

# Outgassing Testing in accordance with ASTM E 595-93



! This Instruction Contains  
Descriptions of  
• **HAZARDOUS OPERATIONS** •

Materials and Processes Laboratory  
Materials Test Branch, Building 4623

National Aeronautics and Space Administration  
George C. Marshall Space Flight Center  
Marshall Space Flight Center, AL 35812

Release Authority	Name	Title	Organization	Date
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Revision	Date	Originator	Description	Affected Pages
Baseline	2/4/05	Eddie Davis	Document converted from ED36-OWI-040. Previous history retained in system as part of canceled or superseded ISO Document files.	All
A	11/17/05	Eddie Davis	Hazardous Operations notification added to cover	Cover, ii

This document baselines the Organizational Work Instruction (OWI) for performing outgassing tests in Building 4623. Any deviation to this procedure must be approved by the test engineer via an approved test plan. Any changes to the test equipment shall be noted on the tester maintenance log and approved by the test engineer. It is the responsibility of the test engineer to obtain NASA Contracting Officer's Technical Representative (COTR) approval where necessary for changes to the test equipment.

Any change to this OWI must be submitted to and approved by the Materials Test Branch Chief, EM10. Revisions may also be submitted to the concurring organizations listed below for review and concurrence by memo. The original OWI and all changes shall be maintained by EM10. Any change to materials used requires a change to mechanical drawings, in addition to EM10 Chemistry Team Leader approval. All documentation must be approved by the appropriate persons mentioned above and incorporated into the OWI before operation of the reconfigured test equipment can resume.

Concurring organizations:  
 Building 4623 Test Operations Contractor  
 EM10 COTR  
 Environmental Health, AD60M

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## 1.0 Scope

### 1.1 Scope

The scope of this Organizational Work Instruction (OWI) is the Outgassing Test in accordance with ASTM E 595-93, as performed in Building 4623 at Marshall Space Flight Center (MSFC).

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### 1.2 Purpose

The purpose of the Outgassing Test is to evaluate the mass loss of materials being subjected to 125 °C at less than  $5 \times 10^{-5}$  torr for 24 hours. This document covers a screening technique to determine the volatile content of materials when exposed to a vacuum environment.

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### 1.3 Applicability

This instruction applies to the Chemistry Team, Materials Test Branch, of the Materials and Processes Laboratory.

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## 2.0 Applicable Documents

ASTM E 595-93(1999), *Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment.*

EM10-OWI-CHM-042, *Test Sample Preparation for Testing in Building 4623.*

EM10-OWI-CHM-050, *Building 4623 Guidelines for Test Operations.*

EM10-OWI-CHM-051, *Receipt, Handling, Prioritizing, and Data Requirements for Samples Submitted for Testing in the Building 4623 of the Materials and Processes Laboratory.*

EM10-OWI-CHM-058, *Chemical Hygiene Plan for Building 4623.*

JSC SP-R-0022A, *General Specification: Vacuum Stability Requirement of Polymeric Material for Spacecraft Application.*

MPD 1840.3. *MSFC Respiratory Protection Program.*

MPR 1040.3. *MSFC Emergency Plan, current revision.*

MPR 1840.2. *MSFC Hazard Communications Program.*

MPR 8715.1. *MSFC Safety, Health, and Environmental (SHE) Program.*

MPG 8823.2. *Pressure Systems Guidelines and Certification Requirement.*

MSFC-SPEC-164B, *Specification for Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems.*

MWI 3410.1. *Personnel Certification Program.*

MWI 8621.1. *Close Call and Mishap Reporting and Investigation Program.*

**Note:** Personnel **shall always refer** to the most current version of the above documents.



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## 3.0 Definitions

### 3.1 Definitions

*Ambient pressure.* The pressure of a surrounding medium, such as air, that comes in contact with some specified body or object; for the purpose of this OWI, the term *ambient pressure* means 14.7 psi.

*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, expressed as a percentage of initial specimen mass, 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. Standard test plates are chromium-plated aluminum, but other materials, which better simulate surfaces of interest, are optional.

*Contamination-sensitive surface.* Any surface of flight hardware that could be adversely affected by contamination, e.g., mirrored optics, windows, detectors, thermal control surfaces.

*NASA.* Marshall Space Flight Center EM10 responsible personnel.

*Outgassing.* Molecules released by the test sample; considered molecular contamination.

*Total mass loss (TML).* Total mass of material outgassed from a specimen 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 initial specimen mass.

*Water vapor regained (WVR) [also known as Regained Mass Loss (RML)].* Mass of water vapor regained by a specimen after the optional reconditioning step. WVR is calculated from differences in specimen mass determined after test for TML and again after exposure to a 50-percent relative humidity atmosphere at 21 °C for 24 hours. WVR is expressed as a percentage of initial specimen mass.

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### 3.2 Acronyms

<i>COTR</i>	Contracting Officer's Technical Representative
<i>CVCM</i>	Collected Volatile Condensable Material
<i>GN<sub>2</sub></i>	Gaseous Nitrogen
<i>MSDS</i>	Material Safety Data Sheet
<i>NASA</i>	National Aeronautics and Space Administration
<i>OWI</i>	Organizational Work Instruction
<i>RML</i>	Regained Mass Loss
<i>TFE</i>	Teflon™
<i>TML</i>	Total Mass Loss
<i>WVR</i>	Water Vapor Regained

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## 4.0 Instructions

### 4.1 Sample Handling

The following handling procedures ensure that specimen contamination does not occur:

**Note:** Specimen materials shall not be contaminated at any step in the sample preparation process. Specimen material shall not be handled with the bare hands: oils from human skin are volatile and condensable and, thus, will cause false TML and CVCM results.



The test conductor **shall**:

4.1.1. **Wear** latex gloves at all times when handling samples, sample hardware, or tools.



**Note:** **Clean** latex gloves before handling samples; **rinse** them with a 1:1 acetone/ethanol solution (section 4.3.1.), and allow them to air dry. **Verify** glove cleanliness by performing infrared analysis on the solvent rinse quarterly.

4.1.2. **Clean** all materials, unless cleaning will adversely affect a sample's material properties.

4.1.3. **Use** cleaning solvents that are known (a) to be non-reactive with the sample material and (b) to leave no residue.

4.1.4. **Discard** exterior surfaces of materials when preparing specimens. **Remove** exterior surfaces of harder materials with a clean jewelers' saw. **Use** a clean jewelers' drill to remove specimen material from the center part of material suspected to be contaminated.

**Note:** *If the exterior surfaces cannot be removed*, most material can be wiped with ethanol. **Perform** a test wipe to be sure that the material will not be harmed with the ethanol. *If ethanol harms the material and if the material will not absorb water*, **wipe** the material with deionized water.



### 4.2 Pre-Test Photography

Before testing, the *test operator* **shall photograph** the bulk sample material in accordance with the test request and **shall place** three copies of the photograph in the test folder. The *test operator* **shall verify** that the entire sample is visible in the photo. Steps for photographing samples are outlined in the *Photography Operating Guide*.

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### 4.3 Sample Preparation

The test conductor **shall prepare** 100 to 300 mg of each sample. An optimum mass of 200 mg is desired. Each sample shall be run in triplicate, so that a total mass of approximately 600 mg shall be prepared. *If smaller quantities are used, the accuracy of the measurements may be impaired.*



**Note:** Two kinds of aluminum containers are used in TVS testing. Sample boats are made of aluminum foil and are approximately 3/8 in. x 1/4 in. x 1/4 in. The 2.5-in. diameter aluminum weigh pans are used to hold the smaller boats or other parts.

The test conductor **shall perform** the following procedures:

4.3.1. **Clean** the aluminum foil by wiping each with a solution of 1 part spectrophotometric grade acetone and 1 part 200-proof ethanol.

4.3.2. **Create** the sample boats by cutting aluminum foil squares approximately 0.8 to 1 in. and then forming these into shape on an aluminum disk 0.75-in. thick with a diameter of 0.5625 in.

4.3.3. **Place** the sample boats in a clean pan or in a 2.5-in. weigh pan and **bake** in the vacuum oven at 150 °C for a minimum of 4 hours. (**See** section 7 for vacuum oven operation.)

4.3.4. **Prepare** the following types of materials as follows:

4.3.4.1. Elastomers, Hardware, and Structural Parts: **Cut** into small pieces on the order of 1/16- to 1/8-in. cubes to fit into the specimen boat; boats are approximately 3/8 in. x 1/4 in. x 1/4 in. **Clean** specimens with ethyl alcohol or a solvent that is compatible with the material and leaves no residue.

4.3.4.2. Adhesive Tapes: **Clean** specimens with ethyl alcohol or a solvent that is compatible with the material and leaves no residue. **Fold** adhesive tape in half so that the adhesives sides are inside. (*If material has a backing, remove the backing first.*) **Trim** folded edge of material.

