National Aeronautics and
Space Administration

METRIC

MSFC-SPEC-2223
REVISION A
EFFECTIVE DATE: June 29, 2007

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

EM50

OUTGASSING TEST FOR MATERIALS ASSOCIATED WITH SENSITIVE SURFACES USED IN AN AMBIENT ENVIRONMENT

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

DOCUMENT HISTORY LOG

Status (Baseline/ Revision/ Canceled)	Document Révision	Effective Date	Description
Revision	A	TBD	General Rewrite

1. PURPOSE

This specification covers the control of materials used in air volumes of controlled environments. These environments may be clean rooms, environmental chambers, or shipping containers. This document establishes requirements and defines test methods to evaluate these materials. This test method is primarily a material screening technique and is not necessarily valid for computing actual contamination on a system or component. The use of materials deemed acceptable in accordance with this test method does not ensure the system or component will remain uncontaminated. Contributing factors not addressed in this test which should be considered include quantity of material used, line-of-sight to sensitive surfaces, and hardware exposure environments, e.g. process cleanliness, ect. Therefore, subsequent functional, developmental, and qualification tests should be used as necessary to ensure material's satisfactory performance.

The purpose of this specification is to present the specification for materials that are used in ambient environment at temperatures of 40 degrees Celsius (C) or less and associated with contamination sensitive surfaces. These surfaces may include but are not limited to optics, windows, thermal "control" or "protective" surfaces.

2. APPLICABLE DOCUMENTS

N/A

3. REQUIREMENTS

Materials used in an air volume with any sensitive surface shall not produce volatile condensable products when heated to 10 C (18 degrees Fahrenheit (F) above that material's intended use temperature. These products shall not adhere to an optical witness sample (OWS) cooled to a temperature equal to the sensitive surface's use temperature. The OWS for the standard test is a magnesium fluoride coated aluminum first surface mirror. Material failure is defined as decrease in reflectance of greater than 3.0% when tested in accordance with the test procedure of Paragraph 8. An increase in OWS reflectance will constitute a failure of the measurement, requiring the measurement to be repeated with a clean OWS.

4. IMPLEMENTATION

The following information shall be provided with the sample:

a. Material Manufacture

b. Manufactures trade name

c. Specification

d. Composition

e. Cure parameters

f. Maximum use temperature required Equipment:

g. Temperature of sensitive surfaces of concern in the vicinity of the material.

h. Other information pertinent to the traceability and understanding of the use of this material.

5. MATERIAL TESTING

a. Purpose:

The purpose of this test is to determine the presence of volatile condensable material by measuring a change in the reflectance of an OWS. This test is conducted under controlled laboratory conditions at ambient pressure.

b. Test Conditions:

Material tests shall be preformed under the following conditions:

a. Specimen Temperature	10 degrees C (18 degree F) above maximum use temperature, +/- 1 degree C
b. OWS Temperature	Optic temperature to which the material is exposed, +/- 1 degree C

NOTE: In selecting the temperatures one must consider the dew point for moisture. Temperatures should be chosen to preclude moisture condensation.

c. Pressure	Ambient

d. Exposure time 7-days

c. Test Equipment:

All test instruments shall be in calibration. All test equipment shall be those listed or the equivalent. These equipment and materials shall consist of the following:

Required Equipment

a. Blue M Powermatic 70 vacuum Oven

b. Mechanical vacuum pump with cryotrap (required only for cleaning).

c. Brinkman RM20 cooling bath

d. Fluke 217A multichannel digital thermometer.

e. LN2 timer

Required Materials:

a. 200 Proof ethanol

b. Lint free, solvent compatible cleaning cloth/wipe

c. Contamination free solvent compatible gloves

d. Aluminum sample pan, cleaned and baked

e. Aluminum-magnesium fluoride coated first surface mirror optical witness sample (OWS), with a diameter of 1-inch or greater, and holder.

6. OVEN CLEANING PROCEDURE AND CERTIFICATION:

6.1 Clean all surfaces and fixtures in the vacuum oven using ethanol and lint free cloth while wearing gloves.

6.2 Close oven and bleed valves

6.3 Turn on vacuum pump

6.4 Fill cryotrap with LN2 and set LN2 timer

6.5 Open vacuum valve

6.6 Set temperature to approximately 150 degrees C (302 degrees F)

6.7 Allow system to bake for 24-hours

NOTE: Monitor the cryotrap to insure it does not run dry. Contamination of the oven will occur due to back streaming vacuum pump oil.

6.8 After 24-hours, turn off heat and allow oven to cool to ambient temperature.

6.9 Close vacuum oven valve and open bleed valve.

6.10 Turn off LN2 timer

6.11 Oven Certification Procedure

6.12 After bakeout, mount OWS in holder to be cooled by circulation bath.

6.13 Attach thermocouple to mirror holder

6.14 Place the sample pan in a direct line of sight to the OWS.

NOTE: OWS Surface must be exposed face downward in line of sight of test sample pan.

