EUROPEAN COOPERATION

E CSS

FOR SPACE STANDARDIZATION

Space product assurance

Measurements of thermo-optical properties of thermal control materials

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-01-709, reviewed by the ECSS Product Assurance Panel and approved by the ECSS Steering Board.

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Introduction

The thermo-optical properties of materials are of importance to enable the calculation of the thermal housekeeping and radiative heat transfer.

This Standard describes the methodology, instruments, equipment and samples, used to calculate the thermo-optical properties of thermal-control materials, i.e. solar absorptance [α_s or α_p] and the infrared emittance [ε_h or ε_n].

In general this procedure has been written in connection with instruments and equipment available at ONERA, INTESPACE and ESTEC; however, any contractor is encouraged to built up his own instrument or equipment provided the accuracy of the results is equivalent to the one specified herein.

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Scope

This Standard describes the methodology, instruments, equipment and samples, used to calculate the thermo-optical properties of thermal-control materials.

The following test methods are detailed in this Standard including the configuration of samples and calculations:

- Solar absorptance using spectrometer: (α_s) – (see subclause 5.2).

- Solar absorptance using portable equipment: (α_p) – (see subclause 5.3).

- Infrared emittance using thermal test methods: (ε_h) – (see subclause 5.4).

- Infrared emittance using IR spectrometer: (ε_h) – (see subclause 5.5).

- Infrared emittance using portable equipment: (ε_n) – (see subclause 5.6).

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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

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Terms, definitions and abbreviated terms

3.1 Terms and definitions

The following terms and definitions are specific to this Standard in the sense that they are complementary or additional to those contained in ECSS-P-001.

3.1.1
absorptance
ratio of the intensity of the incident light to the transmitted or reflected light

3.1.2
emittance (ε)
ratio of the radiant intensity of the specimen to that emitted by a black body radiator at the same temperature and under the same geometric and wavelength conditions

EXAMPLE 1   Hemispherical emittance (ε_h) – conditions for incident or viewing of flux over a hemispherical region.

EXAMPLE 2   Normal emittance (ε_n) – conditions for incidence or viewing through a solid angle normal to the specimen.

3.1.3
solar absorptance (α)
ratio of the solar radiant flux absorbed by a material (or body) to that incident upon it

      NOTE   Differentiation is made between two methods:

- Spectroscopic method using a photospectrometer covering the range from 0,25 µm to 2,5 µm for the determination of α_s.
- Portable equipment using a xenon flash for relative measurements (α_p).

3.2 Abbreviated terms

For the purpose of this Standard, the abbreviated terms given in ECSS-P-001 apply.

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Preparatory conditions

4.1 Hazards, health and safety precautions

For materials and parts with hazardous characteristics see ECSS--Q--40. Attention shall be given to health and safety precautions. Hazards to personnel, equipment and materials shall be controlled and minimized.

4.2 Preparation of samples

4.2.1 Configuration

The material samples shall be prepared according to the relevant process specification or manufacturer's data and shall be representative of batch variance. For instance, the application procedure for paint can result in different thermo-optical properties, depending on the painter and the type of spray gun used. Therefore the samples should be coated or made at the same time as the workpiece.

4.2.2 Cleaning

Cleaning method and other treatment of the sample shall always be the same as for the flight hardware. Further cleaning or treatment of the sample is not allowed. In particular, solar absorptance properties are very sensitive to contamination and if the sample or the flight hardware is contaminated (even by hand grease), the test results are completely erroneous.

4.2.3 Handling and storage

Samples shall only be handled with clean nylon or lint-free gloves and shall be stored in a cleanliness-controlled area, with a room temperature of $(20 \pm 3)\,°C$ and relative humidity of $(55 \pm 10)\,\%$.

a. Coated surfaces shall be shielded from contact by using polyethylene or polypropylene bags or sheets.

b. Mechanical damage shall be avoided in the standard way by packing the polyethylene or polypropylene-wrapped workpieces in clean, dust- and lint-free material.

c. Limited-life materials shall be labelled with their relative shelf lives and dates of manufacture.