4.3.4.3. Greases: **Place** greases in a sample boat.

4.3.4.4. Liquids: **Place** liquids directly in a sample boat, or **absorb** them in an ignited neutral filler, such as asbestos or silica, and then **place** in a boat.



**Note:** The technique used shall be stated in the report. Liquids and greases, especially silicones, are prone to creep; *if the material exhibits creep to such an extent that some flows out of the boat, the test results shall be disregarded.*

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4.3.5. **Review** the test data sheet before testing, and **fill in** the required data *e.g.*, weights, measurements, and test request number.

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#### 4.4 Equipment Checkout

The test conductor **shall perform** the following procedures before starting any test:

4.4.1. **Perform** a vacuum chamber bakeout *if the previous test failed or if the chamber has not been used within the last week*. **Follow** these steps at least 1 day before the regular TVS test. **Wipe** the inside of the chamber with a lint-free cloth and a solution of 1 part spectrophotometric grade acetone and 1 part 200-proof ethanol. **Follow** steps in section 4.6 to run the chamber as if conducting a test, with the following exceptions: the heater bar temperature should be at 150 °C, and the cooling bar at 50 °C for a minimum of 4 hours. It is not necessary to mount the bars together as you would for a test.

**Note:** The cooling bath can be set at 50.5 °C and left for the cleaning run. **Set** the heat bath at 175 °C, and when 150 °C is reached on the bar, **set** the bath to 163 °C.



After the chamber has baked for 4 hours, **follow** the shutdown procedure in steps 4.7.2 through 4.7.8. **Turn off** the cooling bath and power to the test with the main-line power lever on the control box. **Allow** the chamber to cool overnight.

4.4.2. **Perform** additional cleaning at least 1 day before testing. **Clean** all parts (except those made of aluminum) that will fit into beakers in a solution of 1 part spectrophotometric grade acetone and 1 part 200-proof ethanol. **Place** the beakers in the ultrasonic cleaner with 1 in. of water. **Turn on** the sonicator; **allow** the samples to sonicate for 15 minutes. **Do not allow** the beakers to float in the water. **Remove** the beakers, and **allow** all parts to air dry. **Place** parts in a clean pan, and **place** the pan in the vacuum oven at 150 °C for a minimum of 4 hours. (See section 7.1 for vacuum oven operation.)

4.4.3. **Inspect** the bell jar gasket for cleanliness, placement, and signs of cracks.

4.4.4. **Inspect** the system tubing/plumbing and glass components for signs of cracks or stress.

**Note:** *If any defects are noted during pretest inspection or during testing, take the component out of service immediately.*



**Note:** Before any test, **verify** that all mechanical connections for water and compressed gas are leak free. Also **inspect** vacuum system electrical cords to ensure



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all safety requirements are met, *e.g.*, cord strain relief, frayed insulation. **Notify** the test engineer, and then **correct** any unacceptable conditions.

4.4.5. **Verify** that the gate valve, oven pressure, and bell jar regulators are closed by checking their gauges, which read **0** psi when closed.

4.4.6. **Verify** that the GN<sub>2</sub> K bottle is properly belted to the wall.

## 4.5 Pre-Test Procedure

On the day before the test, the test conductor **shall perform** steps 4.5.1 and 4.5.2.



**Note:** **Save** all printouts of weights from the microbalance. **Record** all weights and identification numbers on appropriate data sheets at the end of the test.

4.5.1. **Weigh** three pre-cleaned aluminum foil boats.

4.5.2. **Add** 100 to 300 mg of test specimen to each boat, and **place** in a conditioning chamber [50-percent relative humidity and 23 (±2) °C] for a minimum of 24 hours.

On the day of the test, the test conductor **shall:**

4.5.3. **Turn** the vacuum system power on by lifting the Nema Control Panels' main-line circuit breaker to **ON**; the red mainline power light will illuminate (Figure 4-1A).

4.5.4. After 22 hours into the conditioning period, **start** the Virtual Bench Logger:

4.5.4.1. **Click** on the **Virtual Bench** icon.

4.5.4.2. **Click** on **Virtual Bench Logger**.

4.5.4.3. **Click** on **Start** in the data logger.

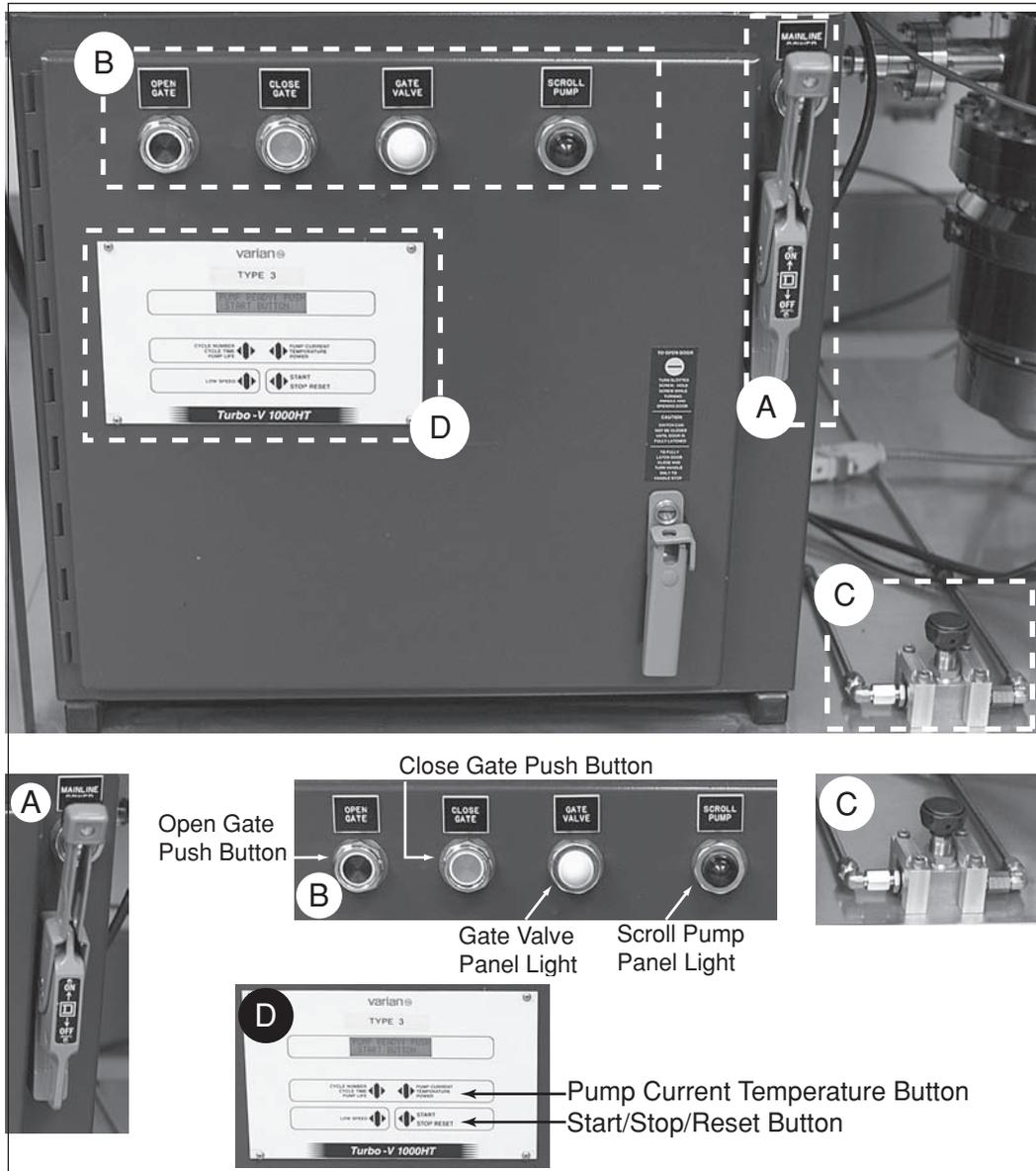
4.5.4.4. **Start** the cooling water bath (Figure 4-2) by depressing the **ON** switch in front of the unit. **Verify** that the set point is 25 °C.

4.5.4.5. **Start** the heater bath by pressing the **ON** switch upward. **Monitor** the temperature, and **allow** the heating oil to heat for 15 to 20 minutes, until the bath is approximately 30 °C.

4.5.4.6. **Turn off** the heater bath.



