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George C. Marshall Space Flight Center
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ED31

MULTIPROGRAM/PROJECT COMMON-USE DOCUMENT

OUTGASSING TEST PROCEDURE FOR NON-METALLIC MATERIALS ASSOCIATED WITH CONTAMINATION SENSITIVE SURFACES IN A THERMAL VACUUM ENVIRONMENT

STANDARD OPERATING PROCEDURE

NOTICE: This document is not intended to be a substitute for adequate training on this system.

DOCUMENT HISTORY LOG

Status (Baseline/ Revision/ Canceled)	Document Revision	Effective Date	Description
Baseline		06/13/2002	Baseline release

TABLE OF CONTENTS

GENERAL OPERATIONAL CONCERNS

Before engaging in any task, operators should first be familiar with all associated documents, guidelines and procedures referenced in this document. Likewise, they should be trained and qualified by approved procedures in ED31-OWI-001 “Personnel Training and Certification.”

Always use appropriate protective equipment (gloves, eye wear, etc.) during cleaning and servicing of test components. All appropriate measures should be employed to minimize introduction of contaminants into chamber. Always wear clean, approved gloves when working inside chamber or handling any object that is to be placed in chamber.

Prior to any test or operation of the vacuum system, inspect all components for proper configuration, loose fittings or fasteners, coolant leaks, fluid level in bath, electrical and instrumentation connections, proper pressure settings on pneumatic systems, and any potential unsafe conditions.

Verify that all necessary equipment is operational. Compare current equipment ECN’s to prior test data sheet to determine if any changes have been made to the equipment configuration. Note all equipment changes on new data sheet and file in VCMO Lab notebook. Verify calibration is current for all critical measurement devices.

Should a component fail during any operational phase, the risk to personnel and the test should be determined and appropriate action taken. Any potential safety hazard should lead to an immediate termination of testing, repressurization of chamber, and correction of the hazard.

WARNING: Care should always be given to back-fill of the chamber. When using a pressurized gas source (GN_2, Missile Grade Air, etc.) always use the minimum pressure possible on regulator and monitor pressure gauges. Although the primary test chamber has an overpressure relief valve, failure to employ these measures could lead to an over-pressurization of the chamber causing damage to test samples, instrumentation, chamber hardware or an explosion.

WARNING: The primary test chamber in Building 4711 room E179B has electrical power fed from two independent sources. The main power feed comes from the UPS system located in E114. This powers all equipment that is necessary for routine testing chamber requirements. The secondary power supply is exclusively for the bell jar shroud bakeout heaters. Any time access panels are to be removed or any electrical work is to be done, verify that BOTH power sources are disconnected and follow proper lockout/tagout procedures.

1. SCOPE/PURPOSE

The purpose of this procedure is to define the operational test method to support MSFC-SPEC-1443 “Outgassing Test for Non-Metallic Materials Associated with Contamination Sensitive Surfaces in a Thermal Vacuum Environment.” This specification was written to supplement JSC-SP-R-0022 “Specification for Vacuum Stability Requirements of Polymeric Material for Spacecraft Application” since it was found that materials could meet the requirements of JSC-SP-R-0022 but still contaminate sensitive surfaces to a point that they become non-functional.

The scope of this procedure is limited to the VCMO test chamber (ECN 1440934) and associated support hardware and instrumentation located in Building 4711 room E179B. Operators are expected to be familiar with ASTM E 595, MSFC-SPEC-1443 and all current governing ISO documents before proceeding with testing.

2. APPLICABLE DOCUMENTS

Documents listed below provide requirements, specifications, standards, and procedures applicable to this procedure. For each of these documents, the latest revision in effect at time of document approval will apply.

ASTM E 595	Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment
ED31-OWI-001	Personnel Training and Certification
ED31-OWI-002	Documentation and Data Control
ED31-OWI-003	Control of Quality Records
JSC-SP-R-0022	Specification for Vacuum Stability Requirements of Polymeric Material for Spacecraft Application
MPG 8730.5	Control of Inspection, Measuring, and Test Equipment
MSFC-SPEC-1443	Outgassing Test for Non-Metallic Materials Associated with Sensitive Optical Surfaces in a Space Environment

3. DEFINITIONS/ACRONYMS

The following definitions relate to terms applicable to this procedure:

Collected Volatile Condensable Material (CVCM): quantity of outgassed matter from a test specimen that condenses on a collector maintained at a specific, constant temperature for a specified time. CVCM is expressed as a percentage of initial specimen mass and is calculated from condensate mass determined from the difference in mass of collector plates before and after test.