4.2.4   Identification

a.   Samples submitted for testing shall be accompanied by a completed "Material identification card".

b.   Hazardous samples shall be accompanied by a completed "Safety data sheet".

4.3   Facilities

4.3.1   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 sample.

4.3.2   Environmental conditions

The ambient conditions for the process and work areas shall be (22 ± 3) °C with a relative humidity of (55 ± 10) % unless otherwise stated.

4.3.3   Equipment

The equipment is specific for each test and defined in the test procedure.

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Test procedures

5.1 Format

One or more of the test methods described in this clause shall be selected and the appropriate procedures detailed for each method shall be adhered to.

The format of each subclause is as follows:

- 5.x.1 General
- 5.x.2 Configuration of samples
- 5.x.3 Test apparatus and setting up
- 5.x.4 Test process and measurement
- 5.x.5 Calculations

5.2 Solar absorptance using spectrometer (α_s)

5.2.1 General

Solar absorptance is calculated using the absorption spectrum of the material over the region from 0,25 μm to 2,5 μm and this spectrum is then multiplied with the solar spectrum.

a. The absorption spectrum shall be measured using an integrating sphere.

b. For absolute measurements a sphere with central sample mounting shall be used.

> NOTE A sphere with a sample holder on the sidewall can also be used. In this case the reflectivity is compared to a known standard (e.g. calibrated Al-mirror or calibrated Spectralon$^{\circledR}$ standard).

5.2.2 Configuration of samples

Typical dimensions of the sample are 15 mm × 15 mm to 25 mm × 25 mm. Depending on the method and equipment used, these dimensions can vary.

Flexible samples shall be mounted on a rigid surface. Measurements are only valid on flat samples. However, it is possible to perform measurements on spherical curved samples provided the radius of curvature exceeds 300 mm.

5.2.3   Test apparatus and setting up

The test apparatus consists of a spectrometer, covering the range from 0,25 μm to 2,5 μm.

a.   The wavelength resolution of the spectrometer shall be compatible with the resolution used for the solar spectrum.

b.   The signal to noise ratio shall be better than:

- ±1% full scale in the region between 250 nm and 2 000 nm;

- 5 % full scale in the region between 2 000 nm and 2 500 nm.

c.   The associated sphere shall have a maximum port to total surface ratio of 5 %.

> NOTE 1   If the test apparatus is used in a central sample mode, i.e. an "Edwards"-type integrating sphere, the measurement is called "absolute".

> NOTE 2   If the sample is mounted on the sidewall, the measurement is done towards a calibrated standard that can be specular (e.g. Al-mirror) or diffuse (e.g. Spectralon®).

d.   The responsible test officer shall make the choice of a standard based on the visual aspect of the sample.

e.   For materials having, in the visible region, a large specular component, a standard mirror shall be used.

f.   When the diffuse component is predominant in the visible region, a diffuse Spectralon® sample shall be used.

g.   The standard used for the measurement shall be indicated in the report.

5.2.4   Test process and measurement

Before starting a measurement sequence the 100 % and 0 % baseline shall be taken (using the standard reference as applicable). The baseline shall be measured at least once a day when equipment is switched on.

5.2.5   Calculation of absorptance

The spectrum is taken between 250 nm and 2 500 nm, and covers 96 % of the total energy.

$$\alpha_s = 1 - R_s$$

$$R_s = \frac{\int_{\lambda_1}^{\lambda_2} R(\lambda)S(\lambda)d\lambda}{\int_{\lambda_1}^{\lambda_2} S(\lambda)d\lambda}$$

where:

$R(\lambda)$ is the spectral reflectance after 100 % reference correction;

$S(\lambda)$ is the spectral solar irradiance (ASTM E 490);

$d\lambda$ is typically 1 nm;

λ_1 is 0,25 μm;

λ_2 is 2,5 μm.

