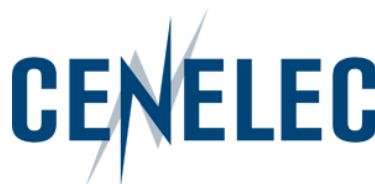
Raumfahrtproduktsicherung - Detektion von organischen Kontaminationen auf Oberflächen mit Infrarotspektroskopie

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Foreword

This document (EN 16602-70-05:2014) has been prepared by Technical Committee CEN/CLC/TC 5 "Space", the secretariat of which is held by DIN.

This standard (EN 16602-70-05:2014) originates from ECSS-Q-ST-70-05C.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2015, and conflicting national standards shall be withdrawn at the latest by April 2015..

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document has been developed to cover specifically space systems and has therefore precedence over any EN covering the same scope but with a wider domain of applicability (e.g. : aerospace).

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

One or more of the following organic substances can contaminate spacecraft materials and hardware, as well as vacuum chambers:

- Volatile condensable products of materials out-gassing under vacuum.
- Volatile condensable products of off-gassing materials.
- Back-streaming 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, which is the most widely used, is a simple, versatile and rapid technique providing high resolution qualitative and quantitative analyses. The technique is therefore baseline for the present Standard.

1

Scope

This Standard defines test requirements for detecting organic contamination on surfaces using direct and indirect methods with the aid of infrared spectroscopy.

The Standard applies to controlling and detecting organic contamination on all manned and unmanned spacecraft, launchers, payloads, experiments, terrestrial vacuum test facilities, and cleanrooms.

The following test methods are covered:

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

Several informative annexes are included to give guidelines to the following subjects:

- Qualitative and quantitative interpretation of spectral data
- Calibration of infrared equipment
- Training of operators
- Use of molecular witness plates
- Collecting molecular contamination
- Contact test to measure the contamination transfer of materials
- Immersion test to measure the extractable contamination potential of materials
- Selection criteria for test equipment

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

2

Normative references

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

EN reference	Reference in text	Title
EN 16601-00-01	ECSS-S-ST-00-01	ECSS system – Glossary of terms
EN 16602-10	ECSS-Q-ST-10	Space product assurance – Product assurance management
EN 16602-10-09	ECSS-Q-ST-10-09	Space product assurance – Nonconformance control system
EN 16602-20	ECSS-Q-ST-20	Space product assurance – Quality assurance
EN 16602-70-01	ECSS-Q-ST-70-01	Space product assurance – Cleanliness and contamination control

3

Terms, definitions and abbreviated terms

3.1 Terms defined in other standards

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

controlled area

3.2 Terms specific to the present standard

3.2.1 absorbance, A

logarithm to the base 10 of the reciprocal of the transmittance

[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.

3.2.2 absorption

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

3.2.3 absorptivity

absorbance divided by the product of the concentration of the substance and the sample path length

NOTE 1 Absorptivity = $A/(lC)$, where A is the absorbance, C is the concentration of the substance and l is the sample path length. The unit normally used are cm for l , and kg m^{-3} for C .

NOTE 2 The equivalent IUPAC term is “specific absorption coefficient”.

[adapted from ASTM-E-131]

3.2.4 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]

3.2.5 diffuse reflection

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

[ASTM-E-131]

3.2.6 Fourier transformation

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

[ASTM-E-131]

3.2.7 infrared spectroscopy

spectroscopy in the infrared region of the electromagnetic spectrum, i.e. with wavelength range from approximately 0,78 μm to 1000 μm (wave number range 12820 cm^{-1} to 10 cm^{-1})

[adapted from ASTM-E-131]

3.2.8 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]

3.2.9 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).

3.2.10 reflectance, R

ratio of the radiant power reflected by the sample to the radiant power incident on the sample

[ASTM-E-131]

3.2.11 specific area

diameter of the infrared beam at the window location

NOTE It is expressed as the ratio of the beam diameter over the area. For example, 7 mm/0,38 cm², 10 mm/0,79 cm² or 12 mm/1,13 cm².

3.2.12 transmittance, T

ratio of the radiant power transmitted by the sample to the radiant power incident on the sample

[ASTM-E-131]

3.2.13 wave number, $\bar{\nu}$

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 λ 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]

3.3 Abbreviated terms

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

Abbreviation	Meaning
ASTM	American Society for Testing and Materials
ATR	attenuated total reflection
AU	absorbance unit
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

4 Principles

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. The most common methods for measuring contamination are:

- **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, clean-room 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.

The direct method has demonstrated higher reliability because the sample does not require transfer from the witness plate and therefore reducing the error for quantification. The indirect method allows sample concentration and can therefore provide higher sensitivity.

5

Requirements

5.1 Preparatory activities

5.1.1 Hazard, health and safety precautions

- a. Unavoidable hazards to personnel equipment and materials shall be controlled by risk management procedures and kept to a minimum.
- b. Hazardous substances, items and operations shall be isolated from other activities.
- c. Items and controls shall be located in order to prevent personnel to be exposed to hazards.

NOTE Typical hazards are electric shock, cutting edges, sharp points, and toxic atmospheres.