Collector Plate: a cooled sample plate on which outgassing vapors condense. The weight of condensed material is used for calculating CVCM. Plates for the standard test are chromium-plated aluminum, but other materials which better simulate surfaces of interest are optional.

Contamination Sensitive Surfaces: any surface of flight hardware that could be adversely affected by contamination, e.g. mirrored optics, windows, detectors, thermal control surfaces, etc.

Contamination Witness Sample (CWS): a test specimen, e.g. OWS, which represents contamination sensitive elements critical to performance of flight instrumentation. The CWS must be defined to accurately represent contamination critical elements or components.

Optical Witness Sample (OWS): a CWS that represents the contamination sensitive optical elements critical to instrument performance. In the standard test the OWS is a one-inch diameter, 1/8 inch thick fused silica substrate polished to at least 0.1 wave at 546.1 nm. Substrates are coated with aluminum then a protective magnesium fluoride (MgF_2) overcoat to optimize reflectance at 121.6 nm. Minimum reflectance is required to be 70% from 120.0 nm to 200.0 nm.

Total Mass Loss (TML): total mass of material outgassed from a specimen that is maintained at a specified constant temperature and operating pressure for a specified time. TML is calculated from the specimen mass as measured before and after test and is expressed as a percentage of the initial specimen mass.

Water Vapor Regained (WVR): mass of water vapor regained by a specimen after the optional reconditioning step. WVR is calculated from the differences in specimen mass determined after test for TML and again after exposure to a 50±5% relative humidity environment at 23±2°C for 24 hours. WVR is expressed as a percentage of initial specimen mass.

The following are acronyms commonly used in this and associated documents:

ASTM - American Society for Testing and Materials

ECN - Equipment Control Number

GN_2 - Gaseous Nitrogen

ISO - International Standards Organization

JSC - Johnson Space Center

MSFC - Marshall Space Flight Center

SP - Set point

TC - Thermocouple

UPS - Uninterruptible Power Supply

VCMO - Volatile Condensable Material for Optics

4. CHAMBER OPERATION

Vacuum pumping system components are “dry” or oil free pumps, so there is no threat of backstreaming molecular contamination into chamber from pumps. Chamber rough pressure is monitored by two TC gauges mounted on the bell collar. Another TC gauge is on the foreline of the roughing pump to monitor the turbo pump backing pressure. High vacuum is measured with a nude ion gauge mounted on the bell collar. A liquid feedthrough is mounted on the collar for the circulating bath ethylene glycol mix that maintains the temperature of the collector base. Cartridge heater power is delivered to the copper sample towers via an electrical feedthrough, and a separate instrumentation feedthrough provides thermocouple connections to the two test towers. The “hot” and “cold” sides of each test tower have two thermocouples each that are averaged by the control software and displayed on the computer screen.

4.1 CHAMBER BAKEOUT FOR SEVERE CONTAMINATION

If gross contamination of chamber and test towers is suspected, a system bakeout must be performed per the following:

Verify that the power strip to the right of computer is switched OFF. Turn on system computer and monitor. When boot is complete, control software should automatically run. Activate controls by going to the *Panel* pull down menu and click on *Start Task.*