For transparent test pieces it is possible to calculate the absorptance by the same method, because:

$$\alpha(\lambda) = 1 - [R(\lambda) + T(\lambda)]$$

It is also possible to calculate absorptance for a spectrum other than the solar spectrum, e.g. for solar simulators.

5.3 Comparative test method (α_p)

5.3.1 General

This method is based on comparing the reflection of a Xenon flash by a known appropriate reference material to the reflection of an unknown sample. The nature of the reference material (chemical composition and surface morphology) shall be representative for the unknown. The solar absorption of the reference surface shall be measured using the method described in 5.2.

This method has limitations due to the fact that the flasher spectrum is not identical to the solar spectrum.

Special precautions shall be taken when using portable reflectometer equipment.

This equipment shall only be used for comparative measurements. It shall be used in conjunction with known reference or calibrated materials, identical to or at least having similar optical behaviour to the material to be measured. If such an approach is followed, the equipment shall give a direct and correct result within the linearity limitation. If the reference used is not identical to the material under test, the result of the reading not only depends on the linearity of the equipment but also on the spectral reflectivity of the material. Any reporting of results shall include the detailed test conditions.

5.3.2 Configuration of samples

The minimal sample dimensions are dictated by the diameter of the aperture on the portable equipment. This diameter is typically between 15 mm and 20 mm.

Measurements are only valid on flat samples. However, it is possible to perform measurements on spherical curved samples, provided the radius of curvature exceeds 300 mm.

5.3.3 Test apparatus and setting up

The equipment consists of a flasher, able to produce a flash with a reproducible spectrum. The total intensity as well as the reflected intensity of the flasher are measured both with the reference surface and the test surfaces.

5.3.4 Test process and measurement

a. Before any measurement is performed the equipment shall be stabilized following the instructions given by the manufacturer.

b. The equipment shall be calibrated following the instructions given by the manufacturer.

c. The calibration shall, as a minimum, include a "zero" or baseline measurement as well as the measurement of the reference material.

d. After calibration with the appropriate reference material, the unknown sample shall be measured.

e. If several materials have to be measured, calibration shall be repeated at regular time intervals, depending on the known stability of the equipment.

f. For statistical reasons each measurement shall be repeated at least 5 times; the average and standard deviation of the measurements shall be calculated and reported.

g. Some equipment makes these calculations automatically through integrated software. The standard deviation between measurements shall be 0,02 or better.

5.3.5 Calculations

Reflectivity reference surface (R_r):

$$R_r = I_r/I_{tr}$$

where:

I_r is the intensity of flash reflected on reference surface;

I_{tr} is the total intensity of flash during reference flashing.

Reflectivity sample surface (R_s):

$$R_s = I_s/I_{tr}$$

where:

I_s is the intensity of flash reflected on sample surface;

I_{ts} is the total intensity of flash during sample flashing.

$$R = I_s/I_r \times R_r$$

where:

R_r is the measured reference reflectivity (using method 5.2.);

R is the calculated sample reflectivity.

$$\alpha_p = 1 - R$$

5.4 Infrared emittance using thermal test methods (ε_h)

5.4.1 General

By means of the dynamic thermal method, one can determine the total hemispherical emittance from the decrease in temperature of a test item with well-defined thermal characteristics.

5.4.2 Configuration of samples

A "standard sample substrate" as detailed in Figure 1 should be used in favour of any other geometrical shape.