**Figure 4-2.**  
Cooling Water Bath



**Figure 4-1.**  
NEMA Vacuum System Control Panel:  
A. Circuit Breaker  
B. Control Panel  
C. Manual Vent Valve  
D. Varian Turbo-V Controller

4.5.4.7. Click on **STOP** in the **Bench Logger**.

4.5.4.8. Click on **Enable Logging**. Fill in the user name and comments, *if applicable*.

4.5.4.9. Click on **Edit**.

4.5.4.10. Click on **Settings**.

4.5.4.11. Click on **File Configuration**.

4.5.4.12. Click on **Browse** button, and ensure that you are working in the “Outgassing Folder” on the **C:** drive.

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4.5.4.13. **Open File Name.** The file name will consist of the Test Request number(s) of each sample being tested (up to three samples) in the following convention: “Test Number Test Number and Test Number.” For example, the file name for Test Request numbers 106000, 102715, and 104321 would be **106000 102715 and 104321**.

4.5.4.14. **Click Save.**

4.5.4.15. **Click Begin on Start.**

4.5.4.16. **Click OK.**

4.5.4.17. **Click on Timing Configuration.**

4.5.4.18. **Click on Begin Logging on Start.**

4.5.4.19. **Set the following parameters:**

**Time Interval = 5**

**Log to Disk = 60**

**Display Length = 28 hours**

**Select: Start Manually and Stop Manually**

4.5.4.20. **Click OK.**

4.5.4.21. **Click OK again.**

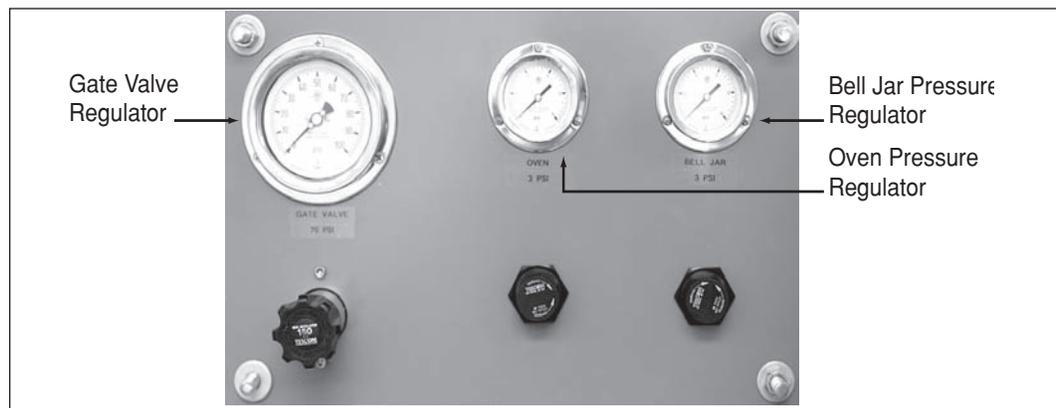
4.5.5. **Verify GN<sub>2</sub> pressure** by opening the K-bottle valve, turning it counterclockwise. The K-bottle is located on south wall.

**Note:** The GN<sub>2</sub> K-bottle shall remain off when not in use.

4.5.6. **Set the gate valve regulator to 70 psi** (Figure 4-3).



**Figure 4-3.**  
Gate Valve, Oven Pressure, and  
Bell Jar Pressure Regulators



4.5.7. **Set the oven pressure regulator to 3 psi** (Figure 4-3).

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4.5.8. **Set** the bell jar pressure regulator to 3 psi (Figure 4-3).

For the following steps, **refer** to Figure 4-1:

4.5.9. **Press** the **ON** button on the Granville-Phillips Vacuum Gauge Controller (Figure 4-4). The A and B convectron gauges will read atmospheric pressure.



**Figure 4-4.**  
Granville-Phillips Vacuum Gauge  
Controller.

4.5.10. **Verify** that the reading in *Pressure A* (measurement of pressure above the gate valve) is greater than or equal to the reading in *Pressure B* (measurement of pressure below the gate valve).

**Note:** *If the Pressure A reading is lower than the Pressure B reading, open the GN<sub>2</sub> valve located on south wall. Pressure shall not exceed 7.7x10<sup>2</sup> torr. If pressure does exceed 7.7x10<sup>2</sup>, shut off the GN<sub>2</sub> valve.*



4.5.11. *If Pressure A exceeds Pressure B, shut off the GN<sub>2</sub> valve, and fully reduce the bell jar and oven regulators.*

4.5.12. **Verify** that the chamber is pressurized. *If the chamber is not pressurized, pressurize the bell jar up to ambient pressure (14.7 psia). Press the **Open Gate** push button (Figure 4-1B). The gate valve will open, and the yellow **Gate Valve** panel light will illuminate (Figure 4-1B). Close the Manual Vent Valve (Figure 4-1C.)*

**WARNING!** Do NOT open the gate valve if the bell jar is under vacuum.



4.5.13. **Raise** the vacuum bell jar by pressing the **Up** manual control button on the Vacuum Bell Jar Controller (Figure 4-5). It may be necessary to reseal the Viton® O-ring after raising the bell jar.

**WARNING!** When lifting the bell jar by using the manual control button, **stop lifting immediately after the bell jar has cleared the cooling plate, heater bar, and separator plate configuration.** (Figure 4-6).



4.5.14. **Weigh** each collector plate four times, and **save** the printout from the microbalance.

Figure 4-5.  
Vacuum Bell Jar Controller.

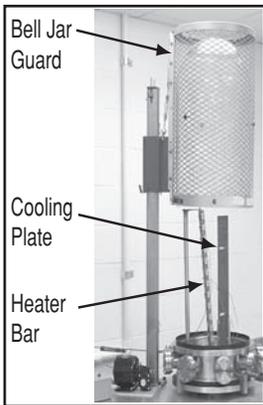
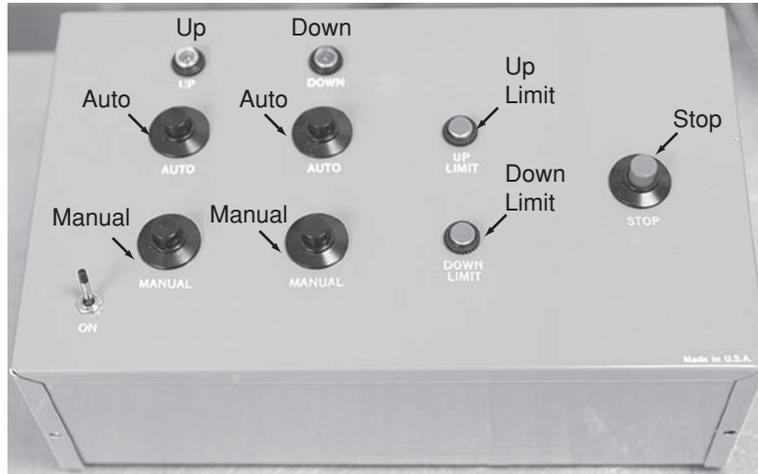


Figure 4-6.  
Vacuum Bell Jar in Raised  
Position.

4.5.15. **Mount** the collector plates in the appropriate cooling plates receptacles. **Screw** the knurled nut onto the back of the collector plates until it is finger tight.

4.5.16. **Place** the aluminum separator bar on the cooling plate with the open face toward the collector plates.

4.5.17. **Lift** the heater bar, and **place** it on the ceramic guides on the cooling plate.

4.5.18. **Attach** the aluminum separator bar with the aluminum clamps, and **tighten** the Allen bolts on the backside of the cooling plate. **Attach** the heating bar to the ceramic spacers with Phillips head screws.

4.5.19. **Weigh** and **record** weights of conditioned sample within 2 minutes after removing it from the conditioning chamber. The sample may be stored in a closed desiccator to prevent water loss or absorption.

4.5.20. **Place** specimen boats into heater bar compartments in the test system.

4.5.21. **Mount** and **screw down** respective cover plates on the entry end of each specimen compartment.

**Note:** **Check** level of fluids in heating and cooling baths before each test.



## 4.6 Detailed Test Procedure

After all pretest conditions have been met, the test conductor **shall perform** the following steps:

**WARNING!** Do NOT operate vacuum system bell jar without the guard in place.



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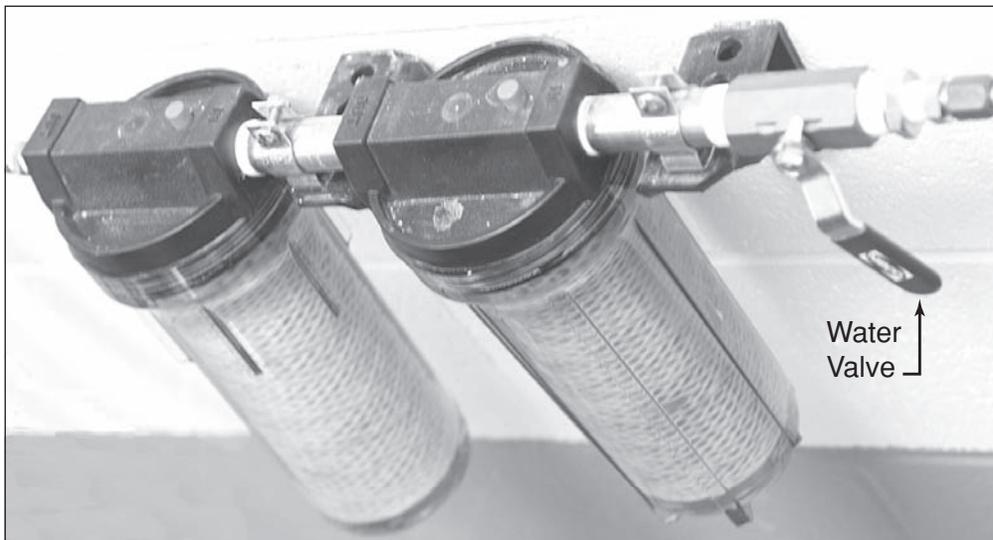
4.6.1. **Lower/close** the vacuum system bell jar using the **Down** manual control button (Figure 4-5). Lightly press the manual **Down** button. A quick tap of the button will lower the bell jar all the way to the flange.