1. Open vent valve and using TC gauge #1 verify that chamber pressure is at ambient.
2. Using hoist, raise bell to provide access to test towers.
3. Follow prescribed cleaning guidelines in ASTM E 595 sections A1.1.4 through A1.1.8 and reinstall components.
4. Prepare towers for bakeout by verifying that specimen containers are empty, CWS are removed, thermocouples are secure, and heaters are correctly mounted.
5. Verify that o-ring seal is correctly in place and free from particulate contamination.
6. Slowly lower bell with hoist until bell is completely seated on collar and tension is released from hoist cable.
7. Close vent valve and verify that backfill valve is closed.
8. Open gate valve and turn on roughing pump.
9. When TC gauge #1 reads 1 torr press turbo pump start button.
10. When turbo pump controller shows pump has reached normal operation, select BA gauge #1 and turn on emission.
11. Turn on circulating bath unit and set temperature to 100°C. Be sure to turn the refrigeration switch to OFF.
12. On the chamber front control panel, activate bell heaters by turning on the switch labeled “HEATER ON/OFF.” For each of the controllers, raise SP to 75°C. As the heaters reach the setpoint, continue increasing SP by 20-30°C until bell heater setpoints are 150°C.
13. Meanwhile, as the bell is heating up, activate the tower heaters by entering the set point for each at 150°C, click on the OFF/ON virtual switch for each tower to ON, and turn on the power strip to the right of the computer.
14. Once all devices reach their ultimate SP, leave all heaters on for 24 hours. Vacuum level should slowly drop to the low to mid 10^{-7} torr range.
15. After the 24-hour bakeout, lower all heater SP settings to 20°C. On the circulating bath, keep SP unchanged until heater temps. drop below 50°C. Then lower bath SP to 20°C and turn refrigeration switch to ON.
16. When all temperatures within chamber and on bell have reached ambient temperature, turn OFF all bell heaters, turn OFF power strip and virtual switches for tower heaters, and turn off circulating bath.
17. Turn OFF BA #1 gauge by pressing the EMIS button and select TC #1 gauge.
18. Turn OFF turbo and roughing pumps.
19. Open valve on Nitrogen K-bottle. Regulator should be set for minimal flow, and low-pressure needle should be at lowest point. You should hear a slight hiss at the backfill valve.
20. Slowly open backfill valve until chamber begins to repressurize. As pressure increases, open backfill valve a little more if necessary.

21. Once chamber has returned to atmospheric pressure, close backfill valve and close K-bottle valve.
22. Close gate valve.
23. Open vent valve.
24. Raise bell with hoist when ready to certify chamber.

4.2 CHAMBER BAKEOUT FOR ROUTINE TESTING

Between each sample run, the test chamber must be cleaned per the following procedure with these exceptions: First, OWS must meet acceptance criteria from previous test run. Second, temperature profiles of next sample test must be the same as or lower than the previous test. Third, no more than a week has passed between sample runs.

1. Verify that the power strip to the right of computer is switched OFF. Turn on system computer and monitor. When boot is complete, control software should automatically run. Activate controls by going to the *Panel* pull down menu and click on *Start Task.*
2. Open vent valve and using TC gauge #1 verify that chamber pressure is at ambient.
3. Using hoist, raise bell to provide access to test towers.
4. Follow prescribed cleaning guidelines in ASTM E 595 sections A1.1.4 through A1.1.8 and reinstall components.
5. Prepare towers for bakeout by verifying that specimen containers are empty, CWS are removed, thermocouples are secure, and heaters are correctly mounted.
6. Verify that o-ring seal is correctly in place and free from particulate contamination.
7. Slowly lower bell with hoist until bell is completely seated on collar and tension is released from hoist cable.
8. Close vent valve and verify that backfill valve is closed.
9. Open gate valve and turn on roughing pump.
10. When TC gauge #1 reads 1 torr press turbo pump start button.
11. When turbo pump controller shows pump has reached normal operation, select BA gauge #1 and turn on emission.
12. Turn on circulating bath unit and set temperature to 50°C. Be sure to turn the refrigeration switch to OFF.
13. Activate the tower heaters by entering the set point for each at 150°C, click on the OFF/ON virtual switch for each tower to ON, and turn on the power strip to the right of the computer.
14. Once all devices reach their ultimate SP, leave all heaters on for 4 hours, minimum.

15. After the 4-hour bakeout, lower all heater SP settings to 20°C. On the circulating bath, keep SP unchanged until heater temps. fall 10°C below current bath SP. Then lower bath SP to 20°C and turn refrigeration switch to ON.
16. When all temperatures within chamber have reached ambient temperature, turn OFF all heaters, turn OFF power strip and virtual switches for tower heaters, and turn off circulating bath.
17. Turn OFF BA #1 gauge by pressing the EMIS button and select TC #1 gauge.
18. Turn OFF turbo and roughing pumps.
19. Open valve on Nitrogen K-bottle. Regulator should be set for minimal flow, and low-pressure needle should be at lowest point. You should hear a slight hiss at the backfill valve.
20. Slowly open backfill valve until chamber begins to repressurize. As pressure increases, open backfill valve a little more if necessary.
21. Once chamber has returned to atmospheric pressure, close backfill valve and close K-bottle valve.
22. Close gate valve.
23. Open vent valve.
24. Raise bell with hoist when ready to certify chamber.