In particular, the sample shall be made by machining and not by cutting out with shears since this flattens the edges and does not produce a very accurate square shape.
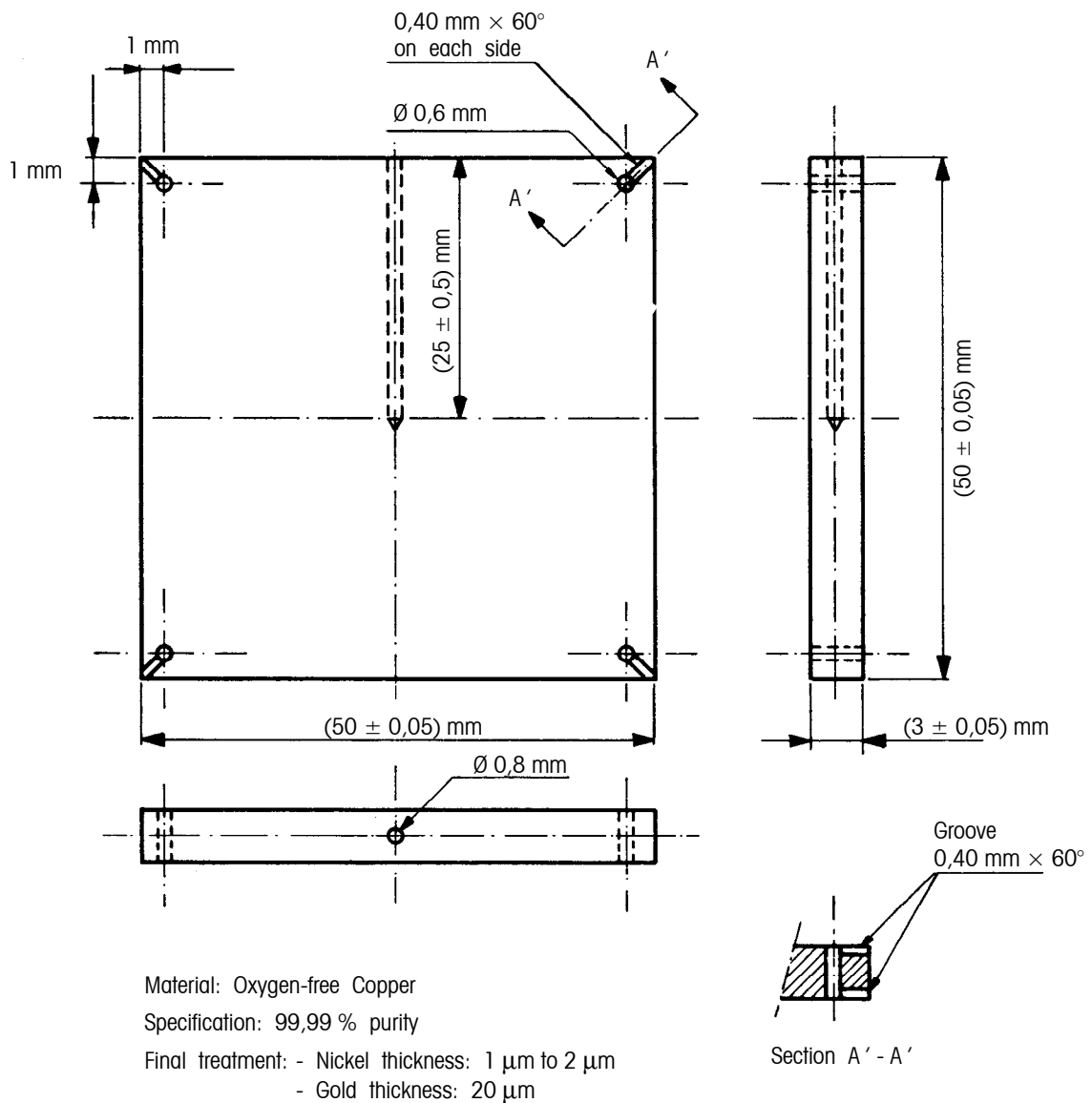
Figure 1: Standard sample substrate

5.4.3　Test apparatus and setting up

For the dynamic thermal method, it is assumed that the specific heat of the test piece is known. A lightweight test piece (i.e. one with a low heat capacity) is bonded by means of double-sided adhesive tape to a gold-plated substrate (standard sample substrate) the heat capacity of which is high with respect to that of the test piece.