- d. Warning and caution notes shall be included in instructions for operation, storage, transport, testing, assembly, maintenance and repair.
- e. Hazardous items, equipment or facilities shall be clearly marked to instruct personnel that they should take the necessary precautions.
- f. Before starting any operation, safety hazards shall be identified, and the necessary precautions taken to minimize risks.

NOTE For example, use of protection devices when chloroform is used.

- g. Operations requiring safety suits and protection devices shall be initiated after the personnel involved have the required protection, including any specific protection devices available at the work-place.

5.1.2 Facilities

5.1.2.1 Cleanliness

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

5.1.2.2 Environmental conditions

- a. The ambient conditions for the test, process and work areas shall be
 - 1. Room temperature (22 ± 3) °C and,
 - 2. Relative humidity (55 ± 10) %.

NOTE Additional conditions can be imposed for critical operations.

5.1.3 Materials

- a. Materials used in the process shall be stored in a controlled area in conformance with clause 5.1.2.
- b. Limited-life materials shall be labelled with their shelf lives and dates of manufacture.

5.1.4 Handling

- a. It shall be demonstrated that no additional contamination is introduced during the handling process.

NOTE 1 Contamination can be avoided by using tweezers and clean gloves, and ensuring that gloves and chemicals are compatible.

NOTE 2 Typically used gloves are of powder-free nylon, nitrile, latex, lint-free cotton.

5.1.5 Equipment

5.1.5.1 Infrared spectrophotometer

- a. The spectrometer shall have the following specification:
 - 1. Spectral range: At least, $4\,000\text{ cm}^{-1}$ – 600 cm^{-1} ($2,5\text{ }\mu\text{m}$ - $16,7\text{ }\mu\text{m}$).
 - 2. Resolution: 4 cm^{-1} .
 - 3. Absorbance of 0,0001 as detection limit for transmission methods.
- b. Interferences of environmental components shall be eliminated

NOTE Major environmental interferences are caused by H_2O and CO_2 . Elimination of H_2O and CO_2 is possible by flushing with the proper gases or applying a vacuum.

- c. Plates of infrared-transparent material shall be available.

NOTE 1 Typical materials are NaCl, MgF_2 , CaF_2 , ZnSe, or Ge.

NOTE 2 An ATR-attachment to the spectrophotometer can be used for direct analysis of the surfaces of materials.

NOTE 3 The results of the ATR infrared spectrophotometer technique are dispersed and

should therefore only be used for qualitative purposes.

5.1.5.2 Alignment of the sample holder

- a. The sample holder in the sample compartment of the infrared spectrometer shall be aligned for obtaining quantitative information.
- b. The sample holder shall be aligned so that the infrared beam is positioned in the centre of the IR transparent window.
- c. For alignment the following steps shall be performed:
 1. A mask plate is made with an aperture of (1 – 2) mm diameter.
 2. This mask is placed in the window holder and pointed in the sample compartment of the spectrometer.
 3. The aperture of the instrument is set to 1 mm.
 4. By adjusting the position of the sample holder across the IR beam, the optimum position is determined.
 5. The sample holder is fixed at this position along the line of the IR-beam.

NOTE It is important to keep the sample holder 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.

6. Once the sample holder is aligned, the diameter of the beam is measured.

NOTE The measurement of the diameter of the beam is performed by masking the window holder, using tape, from the top until the tape absorbs IR light

7. Step 5.1.5.2c.6 is repeated from bottom, left and right.
8. A square is formed on the holder, which marks the area where the IR beam passes through without touching the tape.
9. The size of the square is measured and used in further calculations.

5.1.6 Miscellaneous items

- a. The following items shall be used for acquiring and preparing the samples:
 1. Pre-cleaned standard filter paper of 70 mm diameter.

NOTE For orientation on the cleaning process see F.2.3.
 2. Piece of pre-cleaned foam rubber, approximately (50 × 30) mm.

NOTE See F.2.3.
 3. A PTFE film can be used to protect the foam rubber.
 4. Clean, powder-free and lint-free gloves.

5. Spectral grade solvents.
6. Petri dishes ranging in diameter from (50 - 70) mm.
7. Glass rod or micro-syringe.
8. Glass syringe.
9. Tweezers.
10. Infrared lamp.

5.2 Procedure for sampling and analysis

5.2.1 Summary

A summary of the procedures contained in this clause is given in Figure 5-1.

5.2.2 Direct method

- a. The following steps shall be performed for the determination of organic contamination by the direct method:

1. Position the infrared-transparent windows at or near critical locations.

NOTE For example, inside the compartment, the chamber or the clean-room to be monitored.

2. Verify that the witness plate is subjected to the same conditions that the location to be monitored.

NOTE For a representative measurement these conditions are crucial, e.g. identical temperature and pressure.

3. Before installation, record the spectrum of the cleaned, non-exposed window and retain for use as a background measurement.
4. Immediately after exposure, analyse the infrared-transparent windows with the IR spectrophotometer.

NOTE A waiting period after exposure can cause false results due to creeping of some kinds of contaminants (e.g. silicones).

5.2.3 Indirect method

5.2.3.1 Preparatory activities

- a. Surfaces shall be washed and wiped with solvents and tissues that are compatible with and do not damage the surface to be analysed.

NOTE 1 For example, solvation and swelling of any material that is not regarded as a contaminant.

NOTE 2 Scratching of the surface.