4.6.2. *If the turbo pump is under vacuum and the gate valve is closed, shut off* the vacuum pumps by pressing the **Start/Stop/Reset** button on the Varian Turbo-V controller (Figure 4-1D), and **allow** the vacuum pumps to reach ambient pressure (14.7 psia) before opening the gate valve.

**Note:** Step 4.6.2 prevents damage to the pump.

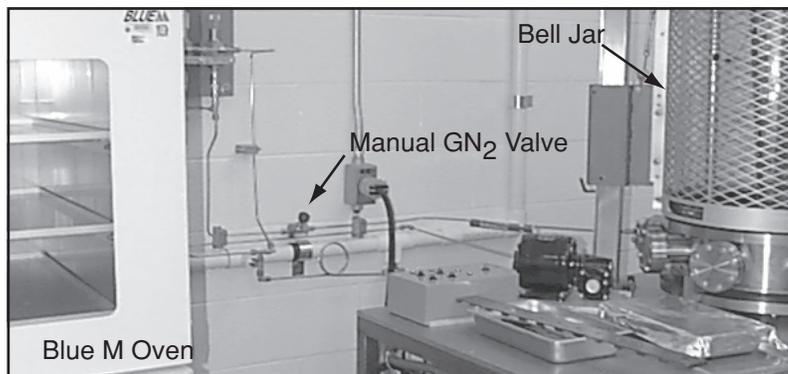
4.6.3. **Verify** that the gate valve located on the vacuum system control panel (Figure 4-1B) is open; *if it is not, press* the **Open Gate** button; a yellow light will come on.

4.6.4. **Start** the cooling water flow (located on the wall behind the test system) by opening the water valve (Figure 4-7).



**Figure 4-7.**  
Outgassing Turbopump Cooling Water System.

4.6.5. **Ensure** that the manual vent valve (Figure 4-1C) and manual GN<sub>2</sub> valve (Figure 4-8) are closed.



**Figure 4-8.**  
Location of manual GN<sub>2</sub> valve with respect to the TVS bell jar and Blue M oven

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**4.6.6.** **Start** vacuum by pressing the **Start/Stop/Reset** button on the Varian Turbo-V controller (Figure 4-1D). The scroll pump will start (indicated by the green front panel light), and the turbo will start.



**Note: Monitor** the pump temperature during the first hour and periodically thereafter. To see the pump temperature, **press** the **Pump Current Temperature** button (Figure 4-1D). The temperature shall not exceed 35 °C. *If the temperature exceeds 35 °C, consult* the test engineer.

**4.6.7.** **Start** the **Virtual Bench Logger** by clicking on the **Start** icon. **Move** the yellow arrow to the far right side of the **Bench Logger**.

**4.6.8.** **Turn on** the bath for the heater bar using the power **ON/OFF** switch on the Temperature Bath Controller for the heater bar (Figure 4-9). **Set** the heater bar bath temperature to 165 °C.

**4.6.9.** When Pressure A reads  $3.5 \times 10^{-3}$ , **turn on Ion Gauge IG1**, by pressing the **ON** button for IG1 on the Granville-Phillips Controller (Figure 4-4).

**4.6.10.** **Evacuate** the vacuum chamber to a pressure of  $5 \times 10^{-5}$  torr or less. *If this pressure is not reached within 15 minutes, notify* the test engineer.

**4.6.11.** **Leave** the heater bar set at 165 °C until it reaches approximately 110 °C. **Lower** the temperature setting to 138 °C, and **continue** to adjust the temperature downward as necessary. The correct temperature is 125 °C ( $\pm 1$  °C). It may be necessary to turn off the heater bath for short periods, *if it appears the temperature will exceed 126 °C.*

**4.6.12.** The test begins when the temperature reaches 124 °C. **Record** this time on the whiteboard.

**4.6.13.** **Monitor** the heater bath temperature for the first 2 hours to ensure the temperature does not exceed 126 °C. The heater bar temperature may require continued adjustment during this time. After final adjustment at 125 °C, the temperature controllers will maintain the temperature within  $\pm 1$  °C automatically.

**4.6.14.** **Maintain** both the heater bar and cooling bar temperatures at the desired level for 24 hours.

**4.6.15.** At the end of the workday, **record** -- on the whiteboard -- the lowest pressure attained.



**Figure 4-9.**  
Temperature Bath Controller for  
the Heater Bar

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## 4.7 Shutdown Procedure

After maintaining the temperature for 24 hours, the test conductor shall **perform** the following steps:

- 4.7.1. **Close** the gate valve, and **shut off** power to the heater bar bath.
- 4.7.2. **Shut off** the vacuum pump by pressing the **Start/Stop/Reset** button on the Varian Turbo-V controller (Figure 4-1D); the turbo will vent.
- 4.7.3. **Turn off** the vacuum pump cooling water.
- 4.7.4. **Set** the oven pressure regulator to 3 psi (Figure 4-3).
- 4.7.5. **Set** the bell jar pressure regulator to 3 psi (Figure 4-3).
- 4.7.6. **Turn off** the heater bar circulator (heater bath).
- 4.7.7. **Open** the GN<sub>2</sub> valve, and **backfill** the bell jar with clean, dry nitrogen regulated within a gauge pressure range of 2 to 4 psi to cool the bar rapidly. **Monitor** the pressure gauges so that the bell jar pressure does not exceed ambient. When ambient pressure is reached, **close** the GN<sub>2</sub> valve. Fully **reduce** the gate valve, vacuum oven, and bell jar regulators. **Turn off** the GN<sub>2</sub> bottle.
- 4.7.8. **Monitor** to ensure that the cooling plate temperature does not go above or below the target temperature. *If the temperature does decrease, raise* the cooling bath setpoint to 25.9 °C.
- 4.7.9. **Allow** the heater bar to cool sufficiently to permit handling (50 °C). **Add** additional GN<sub>2</sub> to the bell jar after temperature has reached 50 °C, because the pressure will drop below ambient. **Repeat** step 4.7.7 to return the pressure in the bell jar to ambient.
- 4.7.10. **Stop** the **Virtual Bench Logger** by pressing **Stop** icon.
- 4.7.11. **Raise** the vacuum bell jar by using the manual control **Up** button (Figure 4-4). **Remove** specimen boats and collector plates.
- 4.7.12. **Store** aluminum specimen boats and collector plates in the glass desiccator jar immediately. After specimens have cooled to room temperature (but no longer than 30 minutes), **remove** them from the desiccator. **Weigh** each specimen and collector plate within 2 minutes of its removal from the desiccator. **Weigh** each collector plate four times; **weigh** each specimen once. **Set** the collector plates to the side.

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**Note:** Determination of TML and WVR is affected by the material's ability to gain or lose water vapor within the allowed 2-minute time frame.

4.7.13. **Place** specimens in the conditioning chamber for 24 hours.

4.7.14. **Lower** the bell jar.

4.7.15. **Turn off** the cooling bar circulator.

4.7.16. **Turn off** the bell jar lift power switch.

4.7.17. **Turn off** the pressure gauge power switch.

4.7.18. **Turn off** the mainline power circuit breaker (Figure 4-1A).

4.7.19. **Reweigh** conditioned specimen after the 24-hour post-test conditioning period; this determines the specimens' WVR.

## **4.8 Data Recording and Reduction**

4.8.1. **Save** temperature data to floppy disk. (See section 7.2.1 for detailed procedure.)

4.8.2. **Print** a graph of temperature data. (See section 7.2.2 for detailed procedure.)

4.8.3. **Calculate** TML, CVCM, and WVR values. (See section 7.2.1 for detailed procedure.)

4.8.4. **Enter** sample data in MAPTIS. (See section 7.2.3 for detailed procedure.)

4.8.5. **Verify** the following information is placed in the completed sample folder.