Initially, the gold-plated substrate's emittance is measured. A copper-Constantan® thermocouple is fixed in the centre of the gold-plated substrate. Four nylon threads (e.g. $\varnothing$ = 0,16 mm) secure the substrate and test piece to the centre of a sample holder which is fixed on the axis.

This is then lowered to the centre of the cryogenic shroud, which is coated in black and cooled with liquid nitrogen.

The dimensions are such that the shroud area is more than 100 times the total area of the test piece.

A window made of Suprasil® quartz (working diameter: 90 mm) enables this test piece to be illuminated by an external heat source.

5.4.4 Test process and measurement.

After a sufficient vacuum is attained ($< 10^{-6}$ hPa), and the temperatures of the shroud and sample holder are stabilized, the test piece is heated up to 30 °C. The decreasing temperature is then recorded down to 20 °C. The total hemispherical temperature is then calculated for a temperature of 25 °C. It is possible to do this for any temperature between –50 °C and +75 °C by using a similar method.

NOTE The errors of measurement depend mainly on the following quantities:

- specific heat of the test piece: C_e;
- temperature: T;
- time: t.

The first quantity, C_e, can be measured from the test piece by means of thermal analyses such as with a differential scanning calorimeter, or is taken from the literature for clearly defined materials.

5.4.5 Calculations of total hemispherical emittance

Calculation of the emittance is performed using the following formulas:

$$\varepsilon = \frac{(M_r Cr)_0 + (M_e C_e)_{Tmoy}}{4\sigma S T_0^3 (t_2 - t_1)} [(\alpha T_0) \ln A + \ln B + 2C]$$

$$A = \frac{\left(T_2^2 + T_0^2\right)\left(T_1^2 - T_0^2\right)}{\left(T_2^2 - T_0^2\right)\left(T_1^2 + T_0^2\right)}$$

$$B = \frac{(T_2 + T_0)(T_1 - T_0)}{(T_2 - T_0)(T_1 + T_0)}$$

$$C = (1 - \beta T_0^2)\left(\arctan\frac{T_2}{T_1} - \arctan\frac{T_2}{T_0}\right)$$

and

$$\varepsilon_e = \frac{\varepsilon S - \varepsilon_r S_r}{S_e}$$

where:

(t_1, T_1) and (t_2, T_2) are two points on a cooling curve at times t_1 and t_2 for which the corresponding temperatures are T_1 and T_2;

(MC) is the total heat capacity of a test piece with gold-plated substrate.

If the variation of specific heat is assumed to be parabolic with the temperature, then:

$$(MC)_T = (M_r C_r + M_e C_e)$$

and

$$(M_rC_r)_T = (M_rC_r)_0(1 + \alpha T + \beta T^2)$$

where:

ε is the total hemispherical emittance of test piece plus substrate;

ε_r is the total hemispherical emittance of the gold-plated substrate;

ε_e is the total hemispherical emittance of the test piece;

$(M_rC_r)_0$ is the thermal mass of the substrate at 0 °C (JK^{-1});

M_r is the weight of gold-plated piece (kg);

C_r is the specific heat of gold-plated piece (J kg K^{-1});

M_e is the weight of the test piece (kg);

C_e is the specific heat of the test piece (J kg K^{-1});

S_r is the gold-plated surface area (m^2);

S_e is the surface area of the test piece (m^2);

S is the total emitting surface area, i.e. $S = S_e + S_r$ (m^2);

T_0 is the temperature of the cryogenic shroud (K);

t is time (s);

σ is the Stefan-Boltzmann constant = $5,7 \times 10^{-8}$ W m^{-2} K^{-4}.

5.5 Infrared emittance using IR spectrometer (ε_h)

5.5.1 General

This method is based on optical measurements of absorptance of materials in the infrared range from 3 μm to 21 μm. This absorptance is determined measuring total hemispherical reflectance of these materials using an integrating sphere coupled with an infrared spectrometer.

