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EM41

MSFC TECHNICAL STANDARD

OUTGASSING TEST FOR
NONMETALLIC
MATERIALS ASSOCIATED
WITH SENSITIVE OPTICAL
SURFACES IN A SPACE
ENVIRONMENT

CHECK THE MASTER LIST—
VERIFY THAT THIS IS THE CORRECT VERSION BEFORE USE

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1. PURPOSE

The purpose of this Specification is to supplement the Standard Test Method for Total Mass Loss (TML) and Collected Volatile Condensable Materials (CVCM) from Outgassing in a Vacuum Environment (ASTM-E595) by establishing outgassing requirements and test guidelines for non-metallic materials used in the space thermal/vacuum environment in the vicinity of sensitive optical surfaces.

1.1 Scope

This Specification presents criteria for the evaluation of materials and defines the test method to be used to evaluate these materials. Programs are encouraged to develop criteria, specific to the needs of the Program, that insure that the optical surface functions at the level established by the Program.

This standard applies the following: All mandatory actions (i.e., requirements) are denoted by statements containing the term, “shall.” The terms: “may” or “can” denote discretionary privilege or permission; “should” denotes a good practice and is recommended, but not required; “will” denotes an expected outcome; and “are/is” denotes descriptive material.

2. APPLICABLE DOCUMENTS

The documents listed below provide requirements, specifications, standards, and procedures applicable to this standard to the extent specified herein. For each of these documents, the latest revision in effect at the time of document approval shall apply.

2.1 Government Documents

EM40-OWI-094 Operation of Vacuum Ultraviolet Reflectometer

2.2 Non-Government Documents

ASTM-E595 Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment

3. REQUIREMENTS

The allowable degradation in performance, of an optical surface, shall be established by the Program responsible for the optical surface or the system in which the optical surface is intended to function. Criteria for an acceptable level of molecular deposition on that surface or

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degradation in performance will be used to determine the acceptability of a material, as measured by this method, and evaluated against the Program establish criteria.

Materials used adjacent to or with a line of sight to an optical surface will also be screened by this method and evaluated using the Program established criteria.

A suggested guideline for accepting a material is:

The material shall have a maximum total mass loss (TML) of 1.0% of the original sample mass and a maximum collected volatile condensable material (CVCM) deposition of 0.1% of the original sample mass. The reflectance of an optical witness sample (OWS), a magnesium fluoride coated aluminum, first surface mirror, shall be within the allowable limits of vacuum ultraviolet (VUV, 121.6 nm – 200.0 nm) reflectance change (3.0% maximum decrease in $\Delta R/R_0$) when tested in accordance with the test procedure in Paragraph 6.

4. SELECTION AND VERIFICATION REQUIREMENTS

Program specific criteria for nonmetallic materials should be established to insure that the nonmetallic materials selected for the construction of optical instruments, or to be used adjacent to those instruments, do not degrade the performance of the instruments optical surfaces beyond a program determined limit

a. The material may be brought within vacuum stability limits by vacuum baking for a specified period of time (usually 48-hours at maximum) that the vacuum baked material meets the Program criteria for TML, CVCM, and $\Delta R/R_0$

b. If the TML and CVCM are less than or equal to the Program defined limits and it can be shown that contributions to the TML greater than the Program defined limit are due to absorbed water vapor, WVR, (See Paragraph 8) the material may be used if optical requirements are met.

c. The total mass of materials selected under Paragraph 4b above, requiring a Material Usage Agreement (MUA) and used in any given compartment, shall be monitored and reviewed by the Program, at Program defined intervals, to insure that optics and contamination sensitive surfaces are not degraded by the use of those materials. The Program shall define the criteria for acceptable/unacceptable levels of contamination at the beginning of the Program, prior to the use of any material.

d. Materials selected under Paragraph 4 c above shall be reviewed per a schedule determined by the Program to insure that problems related to the use of those materials do not evolve during the life of the Program.

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5. IMPLEMENTATION

The contractor/hardware developer shall provide, for approval, by the Program, a list of all materials selected for use in the vicinity of sensitive optical surfaces, with a line-of-sight to a contamination sensitive surface, or in the same optical volume (defined compartment) as sensitive optical surfaces.