- Sample Preparation Sheet (MAPTIS printout)
- Pretest photographs (three)
- Sample Temperature Graph (from step 4.9.2)
- Collector Plate Temperature Chart (from step 4.9.1)
- Collector Plate Weight Sheet (from step 4.9.3)
- Pretest Checklist (Figure 7-3)
- Calibration Sheet (Figure 7-4)
- Final Data Sheet (Figure 7-5).

4.8.6. **Return** the sample Test Folder to the test engineer.

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## 5.0 Notes

### Custodians for EM1-OWI-CHM-040

Master List and Document Control	EM10 Management Support Assistant
Alternate Document Control	EM10 Group ISO Representative
Records	Materials Test Branch ISO Representative
Calibration	Materials Test Branch Calibration Contact
Memoranda	Materials Test Branch ISO Representative

**CHECK THE MASTER LIST -- ONLY THE LATEST VERSION IS VALID**

## 6.0 Safety Precautions and Warning Notes

### Warning

Death, severe personal injury, or loss of major equipment may result if maintenance or operating procedures, techniques, restrictions, etc., are not followed exactly.

### 6.1 Hazards

Safety **shall have precedence** over all activities. The nature of Outgassing Testing in Building 4623 involves several potential hazards, including:

- Transporting K-bottles
- Electric shock related to operation of equipment
- Potential touch temperature risks from Blue M Oven and bell jar when handling test equipment
- Systems pressurized with nitrogen
- Handling of hazardous chemicals.

### 6.2 Safety Precautions

6.2.1. Testing **shall be planned** so that at least one test operator is in the test area and one other person is in Building 4623 during testing. After normal business hours and on weekends, a test engineer **shall be** in Building 4623 during all test activities. **No more than five persons shall be** in the test area at any given time. Operation of tests **shall comply** with EM10-OWI-CHM-050, *Building 4623 Guidelines for Test Operations*.

6.2.2. The test controls **shall not be operated** when personnel are working with the tester. A sign **shall be placed** on the door into the TVS test area warning that personnel are working with the TVS.

6.2.3. The test conductor **shall read** the Materials Safety Data Sheets (MSDSs) for all chemicals used or encountered during testing, **shall read the test material's MSDS to ensure familiarity with all safety precautions associated with the material**, and **shall wear** safety apparel appropriate for the test specimens and conditions:

- Safety shoes when there is a danger of foot injuries from falling or rolling objects, objects piercing the sole of the shoe, or when feet may be exposed to an electrical hazard.
- Clean laboratory jackets when working with enriched oxygen or other oxidizers, combustion by-products, compressed gases, or flammable solvents
- Chemically resistant goggles and gloves while cleaning test equipment and while working with solvents
- Clean thermal gloves and goggles when pouring, handling, or transferring cryogenic fluids. (Hydrocarbon residue can contaminate test equipment, affect oxygen compatibility test results, or damage the tester.)
- A respirator when working with solvents in closed or poorly ventilated spaces. **Note** that the appropriate respirator shall be worn as indicated on the MSDS. Cartridge respirators are only good for the constituents listed on the filtration



**CHECK THE MASTER LIST -- ONLY THE LATEST VERSION IS VALID**

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cartridge and for dust particle filtration. Personnel shall be qualified to use the respirator, and the respirator shall be supplied by MSFC.

- Hearing protection during testing
- Safety glasses at all times while in the test cells.

**6.2.4. Smoking is not permitted** in Building 4623. The test area is generally an oxygen-enriched environment. Open flames or other high-temperature sources **shall not be permissible** in the testing area while enriched-oxygen conditions exist.



**6.2.5.** Personnel **shall evacuate** the test area immediately when the oxygen alarm sounds and lights flash.

**6.2.6.** Nothing **shall be stored** in the TVS laboratory other than parts or components of the testing apparatus that are designated as spare parts and the tools necessary for routine equipment maintenance. All other materials shall be removed from the test area.

**6.2.7.** The test conductor **shall ensure** all electrical components, wiring, *etc.*, are in good condition and properly connected and grounded, **shall not operate** electrical devices when floors in the test cell are wet, and **shall use caution** when operating any electrical equipment.



**6.2.8.** When handling cylinders and dewars or when making connections for compressed gases and/or liquids, personnel **shall refer** to *Working Safely with Compressed Gases and Cryogenics* and *NSTC 313-Cryogenics Safety*, seeing the test engineer for these resources. Personnel **shall comply** with the suggestions inside these presentations.

**6.2.9.** The Outgassing Test System **shall not be operated** without the bell jar guard in place.

**6.2.10.** The GN<sub>2</sub> **shall remain off** while not in use.

### 6.3 Special Precautions Associated with Compressed Gases and Liquids

**6.3.1.** All operations involving compressed gases and liquids **shall be conducted** with at least 2 people, in visual contact, in the facility.

**6.3.2.** All operating personnel **shall be instructed** on the nature of hazards associated with compressed gases and liquids.

**6.3.3.** Before removal of any component of the system for servicing, the operator **shall secure and inspect** the system to ensure that no unsafe condition exists.

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6.3.4. Personnel **shall perform** continuous monitoring, *e.g.*, **check** operating pressures, look for leaks, listen for unusual noises, during all operations. Personnel shall ensure that oxygen leak levels are adequate throughout operations.

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## 6.4 Emergency Shutdown Procedure

Not applicable. This tester does not have to be shut down to be considered safe in an emergency situation.

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## 6.5 Accident Reporting

6.5.1. From a safe location, the *test operator* **shall immediately call 911** and **notify** the EM10 Materials Test Branch Chief.

6.5.2. From a safe location, the notified *EM10 Materials Test Branch Chief* **shall immediately report** the accident to the NASA Safety Monitor and the appropriate supervisor(s).

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## 6.6 Emergency Response Plan

Emergency procedures and plans for Building 4623 are incorporated into the OWIs and are stated in MPR 1040.3. *MSFC Emergency Plan*, current revision. Plans **shall be modified** if operations change in a significant manner.

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## 6.7 Mishap Reporting

Personnel **shall report** all mishaps occurring in Building 4623 to the test engineer, who **shall report** the mishap to the area coordinator/Safety Monitor. An initial verbal report **shall be made** within 8 hours, followed by a written report within 3 days. The *EM10 Materials Test Branch Chief* **shall prepare** a managerial report within 7 days. Both reports **shall be reviewed** by the test operator's supervisor and by the NASA Safety Monitor. The detail and extent of the mishap report **shall depend** on the nature and extent of the damage. *If personnel injury or equipment damage does occur*, the mishap report **shall be completed** in accordance with MWI 8621.1, *Close Call and Mishap Reporting and Investigation Program*.

## 7.0 Attachments, Data, Reports, and Forms

### 7.1 Attachment: Blue M Oven Vacuum Bakeout Procedures

This section provides the proper procedures for using the Lindberg/Blue M Vacuum Oven (Figure 7-1) for hardware bakeouts. The oven **shall be used** exclusively for bakeout of clean outgassing test system parts.

The test conductor **shall**:

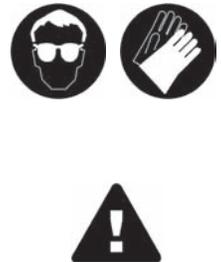
**7.1.1. Observe** the following precautions when operating the oven:

- **Wear** protective eye-wear.
- **Wear** protective gloves.
- **Do not allow** the load to touch the oven wall.

**CAUTION: Do not attempt** to operate the oven above the maximum-rated temperature of 260 °C. Operation above 260 °C can cause damage to the oven.



Figure 7-1.  
Blue M Vacuum Oven.



**7.1.2. Turn on** the main power by using the **ON/OFF** switch.

**7.1.3. Press** any key to illuminate the decrease, increase, and next parameter keys (Figure 7-2). **Set** the temperature setpoint to 150 °C by using the decrease or increase keys. (The desired setpoint shows on the bottom line of the display.) The oven takes ~2 hours to reach 150 °C.

**7.1.4. Place** the hardware in the vacuum oven.

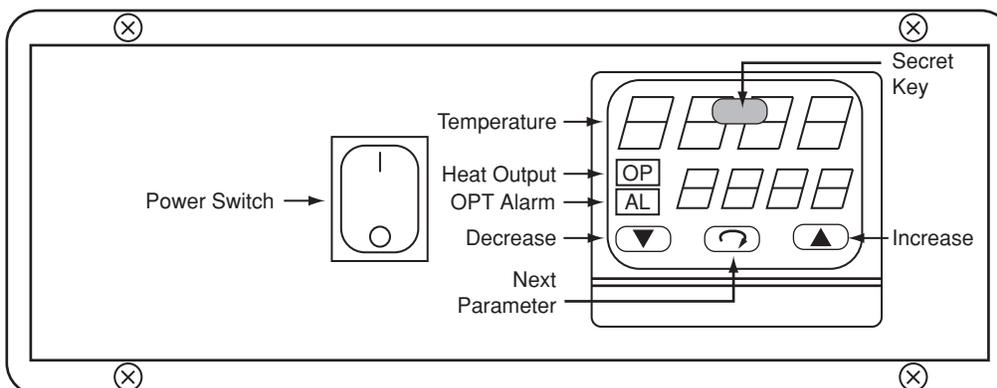


Figure 7-2.  
Blue M Vacuum Oven Controller Panel.