4.3 **CHAMBER CERTIFICATION**

Before sample testing takes place, the chamber must be certified that it meets cleanliness requirements per the following:

1. Verify that the power strip to the right of computer is switched OFF. Turn on system computer and monitor. When boot is complete, control software should automatically run. Activate controls by going to the *Panel* pull down menu and click on *Start Task.*
2. Open vent valve and using TC gauge #1 verify that chamber pressure is at ambient.
3. Using hoist, raise bell to provide access to test towers.
4. Prepare towers for certification by verifying that specimen containers are empty, no collector plates are installed, and thermocouples are secure.
5. Obtain a pre-certified OWS, and mount in a clean OWS holder.
6. Install OWS in chamber in one of the collector plate positions in the tower that is to be used for testing.
7. Repeat steps 5 & 6 for other tower, if both are to be used.
8. Install separator plate and heater bar with cover plates on tower.
9. Verify that correct cartridge heaters are installed in appropriate heater bar.
10. Verify that o-ring seal is correctly in place and free from particulate contamination.
11. Slowly lower bell with hoist until bell is completely seated on collar and tension is released from hoist cable.
12. Close vent valve and verify that backfill valve is closed.

13. Open gate valve and turn on roughing pump.
14. Turn on circulating bath unit and set temperature to predetermined collector plate temperature for upcoming sample test. Be sure that the refrigeration switch is ON.
15. When TC gauge #1 reads 1 torr press turbo pump start button.
16. When turbo pump controller shows pump has reached normal operation, select BA gauge #1 and turn on emission. Hold until pressure is less than $5x10^{-5}$ torr.
17. **Do not** proceed to next step until collector plate temp. has reached the set point. Set point shall be achieved within first hour of pump down.
18. Enter predetermined heater SP for upcoming test specimen in the appropriate tower block on the computer screen. Activate heaters by clicking on virtual ON/OFF switch so that it indicates ON and turn on power strip to the right of computer.
19. Temperature of the “Hot” side must reach the desired set point within 1 hour. If the set point is above 150°C, up to 2 hours may be required. Once this is achieved, begin the 24-hour soak time, maintaining the desired temperatures.
20. After 24-hour soak, change heater set points to ambient, turn OFF power strip, and deactivate controller by clicking on the virtual ON/OFF switch so that it indicates OFF.
21. Turn off BA #1 gauge and select TC#1 gauge.
22. Turn OFF turbo and roughing pumps.
23. Open valve on Nitrogen K-bottle. Regulator should be set for minimal flow, and low-pressure needle should be at lowest point. You should hear a slight hiss at the backfill valve.
24. Slowly open backfill valve until chamber begins to repressurize. As pressure increases, open backfill valve a little more if necessary.
25. Once chamber has reached a pressure of about 150 ($1.5x10^{+2}$) torr, close backfill valve and close K-bottle valve. This will allow the heater bars to cool quicker.
26. When the heater bar (tower “hot” side) cools to 10°C above ambient, change circulating bath set point to ambient temperature.
27. Once collector plate (tower “cold” side) has reached room temperature, turn OFF the circulating bath and complete backfill of chamber by again opening K-bottle valve and slowly opening backfill valve.
28. Once chamber has returned to atmospheric pressure, close backfill valve and close K-bottle valve.
29. Close gate valve.
30. Open vent valve.
31. Raise bell with hoist.
32. Remove OWS holder from tower, immediately place in desiccator, and evaluate OWS per section 7.7.4 of MSFC-SPEC-1443 as soon as possible.
33. If OWS reflectance measurements are acceptable, proceed to next section. If OWS fails requirements, repeat sections 4.1 & 4.2 of this document until chamber meets cleanliness requirement.