As for solar absorptance measurements, one can make absolute measurements with a central sample holder, but another solution is to measure the reflectivity of the sample on the wall comparing the reflectivity of the sample with a calibrated standard (mirror or Infragold®). The spectrum obtained is weighted by blackbody spectrum and integrated.

5.5.2 Configuration of samples

The size of the samples depends on the configuration adopted (either central or tangential) and the sizes of the sphere, of the beam, of the sample holder and of the measurement port. The sample shall be large enough to receive the complete incident beam but small enough to disturb, at the minimum, the sphere integrity.

A classical size is 20 mm × 20 mm or 20 mm diameter for an integrating sphere of 150 mm in diameter with a central sample mounting, with a maximum thickness of 5 mm.

The samples shall be rigid.

5.5.3 Test apparatus and setting up

The test apparatus consists of a IR reflectometer covering the range from 3 μm to 21 μm equipped with an integrating sphere. The range of measurement is limited by the material used for the sphere walls (Infragold®).

a. The spectrometer shall be purged with a permanent dry air flux in order to eliminate CO_2 and H_2O absorption bands.

b. The signal to noise ratio over the whole interval from 2,5 μm to 20 μm shall be better than 1 % full scale.

5.5.4 Test process and measurement

The measurement on the sample is made after a baseline measurement is obtained either on the standard sample for a tangential sample, or on the wall sphere if the sample is centrally mounted.

The baseline and the sample measurement shall be made in the same conditions of the purge of the spectrometer.

5.5.5 Calculation of emittance

The spectrum obtained by the above test method is then weighted and integrating following the formula:

$$\varepsilon = \frac{\int\limits_{3\ \mu m}^{20\ \mu m} A(\lambda)E(\lambda)d(\lambda)}{\int\limits_{3\ \mu m}^{20\ \mu m} E(\lambda)d(\lambda)}$$

where:

$A(\lambda)$ is the spectral absorptance of the sample after 100 % reference correction ($A(\lambda) = 1 - R(\lambda)$ or $A(\lambda) = 1 - (R(\lambda) + T(\lambda))$ if the sample is transparent);

$E(\lambda)$ is the blackbody emittance spectrum at 300K and can be calculated with the Planck law:

$E(\lambda) = \dfrac{2\pi hc^2\lambda^{-5}}{e^{hc/\lambda kT} - 1}$ (W/m^{2}μm);

$h = 6{,}626 \times 10^{-34}$J s and $k = 1{,}381 \times 10^{-23}$ J K^{-1}.

The emittance at other temperatures can also be determined with measurements in another wavelength range. In this case, the blackbody spectrum shall be calculated at this new temperature.

5.6 Infrared emittance using portable equipment (ε_n)

5.6.1 General

This method is used to cover determination of the total normal emittance of opaque surfaces when using portable reflectometer instruments.

This test method is suitable for measuring over large surfaces where a non-destructive test is desired.

Depending on the equipment, the signal obtained is integrated over a defined spectral range (e.g. the "Gier Dunkle" DB--100 equipment is an infrared reflectometer and has an integration from 5 μm to 25 μm).

5.6.2 Configuration of samples

The minimal sample dimensions are dictated by the diameter of the aperture on the portable equipment. This diameter is typically between 15 mm and 20 mm.

Measurements are only valid on flat samples. However, it is possible to perform measurements on spherical curved samples, provided the radius of curvature exceeds 300 mm.

5.6.3 Test apparatus and setting up

The surface to be measured is placed against an aperture on the portable sensing component. The specimen is alternately irradiated with infrared radiation from

two heat sources, one at near ambient and the other at a slightly elevated temperature.

The detector receives both the radiation emitted from the test specimen and a constant radiation of all other surfaces inside the optical path. Only the reflected energy from the test specimen varies with the alternating irradiation and the detection amplifying system is made to respond only to this modulated signal.

The instrument shall be calibrated with standards of known emittance.

5.6.4 Test process and measurement

The measurement on the sample is made after calibrating the instrument with known reflectance standards.