**7.1.5. Press** the Next Parameter button until AL.SP (alarm setpoint) shows on the top line of the display. Then, **press** the decrease and increase buttons to set the temperature controller alarm (typically 5 °C above the main temperature setpoint).

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7.1.6. **Start** the vacuum pump. (The switch is located on a leg of the oven table.)

7.1.7. The vacuum and vent valve controls located on the top front panel are not operational. **Fully open** the vacuum valve (located on the rear of the unit) by turning the handle counterclockwise.

7.1.8. When the temperature setpoint (150 °C) is reached, **allow** the items to bake for 4 hours. *If the oven fails to reach the setpoint, press the **Overtemperature-Reset** button on the rear of the unit. If the oven still fails to operate, contact the test engineer.*

7.1.9. To shut down the oven, **complete** the following steps:

7.1.9.1. **Close** the vacuum valve located on the rear of the oven.

7.1.9.2. **Turn off** the vacuum pump.

7.1.9.3. **Verify** the oven pressure regulator is set to 3 psi.

7.1.9.4. **Open** the gas valve located on top front of oven.

**WARNING! Do not open vacuum or vent valves at this location.**

7.1.9.5. **Turn off** the main power.

7.1.9.6. After pressure reaches 0 psig, **remove** items from the oven.

7.1.9.7. **Close** the gas valve on the front of the vacuum oven.



## 7.2 TVS Data Entry Procedure

This section describes the TVS data entry procedures for:

- Completing the Test Data Sheet and Collector Plate Sheet with automatic calculations of TML, CVCM, and WVR values (**See** section 7.3 for the equations for these values.)
- Printing graphs of temperature data
- Entering sample data into MAPTIS.

### 7.2.1. **Complete** Test Data Sheet and Collector Plate Sheet

The test conductor shall perform the following procedures:

7.2.1.1. **Insert** the floppy in a floppy disk drive of the TVS computer.

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7.2.1.2. **Save** the temperature data from the Data Logger to a floppy disk. Temperature data are located in the directory C:\Out Gassing.

7.2.1.3. **Ensure** that the TVS sample folder contains the following: Test Request, photos, product data sheets, and the microbalance printouts.

7.2.1.4. From the test operator's desktop computer, **open** the Excel file named **Updated OG Test Data Sheet blank**.

7.2.1.5. **Open** the worksheet labeled <Collectplatewt>.

7.2.1.6. **Enter** the collector plate weights from the microbalance printout from "Before" and "After" the TVS test, and **print** the worksheet.

7.2.1.7. **Open** the worksheet labeled <Test Data Sheet>. **Ensure** that the collector plate data carried over to the Test Data Sheet in the section labeled "Collected Volatile Condensable Materials (CVCM)."

7.2.1.8. **Enter** the pertinent sample information at the top portion of the spreadsheet.

7.2.1.9. In the section labeled "Total Mass Loss (TML)," **enter** the following weight data:

- (a) *Position*: position on the TVS tester sample holder from 1 (bottom slot) to 12 (top slot)
- (b) *Initial Specimen WT + Boat WT (mg)*: initial mass of the sample plus the boat weight
- (c) *Initial Boat WT (mg)*: mass of the sample boat alone
- (d) *Initial Specimen WT (mg)*: automatically calculated from (a) and (b) above
- (e) *Final Specimen WT + Boat WT (mg)*: mass of the sample and boat after the 24 hours at vacuum
- (f) *Final Specimen WT (mg)*: calculated automatically from (c) and (e) above.

7.2.1.10. In the section labeled "Collected Volatile Condensable Materials (CVCM)," **enter** the following data:

- (a) *Position*: position on the TVS tester sample holder from 1 (bottom slot) to 12 (top slot)
- (b) *Collector Plates*: the number designation on the back of the collector plates.

7.2.1.11. In the section labeled "Water Vapor Regained (WVR) After 24 Hours," **enter** the following data:

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- (a) In the subsection labeled “Specimen WT + Boat WT (mg),” **enter** the masses of the samples + the masses of the sample boats after the 24-hour conditioning in the conditioning chamber.
- (b) **Verify** that the remainder of the subsections in “Water Vapor Regained (WVR) After 24 Hours” section fill automatically and that all other calculations have occurred automatically.



**Note:** Upon completion of step 7.2.1.11, the Excel program automatically calculates the TML, CVCMM, and WVR values.

7.2.1.12. **Click** the box labeled “Rate Results,” and the rating will be generated.

7.2.1.13. **Print** the <Test Data Sheet> and <Collectorplatewt> worksheets.

7.2.1.14. **Sign** the Test Data Sheet.

7.2.1.15. **Print** the worksheet labeled <Calibration Sheet.> **Verify** all the equipment is in calibration, **sign** this sheet, and **place** it in the sample Test Folder.

## 7.2.2. Temperature Graphs

The test conductor **shall perform** the following procedures:

7.2.2.1. In Excel, **open** the temperature data file saved onto the floppy disk. (See steps 7.2.1.1 and 7.2.1.2 above). The **Text Import Wizard** will appear.

7.2.2.2. **Select Next**; **select Next** again; then **select Finish**.

7.2.2.3. All the temperature data for all the TVS thermocouples will displayed. **Retain** the data from *Conditioning box (Deg C)*, *Sample temp. (Deg C)*, and *Collector plate temp. (Deg C)*. **Delete** the other thermocouple data (*TC2*, *TC4*, *TC5*, *TC6*, and *TC7*).

7.2.2.4. **Select** the entire imported worksheet, and **copy** it to the worksheet page <Temp & Vac. Data>.

7.2.2.5. Immediately to the right of the *Collector plate temp. (Deg C)* side of the <Temp & Vac. Data> worksheet, **enter** the lowest vacuum value attained during the TVS test at the time that corresponds closest to when this value was recorded. Enter the vacuum value in the format “**5.5 x 10<sup>-6</sup> torr**”.

7.2.2.6. At the row labeled *Sample temp. (Deg C)*, **highlight** all temperatures after the time the test started (at 124 °C).

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7.2.2.7. Select the **Chart Wizard** icon.

7.2.2.8. Select *XY Scatter* with *data points connected by smoothed lines without markers*.

7.2.2.9. Select *Next*, and select *Next* again.

7.2.2.10. Enter the item number and the sample name as the chart title.

7.2.2.11. Enter “*5 minute 2 sec. time intervals*” as the X axis; enter “*Temp. Deg. C*” as the Y axis.

7.2.2.12. Select *Next*; then select *Finish*.

7.2.2.13. Adjust the X axis and Y axis values to generate an attractive graph.

7.2.2.14. Delete the designation “Series 1” to the right of the graph.

7.2.2.15. Print the graph, and place it in the sample Test Folder.

7.2.2.16. On the worksheet row labeled *Collector plate temp. (Deg C)*, highlight all the temperatures after the time the test started (25 °C).

7.2.2.17. Follow steps 7.2.2.7 through 7.2.2.15 above. In step 7.2.2.10, enter item number and the words “Collector Plates” as the chart title.

7.2.2.18. Name the Excel file by the item number, and save it.

### 7.2.3. MAPTIS Data Entry

The test conductor **shall perform** the following procedures:

7.2.3.1. Log onto MAPTIS

7.2.3.2. Enter 2.B. (Material Selection, Nonmetals)

7.2.3.3. Choose 3. (MSFC (Since 1989))

7.2.3.4. Choose 34. (Sample Preparation Menu)

7.2.3.5. Choose 1. (Data Entry/Update)

7.2.3.6. Query the item number in the field “Request #.”

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7.2.3.7. **Press** the Enter key until the cursor reaches the field “Prep Photo:.” In this field, **enter** the photo number (the last four numbers in the item number and “TV”) in this format: 7653TV.

7.2.3.8. **Enter** the sample color, if it is not there.

7.2.3.9. **Enter** the cure number, if this is relevant.

7.2.3.10. **Enter** the relevant data in these fields: NHB Test: (TVS), Test Number (usually 1), Prep’d By (name of person who prepared the sample for TVS), and Prep Date (format DD-MMM-YYYY).

7.2.3.11. **Record** the relevant cleaning number in Clean No.

7.2.3.12. **Press** the Enter key until reaching the table. In “#,” **enter** 1, 2, and 3. **Press** the Enter key to move across the table to “Weight grams,” and **enter** the three sample weights.

