



IPC-TM-650 TEST METHODS MANUAL

1 Scope This test method establishes a procedure for determining the thermal decomposition temperature (T_d) of base laminate materials using thermogravimetric analysis (TGA). Use of this test method for printed wiring boards or other composites may not yield comparative results.

2 Applicable Documents All terms and definitions in this document conform to IPC-T-50, *Terms and Definitions for Interconnecting and Packaging Electronic Circuitry*.

3 Test Samples

3.1 Sample Construction The sample may be an unclad laminate material or laminate material with copper completely removed and that has been cut (using water cooling/cleaning only, no oil) approximately square to fit into the TGA sample pan. Typical sample mass (weight) is 10 mg to 30 mg. Samples shall be cut to the specified size using appropriate procedures and equipment to minimize mechanical stress and/or thermal shock.

Note: Samples of the same mass but with a smaller surface area may lose mass at a slower rate.

3.2 Surface Preparation All edges of the sample shall be finished smooth and burr-free by sanding or equivalent (to allow the sample to rest completely flat on the sample pan). Use care to minimize the introduction of mechanical stress or heat to the sample.

3.3 Mass Measurement The accuracy of the mass measurements shall be within ± 0.01 mg.

4 Equipment/Apparatus or Material

4.1 Thermogravimetric Analyzer (TGA) Thermal gravimetric analysis instrument shall comprise the following:

4.1.1 Microbalance, null type, sensitive to 0.001 mg.

4.1.2 Furnace equipped with dry (dew point below -68°C [-90°F], moisture less than 3.5 ppm) nitrogen (less than 20 ppm oxygen) purge.

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4.1.3 Temperature programmer capable of providing controlled $10^\circ\text{C} \pm 0.1^\circ\text{C}$ [$18^\circ\text{F} \pm 0.18^\circ\text{F}$] per minute heating rate from ambient to 800°C [1472°F].

4.2 Mass Measurement Capability The TGA shall be capable of measuring mass to within 0.01 mg of actual value.

5 Procedure

5.1 Test Sample Preparation The test samples should be baked at $110^\circ\text{C} \pm 2^\circ\text{C}$ [$230.0^\circ\text{F} \pm 3.6^\circ\text{F}$] for 24 hr and placed in a desiccator for cooling to room temperature (equilibration) prior to testing. In standard lab conditions, the TGA test should be started within 15 minutes of removing test sample from the dessicator because samples may gain mass due to moisture absorption.

5.2 Equipment Startup and Calibration (Follow Manufacturer's Recommendations.)

5.2.1 Calibrate the balance to within ± 0.01 mg.

5.2.2 Calibrate the temperature sensor to within $\pm 1.0^\circ\text{C}$ [1.8°F].

5.2.3 Set the purge rate of 55 cc/min (0.9 mL/s). Run the TGA gas purge for 30 minutes before inserting a sample. The rate of flow of the gas in the cell will have a significant effect on the calibration, therefore, the instrument must be calibrated with the same flow rate as is used during the test. The temperature sensor should be positioned so that it does not come into contact with sample at any time. After the temperature sensor has been correctly positioned, the instrument can be calibrated. Neither the sensor position nor the flow rate should be subsequently changed.

5.3 Heating Sample

5.3.1 Place the sample in the TGA and measure its mass.

5.3.2 Heat the sample at a rate of $10^\circ\text{C}/\text{min}$ from ambient (not to exceed 50°C) to 550°C .

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5.3.3 Record the temperature, T_d (2%), at which the mass of the sample is 2.0% less than its mass measured at 50°C.

5.3.4 Record the temperature, T_d (5%), at which the mass of the sample is 5.0% less than its mass measured at 50°C.

6 Results Report the following information:

- a) The initial mass of the sample.
- b) The room temperature and relative humidity under which testing was conducted.
- c) The thermal decomposition temperature, T_d (2%).
- d) The thermal decomposition temperature, T_d (5%).

7 References

IPC-TM-650 Method 2.3.40, Thermal Stability

ASTM D-3850 Standard Test Method for Rapid Thermal Degradation of Solid Electrical Insulating Materials by Thermogravimetric Analysis