The calibration shall be performed on at least two reference samples having low (black) and high (gold) reflectance properties and verified periodically by re-measuring the standards.

For semi-transparent samples, a correction shall be made for transmittance losses. Another possibility is to cover the semi-transparent sample with an opaque material from behind. In this case, the reflectance of this combination can be measured.

5.6.5 Calculation of the normal emittance

The normal emittance for opaque material is defined as:

$$\varepsilon_N = 1 - R_S \qquad\qquad (1)$$

$$I_D = R_S \sigma\, T_{IR}{}^4 \qquad\qquad (2)$$

where:

ε_N is the normal emittance;

R_S is the sample reflectance;

σ is the Stefan-Boltzmann constant;

T_{IR} is the infrared source temperature;

I_D is the energy seen by the detector.

By keeping the infrared source at a fixed temperature, the terms σ and T_{IR} become constant:

$$K = \sigma\, T_{IR}{}^4 \qquad\qquad (3)$$

with (2) and (3):

$$I_D = R_S\, K \qquad\qquad (4)$$

The sample reflectance R_S is proportional to the measured signal I_D. The normal emittance can be obtained by subtracting the measured reflectance from unity.

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6

Quality assurance

6.1 General

The quality assurance requirements are defined in ECSS-Q-20. Specific requirements are given in the following three subclauses.

6.2 Data

The quality records (e.g. log sheets) shall be retained for at least ten years or in accordance with project contract requirements, and contain 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 which shall be recorded;

e. the deduced results.

6.3 Nonconformance

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

6.4 Calibration

a. Each reference standard and piece of measuring equipment shall be calibrated.

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

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

6.5 Traceability

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

7

Audit of measurement equipment

7.1 General

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

The thermo-optical property data from test houses for the projects of the customer, obtained in the manner laid down in this Standard, are only accepted for the projects of the customer if the test house is certified to perform the relevant procedure in this Standard. The standard audit requirements are referred to in ECSS-Q-20.

7.2 Initial audit of the system (acceptance)

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

b. This initial audit shall, at least, consist of (but not necessarily be restricted to) an inspection of the apparatus and associated equipment, the performance of a test on a defined set of materials, the reporting of the nonconformances and the audit findings.

7.3 Annual regular review (maintenance) of the system

a. Inspection of apparatus and associated equipment.

b. Mutual comparability evaluation (testing).

c. Nonconformance:

If the inspection of the system or the "round robin test" shows a nonconformance with the applicable audit specification of the customer or the acceptable limits of the test results, actions shall be undertaken by the test house in order to determine the reasons for the nonconformance and a further test shall be performed in accordance with subclause 7.2 before a certificate of conformance is renewed.

d. Reporting of audit findings:

1. A detailed written report of the result of the regular review shall be delivered to all participants within six weeks after the end of the regular review or evaluation testing.

2. The certificate of conformance shall be renewed every three years after a successful audit.

7.4 Special review

a. All modifications of the apparatus or associated equipment shall be reported and, if deemed necessary, be audited by the customer before utilization of the modified system for the customer's project.

b. Major modifications shall result in the retesting of apparatus as described in subclause 7.2.

Bibliography

ASTM E 490	*Standard Solar Constant and Zero Air Mass Solar Spectral Irradiance Tables*
ECSS-Q-20	*Space product assurance — Quality assurance*
ECSS-Q-20-09	*Space product assurance — Nonconformance control system*
ECSS-Q-40	*Space product assurance — Safety*

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ECSS Document Improvement Proposal

1. Document I.D. ECSS-Q-70-09A	2. Document date 29 August 2003	3. Document title Measurements of thermo-optical properties of thermal control materials

4. Recommended improvement (identify clauses, subclauses and include modified text or graphic, attach pages as necessary)

5. Reason for recommendation

6. Originator of recommendation

Name:	Organization:	
Address:	Phone: Fax: e-mail:	**7. Date of submission:**

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Note: The originator of the submission should complete items 4, 5, 6 and 7.

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