7.2.3.13. **Press** the Enter key to the field “DESCRIPTION OF PREPARATION METHOD:” and **add** relevant comments describing sample preparation. (**Query** previous TVS sample data for examples.)

7.2.3.14. **Press** the F3 and the F4 keys to save the data and exit this screen. This will return the screen to the *Sample Prep Menu* page.

7.2.3.15. **Choose** 2 (Sample Prep Reports).

7.2.3.16. **Choose** 1 (Data Sheet).

7.2.3.18. **Choose** 1 (Sequence).

7.2.3.19. **Enter** the item number for querying in the field. The sample information will scroll past

7.2.3.20. **Press** the Return key; **enter** 4, which will send the query answer, then **enter** the number of a printer to select it.

7.2.3.21. **Place** the printed data sheets in the folder.

### 7.3 TML, CVCM, and WVR Calculations

This section presents the equations that shall be used to calculate TML, CVCM, and WVR values.

7.3.1. TML value shall be calculated according to the equations presented in Table 7-1.

Calculation	Initial Mass (mg)	Final Mass (mg)
Specimen and Boat	$S_1 + B_1$	$S_F + B_1$
Boat	$B_1$	$B_1$
Specimen Alone	$(S_1 + B_1) - B_1$	$(S_F + B_1) - B_1$
Difference or Mass Loss	$L = S_1 - S_F$  $L/S_1 \times 100 = \%TML$	

Table 7-1.  
Calculating TML Value

7.3.2. CVCM value **shall be calculated** according to the following equation:

$$C_O/S_1 \times 100 = \% CVCM$$

where:

$C_O$  = mass of condensables (mg=  $C_F - C_1$ )

$C_1$  = initial mass of collector plate (mg)

$C_F$  = final mass of collector plate and condensables (mg)

$S_1$  = initial specimen mass (mg)

7.3.3. WVR value shall be calculated according to the following equation:

$$[(S'_F - S_F) \div S_1] \times 100 = \% WVR$$

where:

$S'_F$  = reconditioned mass of specimen after 24 hr at 50% relative humidity (mg)

$S_F$  = specimen mass (mg) before reconditioning

## 7.4 Forms

Figures 7-3, 7-4, and 7-5 illustrate forms used in the conduct of the Outgassing Test.

**Figure 7-3.**  
Outgassing Tester Pre-Test  
Checklist.

<b>Outgassing Tester Pre-Test Checklist</b>	
Request No. _____	
Initial _____	
1.	<input type="checkbox"/> ASTM E 595-93 reviewed?
2.	<input type="checkbox"/> Outgassing Test OWI reviewed?
3.	<input type="checkbox"/> All safety equipment available and working properly?
4.	<input type="checkbox"/> Test equipment (bell jar, system tubing/plumbing, gauges) checked out according to OWI?
5.	<input type="checkbox"/> Sample MSDS read?
6.	<input type="checkbox"/> Test plan read, and changes noted and approved by engineer?
7.	<input type="checkbox"/> Pre-test photographs taken and in test folder?
8.	<input type="checkbox"/> Samples prepared according to ASTM E 595-93?
9.	<input type="checkbox"/> Test samples concur with the test request?
Discrepancies:	
_____	_____
Sample preparation technician	Date
_____	_____
Test operator	Date
1/05	EM10-F-CHM-066

Note: Representative Gases and Liquids Report. For Illustration only.

**CHECK THE MASTER LIST -- ONLY THE LATEST VERSION IS VALID**

### OUTGASSING CALIBRATION SHEET

Balance (Cat.1)

Make: Mettler Toledo                      Model: S4  
 Serial: 1119153953                      NEMS/Calibration: M647054  
 Cal. Due Date:                              1/14/2004

Humidity Controller (Cat.1)

Make: ETS                                      Model: 514  
 Serial: 614                                      NEMS/Calibration: 1965931  
 Cal. Due Date:                              1/14/2004

Thermocouple (Cat.1)

Make: Omega                                  Model: Type T  
 Serial: N/A                                      NEMS/Calibration: M646390  
 Cal. Due Date:                              1/14/2004

Thermocouple (Cat.1)

Make: Omega                                  Model: Type T  
 Serial: N/A                                      NEMS/Calibration: M646395  
 Cal. Due Date:                              1/14/2004

Vacuum Gauge Controller (Cat.1)

Make: Granville Phillips                      Model: 307001  
 Serial: 14029                                  NEMS/Calibration: M644323  
 Cal. Due Date:                              1/17/2004

Convectron Gauge (Cat.1)

Make: Granville Phillips                      Model: 008253  
 Serial: N/A                                      NEMS/Calibration: M644200  
 Cal. Due Date:                              1/17/2004

Convectron Gauge (Cat.1)

Make: Granville Phillips                      Model-, 008253  
 Serial: N/A                                      NEMS/Calibration: M644201  
 Cal. Due Date:                              1/17/2004

Ion Gauge (Cat.1)

Make: Granville Phillips                      Model: 27401  
 Serial: N/A                                      NEMS/Calibration: M643009  
 Cal. Due Date:                              1/17/2004

Ion Gauge (Cat.1)

Make: Granville Phillips                      Model: 274016  
 Serial: N/A                                      NEMS/Calibration: M643010  
 Cal. Due Date:                              1/17/2004

Test Request No.:  
 Test Technician:                                      Date:

**Figure 7-4.**  
 Calibration Sheet (sample).

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Figure 7-5.  
Outgassing Test Data Sheet.

OUTGASSING TEST DATA SHEET			
Test Request No.: _____ Project: _____			
Date: _____ Requestor: _____			
Material: _____ Manufacturer: _____			
Use: _____			
Sample Preparation			
Sample Temperature _____ (C)		Collector Plate Temperature _____ (C)	
Pressure _____ (torr)		Time at Test Temperature _____ (hr)	
Remarks: _____			
_____			
_____			
Test results			
Total Mass Loss (TML)			
	Sample 1	Sample 2	Sample 3
Position Number			
Initial Specimen + Boat WT			
Initial Boat WT			
Initial Specimen WT			
Final Specimen + Boat WT			
Boat WT			
Final Specimen WT			
Specimen Loss			
% TML			
Average % TML			
Collected Volatile Condensable Material (CVCM)			
Collector Plates			
CF (After)			
CI (Initial)			
% CVCM			
Average % CVCM			
Water Vapor Regained (WVR)			
Specimen + Boat			
WVR			
% WVR			
Average % WVR			
Test Operator: _____			
1/05		EM10-F-CHM-067	

Note: Representative Test Data Sheet. Refer to Forms Master List for current version.

**CHECK THE MASTER LIST -- ONLY THE LATEST VERSION IS VALID**

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## 8.0 Records

Records for Outgassing Testing shall consist of memoranda that contain test results and that are stored electronically in the Materials and Processes Technical Information System (MAPTIS) and calibration records.

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### 8.1 Memoranda

Memoranda containing test results shall be retained indefinitely by EM10. These memoranda shall be stored electronically in the MAPTIS database and shall be accessible by test request number or memorandum number.

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### 8.2 Calibration Records

8.2.1. All equipment requiring calibration shall be in current calibration, in accordance with EM10-OWI-CHM-050, *Building 4623 Guidelines for General Operations*.

8.2.2. The Outgassing Calibration Sheet (Figure 7-4), will be used to document the calibration of TVS equipment.

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### 8.3 Maintenance of Records

8.3.1. Memoranda less than 10 years old shall be maintained in ready-access files in MAPTIS; memoranda 10 years old or older shall be automatically transferred to historical files.

8.3.2. Calibration records shall be maintained on site for a minimum of 10 years, filed and indexed by test request number. These shall be stored in a manner that will protect them, *e.g.*, in a test folder stored in a metal file cabinet. After 10 years, calibration records shall be transferred to historical files.