The following is required:

- a. Material manufacture
- b. Manufacture's trade name and part number
- c. Maximum use temperature
- d. Thermal/vacuum stability (CVCM and TML) data.
- e. Temperature of optics in the vicinity, with a line-of-sight to, or in a defined compartment with the material.
- f. Rational for the use of the material that failed the requirements of Paragraph 5.0 and a report of the weight and surface area used.
- g. Quantity and surface area of the material to be used.

6. MATERIAL TESTING

6.1 Purpose

The purpose of this test is to measure total mass loss (TML), collected volatile condensable materials (CVCM), and reflectance change of an optical witness sample (OWS) due to outgassing contaminants under controlled laboratory conditions.

6.2 Test Conditions

The test on non-metallic materials shall be conducted under the following conditions:

Temperature of Specimen	Normally 125 degrees Celsius (°C) but for special cases, at least 10 degrees C above maximum use temperature (+/- 1 degree °C). (ASTM-E595)
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Temperature of Collector 25 degrees C or the temperature of the optics exposed to the Plates or OWS material being evaluated (the lesser temperature (+/- 1 degree C). The measurement can also be made at a temperature that is colder (typically 10 degrees C colder) than the optics if a conservative margin will insure that the material will not degrade the performance of the optics. This test configuration is not meant to evaluate samples in the cryogenic temperature range.

Pressure 1×10^{-5} Torr

Vacuum Exposure Time 24-hours

6.3 Test Equipment

All laboratory test instrumentation shall be in current calibration and shall reflect appropriate documentation from the applicable calibration laboratory. The test apparatus shall conform to critical dimensions of Table 1 (test apparatus dimensions) and Figure 2 of ASTM-E595. A minimum of three samples, two collector plates, and one OWS shall be included in each test.

Test equipment shall consist of the following:

- a. A vacuum system capable of maintaining the required vacuum level for a period of 24 hours.
- b. Specimen holder made of aluminum foil.
- c. Collector plate made of highly polished stable metal surface.
- d. Optical witness sample holder made of aluminum and capable of holding a 2.54-cm diameter mirror.
- e. A test apparatus made of copper. The apparatus shall be capable of accommodating a minimum of three samples in the heater bar, and two collector plates and one OWS holder on the cooling plate. The heater bar and cooling plate shall be capable of maintaining the samples, collector plates, and OWS, respectively, at their appropriate temperatures +/- 1 degree C.
- f. A balance having +/- 1 microgram sensitivity for weighing samples and collector plates.
- g. A reflectometer capable of making vacuum UV specular measurements over the 121.6-nm to 200.0-nm wavelength range per EM40-OWI-094.

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6.4 Cleaning Procedures

Cleaning and storage procedures shall be found in Annex 1 of ASTM-E595.

6.5 Sample Preparation:

NOTE: Specimen handling procedures outlined in Paragraph 7.7 of ASTM-E595 shall be followed.

6.5.1 Specimen Size

Material to be tested shall be prepared in 100 milligram to 300 milligram specimen sizes and placed in aluminum holders after preparation as specified below.

6.5.2 Solid Material

Specimen shall be cut into small pieces having 6 millimeter maximum dimension. Sample materials (coatings or tapes) applied to a substrate, aluminum foil or Teflon sheet, for testing shall be applied to a substrate with known density so that actual sample material weight may be determined. Sample shall be placed in a desiccator after preparation and remain there until the samples are placed in the test chamber.

6.5.3 Coatings

Materials that are normally used as coatings shall be applied to aluminum foil or Teflon sheet with a known density and prepared as noted in Paragraph 6.5.2.

6.5.4 Solvent Containing Materials

Prior to testing solvent containing materials, such as inks and paints or room temperature cured materials, the sample shall be pre-conditioned for 24-hours at 65 degrees C (+/- 1 degree C) in an air circulating oven to simulate the material exposure up to the time of launch.

6.5.5 Tapes

Tapes shall be tested in the as applied configuration using aluminum foil or Teflon sheet with known density as an application substrate and prepared in accordance with Paragraph 6.5.2. Actual weight of adhesive on the tape should be known. Total edge exposure length during the test shall be reported (See Paragraph 10).

6.5.6 Liquids

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Liquids shall be tested in the as received condition.

6.5.7 Cure Procedures

All materials shall be cured or applied in accordance with the manufacture's procedures or the applicable contractor process specification prior to test.

7. TEST PROCEDURES

Test procedures outlined in Paragraph 8 of ASTM-E595 shall be followed except for the sample temperature and cooling plate temperature which will be determined by procedure previously defined in Paragraph 6.2 of this document.

