1 Scope  This test method is used to determine the total mass loss (TML) and collected volatile condensable material (CVCM) of materials when exposed to a heated vacuum environment. Mass loss may be due to outgassing of low molecular weight materials present in printed boards such as trapped plating solutions, improper lamination, and uncured adhesives which are known to cause contamination or corrosion of spacecraft equipment.

2 Applicable Documents


3 Test Specimens  The test specimen shall be cut into small pieces that can fit into the specimen boats and whose total mass shall be about 200 mg. If smaller masses are used the accuracy of the TML and CVCM determinations may be impaired. It is imperative that the specimens not be contaminated during the preparation process. Specimens are not to be handled with bare hands since human skin oils are volatile and condensable by this method thereby creating misleading TML and CVCM results. If there is any doubt about specimen contamination the specimens should be cleaned using solvents known to be nonreactive and that leave no residue. An average of at least three (3) samples shall be made for each test.

4 Apparatus or Material

4.1 Multiple specimen vacuum chamber capable of maintaining a vacuum of at least 7 X 10⁻³ Pa (5 X 10⁻⁵ Torr) with resistance heated copper bars capable of maintaining 125 °C ± 1 °C [257 °F ± 1.8 °F] during the 24 hour test run and typically containing 24 specimen chambers. Typically, three (3) of the specimen chambers are maintained as controls. The open end of each specimen chamber allows vapors from the specimen to pass through a hole into a collector chamber where the vapors are condensed on a collector plate that is maintained at 25 °C ± 1 °C [77 °F ± 2 °F] throughout the test. See ASTM E 595 for further details and requirements for the construction and cleaning of the test apparatus.

4.2 An analytical balance capable of measuring the specimens, boats, and collector plate mass to the nearest microgram (0.000001 gram).

4.3 Glass desiccator using active silica gel desiccant. Low vapor pressure grease shall be used for the ground glass joints.

4.4 Conditioning chamber capable of maintaining 50% ± 5% relative humidity and 23 °C ± 2 °C [73 °F ± 4 °F].

4.5 Prepared aluminum foil specimen boats.

4.6 Suitable cleaning solvents  Mixtures of 1:1:1 by volume chloroform:acetone:ethanol and 1:1 by volume acetone:ethanol solvent blends have been successfully used for cleaning and degreasing the apparatus, aluminum boats, and collector plates. All solvents shall be spectrophotometer grade purity or equivalent. See Annex A1 of ASTM E 595 for details regarding cleaning and storage procedures for the equipment used in this test.

4.7 Nitrogen gas, 99.9% pure, or better, with a dew point of -60 °C [-76 °F] or less. The nitrogen gas shall be filtered using a Molecular Sieve 5A or equivalent.

4.8 Wiping materials and swabs for cleaning. These material shall be preextracted using solvents with which they will be used.

4.9 Suitable gloves or finger cots to be used during sample preparation.

5 Procedure

5.1 Weigh a prepared aluminum foil boat to the nearest microgram and return it to the glass storage desiccator.

5.2 Weigh a prepared collector plate to the nearest microgram and mount it into its cooling plate receptacle.

5.3 Add the test specimen to the boat and condition the specimen at 50% ± 5% relative humidity at 23 °C ± 2 °C [73 °F ± 4 °F] for a minimum of 24 hours. Weigh the conditioned specimen and boat to the nearest microgram.
5.4 Exposure

5.4.1 Prior to operation, all temperature-vacuum apparatus such as the heating bar, separators, and cooling plates shall be clean and aligned.

5.4.2 Place the test specimen and boat into the specimen compartment in the temperature-vacuum system. Mount the respective cover plates of each specimen compartment and at least three (3) control compartments.

5.4.3 Close and activate the vacuum system and allow the system to evacuate to 7.0 \times 10^{-3} \text{ Pa} \ (5 \times 10^{-5} \text{ Torr}) or less within one hour. During this period, control of the collector plate temperature at 25 \ ^\circ \text{C} \pm 1 \ ^\circ \text{C} \ (77 \ ^\circ \text{F} \pm 2 \ ^\circ \text{F}) shall be achieved.

5.4.4 When the required vacuum has been achieved, turn on the heater bar and adjust the controller to heat the bar to 125 \ ^\circ \text{C} \pm 1 \ ^\circ \text{C} \ (257 \ ^\circ \text{F} \pm 2 \ ^\circ \text{F}) within 60 minutes.

5.4.5 Maintain the collector plate temperatures at 25 \ ^\circ \text{C} \pm 1 \ ^\circ \text{C} \ (77 \ ^\circ \text{F} \pm 2 \ ^\circ \text{F}) and the heater bar temperature at 125 \ ^\circ \text{C} \pm 1 \ ^\circ \text{C} \ (257 \ ^\circ \text{F} \pm 2 \ ^\circ \text{F}) for 24 hours. After this time period close the vacuum valve to the pumping system and turn off the current to the heater bars.

5.4.6 Open the vent valve and backfill with clean, dry nitrogen at a gage pressure of 10 to 30 kPa (2 to 4 psi) above atmosphere to rapidly cool the bars to 50 \ ^\circ \text{C} \ (122 \ ^\circ \text{F}) within two hours, nominally.

5.4.7 Turn off the collector-plate heat exchangers, return the vacuum chamber to room pressure using clean, dry nitrogen, and open the chamber. Remove the aluminum specimen boats and their respective collector plates and the control collector plates and immediately store in the dry desiccator (see 4.3).

5.4.8 After allowing the specimens to cool to approximately room temperature, but after no more than 1/2 hour, weigh the specimens and boats and the collector plates to the nearest 1 microgram within two minutes of removal from the desiccators.

5.5 Evaluation of Test

5.5.1 Measurements of the control collector plates are used to detect contamination and/or poor technique. Mass loss of greater than 20 micrograms is an indication of poor cleaning of the collector plates. Mass gain of greater than 50 micrograms is an indication of poor cleaning of elements of the apparatus, cross contamination between specimen compartments, or poor vacuum technique. Any change of 50 micrograms or greater calls for a review of or change in technique. All data obtained during such runs shall be discarded and the test rerun when the system(s) are corrected.

5.5.2 Calculation of Total Mass Loss (TML). Calculate the TML as follows:

Initial Mass

Final Mass

Specimens Weights:

\[ S_I = (S_I + B) - B \]

\[ S_F = (S_F + B) - B \]

Mass Loss (L) = \( S_I - S_F \)

Total Mass Loss (TML) (%) = \( (L/S_I) \times 100 \)

Where:

\[ B = \text{Mass of boat in grams} \]

\[ S_I = \text{Initial specimen mass in grams} \]

\[ S_F = \text{Final Specimen mass in grams} \]

\[ L = \text{Mass Loss in grams} \]

5.5.3 Calculate the Collected Condensable Volatile Material (CVCM) as follows:

\[ \text{Mass of condensable material } (C_O) = C_F - C_I \]

\[ \text{CVCM (\%)} = \left( C_O/S_I \right) \times 100 \]

Where:

\[ C_F = \text{Final mass of collector plate in grams} \]

\[ C_I = \text{Initial mass of collector plate in grams} \]

\[ C_O = \text{Mass of condensable material in grams} \]

\[ S_I = \text{Initial specimen mass in grams} \]

6 Notes

6.1 A useful outgassing test data sheet format can be found in Appendix X1 of ASTM E 595.

6.2 Additional information regarding this test method and suggested requirements for certain spacecraft applications can be found in NASA SP-R-0022.