8.3.3. The original test records shall be saved for a minimum of 5 years.

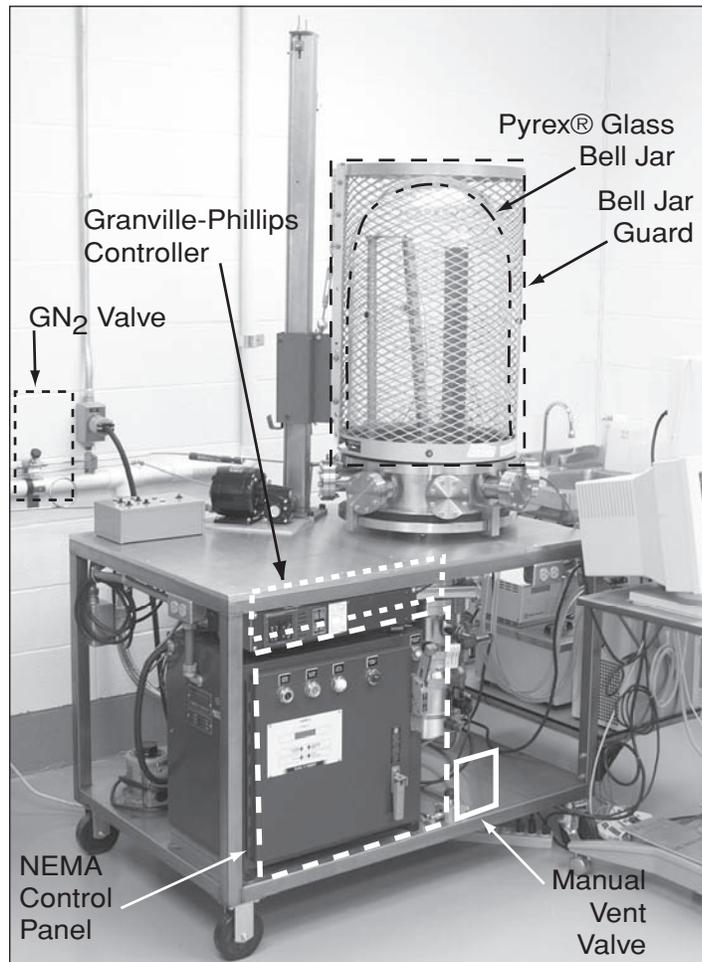
## 9.0 Tools, Equipment, and Materials

### 9.1 Test System Standard Configuration

The Outgassing Test System resides in Building 4623, Room 134. The system (Figure 9-1) is equipped with the following:

- 18-in. diameter by 30-in. high Pyrex® glass bell jar
- Manual vent valve
- Electropneumatic gate valve
- Granville-Phillips controller
- Spool housing for gauge tubes
- Varian V1000HT turbo pump
- Edwards High Vacuum scroll pump
- Stainless-steel framing
- NEMA control panel.

**Figure 9-1.**  
Outgassing Test System.



The Outgassing Test System tests outgassing properties of materials at elevated temperatures. Specimens are loaded into chambers that have been machined into a 25.5-in. long by 1.0-in. thick copper heater bar, which stands in the Pyrex® glass bell jar. After the specimens are loaded, the Varian V1000HT turbo pump and Edwards High Vacuum scroll pump pull a vacuum of  $10^{-6}$  torr on the Pyrex® glass bell jar. When the chamber reaches  $10^{-5}$  torr, heating of the samples begins. The heater bar is heated to 125 °C, using a recirculating flow of hot silicone fluid, and is held at that temperature for 24 hours. During this period, outgassed materi-

als condense on plates attached to a copper chilling bar. The second bar is chilled by recirculated water. After the 24-hour hold, the heating bar bath is turned off, the

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chamber is purged with GN<sub>2</sub> to cool the heater bar, and the samples and condenser plates are removed.

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## 9.2 Required Tester Maintenance

The standard maintenance program for this test system and related control equipment is divided into weekly and as-required service. In addition, the program involves a maintenance log, calibration, and a required spare parts inventory. The test conductor **shall perform** the following procedures:

### 9.2.1. Weekly Maintenance (General Housekeeping)

9.2.1.1. **Verify** that the Outgassing Test System's room is clean by sweeping and mopping the floor, wiping down stainless-steel tables, and eliminating trash.

9.2.1.2. **Verify** that the moisture level in the desiccant is adequate for the humidity chamber.

9.2.1.3. **Verify** that the silica gel desiccant level in the desiccators is adequate.

### 9.2.2. As-Required Maintenance

9.2.2.1. **Replace** test system water filters when the second filter starts to show contamination.

9.2.2.2. **Leak check** seals and flanges whenever the test vacuum level cannot be achieved.

9.2.2.3. **Replace** seals around flanges when they show signs of wear (cracking, etc.)

9.2.2.4. **Perform** leak checks on plumbing system.

9.2.2.5. **Replace** empty GN<sub>2</sub> K-bottles.

9.2.2.6. **Replace** thermocouples that are not operating properly.

9.2.3. Maintenance Log: **Document** any maintenance to the test system or setup in the *Outgassing System Maintenance Log* to provide a history of the test system. Any deviation to standard maintenance shall be documented by the test operator and approved on the maintenance log by the responsible test engineer.

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### 9.3 Calibration

Calibrated equipment lists of all categories for this OWI shall be kept by the primary calibration contact for Building 4623 (Figure 7-4). TVS equipment shall be calibrated annually.

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### 9.4 Additional Laboratory Equipment

9.4.1. Blue-M Vacuum Oven: for vacuum bakeout of hardware to maintain cleanliness

9.4.2. Mettler Microbalance: to weigh samples and collector plates

9.4.3. Personal computer: to monitor and record thermocouple temperatures in the Outgassing Test System

9.4.4. ETS Conditioning Chamber: to assure the samples are maintained at a constant humidity

9.4.5. GN<sub>2</sub> bottle: to fill the bell jar and vacuum oven with an inert atmosphere as necessary

9.4.6. Scalpels, tweezers and other equipment: required for sample preparation

9.4.7. Stainless steel cabinets and tables: to maintain sample and system cleanliness

9.4.8. Nobles clean room vacuum cleaner: to maintain room cleanliness

9.4.9. One glass and one stainless-steel desiccator: to store samples and sample expendables

9.4.10. Buehler Ultrasonic Cleaner: to clean Outgassing System hardware

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### 9.5 Required Spare Parts Inventory

Before beginning each test request, **ensure** that there are spares for all pieces of test equipment, as listed below.

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Part	Quantity
Adhesive Silica Gel Desiccant.....	1 gal
Aluminum Foil.....	1 box
Convectron Gauge with Universal 1/8 NPT and 1/2" Compression Fittings, P/N 275071 .....	2 ea.
Ethyl Alcohol (200 proof) .....	2 gal
Filter Element, NSN# 4330-01-015-9250 .....	2 ea.
GLV-18, #52002 L Gaskets (or equivalent).....	1 ea.
IG Tube, Duel Tungsten Cathodes, P/N 274016 .....	2 ea.
Type T Thermocouple Wires (20 gauge) .....	8 ft
Vacuum Gauge Controller .....	1 ea.
Vacuum Grease, Apiezon L .....	1 tube

For test system replacement parts not listed above, personnel shall **refer** to the *Shawfrank Instruction Manual Bill of Materials List*. Only parts that are listed above or parts that are in the *Shawfrank Construction Manual Bill of Materials* (or equivalent) may be used as replacement parts in this test system.

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## 10.0 Personnel Training

The nature of testing that occurs in Building 4623 is complex and involves potential hazards; therefore, all test operators **shall complete** the requirements for Category 1 Credentials before conducting any test, and all tester maintenance personnel **shall complete** the requirements for Category 2 Credentials.

- **Category 1 Credentials** qualify personnel to perform basic test operations.
- **Category 2 Credentials** qualify personnel to maintain and modify testing apparatus.

### Category 1 Credentials – Basic Operations

To obtain Category 1 Credentials, the test operator **shall complete** training in following areas:

- Handling of Compressed Gas Cylinders
- Oxygen Compatibility
- Use of Personal Protective Equipment
- General Safe Laboratory Practices
- Safe Handling of Cryogenic Fluids (LN<sub>2</sub> and LOX)
- Hazardous Waste Disposal.

**Category 1 Credentialing** also requires:

- Successful completion of an annual physical examination conducted by the medical facility at Marshall Space Flight Center (or equivalent), including a hearing exam
- A demonstration of knowledge of the test and equipment by the completion of two successful test sets under the supervision of the test engineer.
- A demonstration of knowledge of the OWI. Candidate test operators **shall thoroughly read** the test OWI and **sign** a statement confirming that they have read and understand the OWI. Each **shall be issued** a personal copy of the OWI.
- Passing of a written test covering the OWI. The test **shall be administered** by the test engineer.

A copy of the written test, along with the signed statement and the training record, **shall constitute** verification of credentials. Training records **shall be kept** on file as proof of training. These records **shall include** training expiration dates and required refresher courses.

Category 1 Credentials **shall expire** after a period of 2 years. After that time, recredentialing **shall be required**.

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## Category 2 – Tester Maintenance and Modifications

Personnel seeking **Category 2 Credentials shall become** qualified and credentialed through training classes approved by the candidate's supervisor or through training classes completed during previous employment. Training in the following areas **shall be required**:

- Compressed Gases and Working with Compressed Gas Lines and Fittings
- Basic Electrical Wiring.

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<b>EMERGENCY PHONE NUMBERS</b>	
<b>Emergency.....</b>	<b>911</b>
<b>Medical Center.....</b>	<b>4-2390</b>
<b>Industrial Safety.....</b>	<b>4-0046</b>
<b>Chemical Spills.....</b>	<b>4-4357</b>
<b>Safety Monitor</b>	
<b>Building 4623.....</b>	<b>5-0358</b>