7.1 TML, CVCM, WVR, and Optical Reflectance Measurements

NOTE: All weightings shall be made on a balance having +/- 1 microgram sensitivity.

7.1.1 Initial Mass Determination

The CVCM collector plates and specimen holders shall be weighed prior to testing. All weights shall be recorded.

7.2 Initial Optical Reflectance Measurement.

The VUV specular reflectance [near-normal incidence (≤ 20 degrees)] of the OWS to go in test along with a control OWS shall be measured over 121.6 nm to 200.0 nm wavelength range. The initial reflectance measurement (R_{i0} , Paragraph 9) shall be made using a clean OWS prior to deposition of any material on the OWS. For non-continuous scanning reflectometers, reflectance measurements shall be recorded at 121.6, 125.0, 130.0-nm and 10.0nm wavelength increments from 130.0 nm thru 200.0-nm. The reflectometers and instrumentation shall be capable of +/- 1% repeatability in measure specular reflectance, consistently through the period of use. All measurements shall be recorded.

7.3 Final Mass Determination

The specimen and collector plates shall be weighed as soon as possible after removal from the CVCM apparatus. Specimen shall then be returned to the desiccator for 24-hours. After 24-hours the specimen shall be weighed again to determine the amount of water vapor regained (WVR). All weightings shall be recorded.

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7.3.1 Final Optical Reflectance Measurements

The vacuum UV specular, near-normal incidence reflectance of the test over the 121.6-nm to 200.0-nm wavelength range shall be measured. All measurements shall be recorded.

8. TEST RESULTS

Value for TML and CVCM shall be calculated per ASTM-E595, Paragraph 9 (Calculations). Optical reflectance data shall be tabulated for quick reference.

9. CRITERIA OF ACCEPTABILITY

In the absence of Program defined criteria, the collected volatile condensable material content of the material shall not exceed 0.1% of the original mass of the sample. The total mass loss of the material shall not exceed 1.0% of the original mass of the sample. The total water vapor regained mass shall not exceed 1.0% of the original mass of the sample. Upon post-test vacuum UV reflectance analysis, the maximum reduction in reflectance of the OWS, referenced to the pre-test value and normalized to the control OWS value, shall not exceed a 3% decrease at any one wavelength over the 121.6 nm to 200.0 nm wavelength range. If the above values are exceeded refer to Paragraph 4.

An increase in the reflectance of the OWS will result in the rejection of the reflectance measurement data, requiring the test to be repeated with a clean OWS.

The percentage change in reflectance ($\Delta R\%$, referred to as $\Delta R/R_o\%$ in this document) is calculated for each wavelength, as:

$$\Delta R/R \% = (((R_{co}/R_{cf}) R_{tf}] - R_{to})/R_{to}) \times 100$$

Where:

R_{co} = Pre-test reflectance of control OWS

R_{cf} = Post-test reflectance of control OWS

R_{to} = Pre-test reflectance of test OWS

R_{tf} = Post-test reflectance of test OWS

Note: In the calculation, the post exposure OWS measurement is normalized to the control reflectance measurements. Then the percent change in reflectance is calculated using the normalized, post exposure change in reflectance and the pre-exposure reflectance measurement.

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The calculation can also be applied to additional wavelengths in the range of 121.6 nm to 200.0 nm to meet the needs of a specific application.

10. TEST REPORT

The report of results from each test shall include the following:

- (1) Total mass Loss (TML)
- (2) Collectable volatile condensable materials (CVCM)
- (3) Water vapor regained (WVR)
- (4) $\Delta R/R_0$ maximum and corresponding wavelength
- (5) Sample test temperature
- (6) Optical witness sample (OWS) test temperature
- (7) Sample cure conditions if applicable
- (8) Sample cleaning procedure if applicable
- (9) Length of edge exposure for tape sample
- (10) Material acceptability determination made per Paragraph 9.0
- (11) Plans for further testing if applicable
- (12) A statement of any other relevant items which might be deemed important.

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APPENDIX A. ACRONYMS

ASTM	American Society for Testing and Materials
C	Celsius
CVCM	Collected Volatile Condensable materials
MUA	Materials Usage Agreement
nm	nanometer
OWS	Optical Witness Sample
TML	Total Mass Loss
UV	Ultraviolet
VUV	Vacuum Ultraviolet
WVR	Water vapor regained