4.4 CHAMBER OPERATION FOR ROUTINE TESTING

1. Verify that the power strip to the right of computer is switched OFF. Turn on system computer and monitor. When boot is complete, control software should automatically run. Activate controls by going to the *Panel* pull down menu and click on *Start Task.*
2. Open vent valve and using TC gauge #1 verify that chamber pressure is at ambient.
3. Using hoist, raise bell to provide access to test towers.
4. Disassemble test tower components (heater bar and separator plate) so that CWS and collector plates may be installed.
5. Install prepared collector plates onto cooling plate mounts.
6. Install the pre-measured CWS into special holder and install onto cooling plate mount.
7. Install separator plate onto cooling plate.
8. Mount heater bar and remove covers from specimen compartments.
9. Insert samples, prepared per MSFC-SPEC-1443, into bar compartments and replace covers securely.
10. Verify that correct cartridge heaters are installed in appropriate heater bar.
11. Verify that o-ring seal is correctly in place and free from particulate contamination.
12. Slowly lower bell with hoist until bell is completely seated on collar and tension is released from hoist cable.
13. Close vent valve and verify that backfill valve is closed.
14. Open gate valve and turn on roughing pump.
15. Turn on circulating bath unit and set temperature to predetermined collector plate temperature for sample test. Be sure that the refrigeration switch is ON.
16. When TC gauge #1 reads 1 torr press turbo pump start button.
17. When turbo pump controller shows pump has reached normal operation, select BA gauge #1 and turn on emission. Hold until pressure is less than $5x10^{-5}$ torr. Chamber must achieve $5x10^{-5}$ torr or better within an hour of pump down start.
18. **Do not** proceed to next step until collector plate temp. has reached the set point. Set point shall be achieved within first hour of pump down.
19. Enter predetermined heater SP for test specimen in the appropriate tower block on the computer screen. Activate heaters by clicking on virtual ON/OFF switch so that it indicates ON and turn on power strip to the right of computer.
20. Temperature of the “Hot” side must reach the desired set point within 1 hour. If the set point is above 150°C, up to 2 hours may be required. Once this is achieved, begin the 24-hour soak time, maintaining the desired temperatures.
21. After 24-hour soak, change heater set points to ambient, turn OFF power strip, and deactivate controller by clicking on the virtual ON/OFF switch so that it indicates OFF.
22. Turn off BA #1 gauge and select TC#1 gauge.
23. Turn OFF turbo and roughing pumps.

24. Open valve on Nitrogen K-bottle. Regulator should be set for minimal flow, and low-pressure needle should be at lowest point. You should hear a slight hiss at the backfill valve.
25. Slowly open backfill valve until chamber begins to repressurize. As pressure increases, open backfill valve a little more if necessary.
26. Once chamber has reached a pressure of about 150 ($1.5x10^{+2}$) torr, close backfill valve and close K-bottle valve. This will allow the heater bars to cool quicker.
27. When the heater bar (tower "hot" side) cools to 10°C above ambient, change circulating bath set point to ambient temperature.
28. Once collector plate (tower "cold" side) has reached room temperature, turn OFF the circulating bath and complete backfill of chamber by again opening K-bottle valve and slowly opening backfill valve.
29. When chamber has returned to atmospheric pressure, close backfill valve and close K-bottle valve.
30. Close gate valve.
31. Open vent valve.
32. Raise bell with hoist.
33. Immediately remove specimen boats, collector plates and OWS for analysis following prescribed procedures outlined in MSFC-SPEC-1443.
34. Verify that o-ring seal is correctly in place and free from particulate contamination.
35. Slowly lower bell with hoist until bell is completely seated on collar and tension is released from hoist cable.
36. Close vent valve.
37. Shut down control software and turn off computer system.
38. Clean up the lab!

5. RECORDS

Current calibration and verification quality records for required instrumentation are to be kept in the envelope affixed to the chamber bell. Past records are to be maintained in folder with equipment for at least 3 years.

Notes, test equipment list, and other pertinent testing information should be recorded in the associated laboratory notebook labeled "VCMO Chamber." These records should be maintained for at least 3 years and stored near the test chamber.

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20. REVISION:	21. ENGINEERING ORDER:	22. PARTS LIST:	23. CCBD:

III. REPORTS, SPECIFICATIONS, ETC.

24. REVISION:	25. CHANGE:	26. VOLUME:	27. BOOK:	28. PART:	29. SECTION:
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- ☐ Proprietary (see MPD 2210.1)
- ☐ Patent (see MPG 2220.1)
- ☐ ITAR (see MPG 2220.1)
- ☐ EAR (see MPG 2220.1)
- ☐ Other ACI (see NPG 1620.1 and MPG 1600.1) _____
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40. ORG. CODE:	41. PHONE NUMBER:	42. NAME:	43. SIGNATURE/DATE:
ED31	(256) 544-7647	Ralph Carruth, Group Lead	Ralph Carruth 5/29/02

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