6.15 Attach thermocouple to sample pan

6.16 Close oven
6.17 Set oven temperature to test temperature

6.18 Turn on oven heat

6.19 Turn on cooling bath

6.20 Set cooling bath to desired test temperature

6.21 Make necessary adjustments to oven temperature and cooling bath to obtain the desired temperatures

6.22 Allow oven to operate for 24-hours

6.23 After 24-hours, turn off heat and raise the cooling bath temperature to slightly less than the ambient temperature.

6.24 Allow oven temperature to fall below 38 degrees C (100 degrees F) and cooling bath to reach a temperature slightly less than ambient before removing the OWS.

7.0 OPTICAL ANALYSIS OF THE OWS:

7.1 Visually analyze the OWS for surface contamination. If a visible film is present, measure the sample using ellipsometry spectroscopy or other surface analysis techniques as deemed appropriate.

7.2 If no visible contamination is present, perform vacuum ultraviolet (VUV) reflectance measurement over the wavelength range of 121.6-nm to 200.0-nm for a standard test. The acceptance criterion is a loss of reflectance no greater than 3% at each wavelength. An increase in the reflectance at any wavelength will require the test to be repeated with a clean OWS.

7.3 If the OWS fails, repeat oven cleaning bakeout and certification procedures until acceptable results are obtained.

7.4 The posttest optical measurements of a first surface mirror (OWS) are evaluated using the acceptance criteria of a maximum 3% or less loss in reflectance, at any one measured wavelength, over the VUV (121.6nm to 200.0 nm) wavelength range. A control mirror is measured with each test mirror to verify instrument repeatability of +/-1.0%. 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.

8. MATERIAL TEST:

8.1 Wipe test sample with ethanol if sample lends itself to wiping and is compatible with solvent; if not, clean as appropriate.

8.2 Place sample in aluminum sample pan

8.3 Clamp a thermocouple to the material, if possible, otherwise attach it to the sample pan.

8.4 Mount OWS in holder to be cooled by circulating water bath

8.5 Attach thermal couple to mirror holder

8.6 Position the sample pan so that a direct line of sight exists between the sample and the surface of the OWS. The distance between the sample and the OWS shall be no greater than 3.0 centimeters. This distance must be reported along with other pertinent test parameters

8.7 Close oven

8.8 Set oven temperature to test temperature

8.9 Turn on oven heat.

8.10 Turn on cooling bath.

8.11 Set cooling bath to desired temperature.

8.12 Make necessary adjustments to oven temperature and cooling bath to obtain the desired temperatures.

8.13 Allow to operate for 24-hours.

8.14 After 24-hours, turn off heat and raise the cooling bath temperature to ambient temperature.

8.15 Allow oven temperature to fall to below 38 degrees C (100 degrees F) and cooling bath to reach a temperature slightly below ambient before removing the OWS.

8.16 Remove the OWS from the oven and perform optical test specified in paragraph 8.0.

8.17 If acceptable, repeat steps 8.4 through 8.12 then proceed to paragraph 8.18.

8.18 Continue test for 6 additional days.

8.19 After 7 days total exposure turn off heat and increase cooling bath temperature to a temperature slightly below ambient.

8.20 Allow oven temperature to decrease below 38 degrees C (100 degrees F) and cooling bath to reach a temperature slightly below ambient before removing OWS.

8.21 Remove OWS from oven for optical analysis.

8.22 Repeat paragraphs 7.1 and 7.2 of Optical Analysis Paragraph.

9. CALCULATION OF PERCENT REFLECTANCE

Calculate the percent change in reflectance for the following wavelengths, 121.6, 125.0, 130.0, 140.0, 160.0, 180.0, and 200.0nm, using the following formula.

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

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

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.
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. PASS/FAIL CRITERIA

There shall be no visible film on the OWS and the OWS Shall pass the VUV reflectance analysis**.** Material failure is defined as loss of reflectance greater than 3% when tested in accordance with the test procedure of Paragraph 7. The OWS reflectance, referenced to the pre-test reflectance and normalized to the control, shall not decrease by more than 3% over the 121.6-nm to 200.0-nm wavelength range. The percentage change in reflectance is calculated for each wavelength, 121.6, 125.0, 130.0, 140.0, 160.0, 180.0, and 200.0-nm per Paragraph 9. An increase in the reflectance at any wavelength will require the test to be repeated with a clean OWS.

11. TEST REPORT

The test report shall include the following:

a. Results of the OWS analysis.

b. Sample test temperature.

c. Optical witness sample (OWS) test temperature.

d. Sample cure condition if applicable.

e. Sample cleaning procedure if applicable.

f. Total edge exposure for tape samples.

g. Material acceptability.

h. Sample/material name and source (vendor)

i. Sample size

j. Wavelengths measured

k. Analyst and date of test

12. ACRONYMS

C	Celsius
F	Fahrenheit
LN_2	Liquid Nitrogen
OWS	Optical Witness Sample
VUV	Vacuum Ultraviolet