



IPC-TM-650 TEST METHODS MANUAL

1.0 Scope This method is designed to evaluate the resistance of dielectric materials to organic chemicals representative of those used in printed board fabrication and assembly. It is intended to provide a distinction between materials of varying chemical resistance and, when applicable, an indication of the degree of cure.

2.0 Applicable Documents

IPC-TM-650

Method 2.2.18, Determination of Thickness of Laminates by Mechanical Measurement

Weast, R.C., CRC Handbook of Chemistry and Physics, 65th Edition, E-37, 1984

3.0 Test Specimens

3.1 Number Unless otherwise specified, three specimens shall be used.

3.2 Size Unless otherwise specified, specimen shall be 50.8 x 50.8 mm \pm 0.5 mm [2.0 x 2.0 \pm 0.02 in] by the thickness of the material (see 5.1, Preparation). Edges shall be smooth, whether by the cutting procedure, or by sanding, or other means.

3.3 Location Specimen may be cut from anywhere in the sheet of material, except no specimens shall be taken closer than 25.4 mm [1.0 in] from any edge as laminated.

4.0 Apparatus or Material

4.1 Analytical balance with a draft shield and .001 gram or better resolution.

4.2 Air circulating oven, capable of 105 \pm 2°C [221 \pm 3.6°F].

4.3 Desiccator capable of maintaining an atmosphere of less than 30% relative humidity at 23°C [73°F]. See 6.2.5

4.4 Cutting apparatus such as diamond saw, shear paper cutter or router.

4.5 Edge smoothing apparatus such as 400 grit or finer sandpaper.

Number 2.3.4.2	
Subject Chemical Resistance of Laminates, Prepreg, and Coated Foil Products, by Solvent Exposure	
Date 12/94	Revision A
Originating Task Group MIL-P-13949 Test Methods Task Group (7-11b)	

4.6 One liter battery jar, with cover.

4.7 Metal tongs or forceps.

4.8 Metal rack (to support specimens vertically).

4.9 Metal support (e.g., mounting clip used for microsectioning).

4.10 Laminating press capable of the specified temperature and pressure.

4.11 Timer: 1 hour with 1 second resolution.

4.12 Timer: 60 seconds, 1 second resolution.

4.13 Solvent: Methylene Chloride, Reagent Grade or HPLC Grade. MC recovered by distillation back to reagent grade may be used. HPLC Grade shall be used for referee purposes.

4.14 Prepreg (for single sided laminate and coated foil test only).

4.15 Copper foil, 1 oz./ft² treated (for prepreg tests only).

4.16 Ventilation hood

4.17 Micrometer, capable of measuring to within 0.025 mm [0.001 in]

4.18 Water bath at 23 \pm 0.5°C [73 \pm 0.9°F].

4.19 Thermometer capable of measuring to within 0.1°C resolution.

4.20 Etching system capable of metallic cladding removal.

5.0 Procedure

5.1 Sample Preparation

5.1.1 Single-sided Laminate Single-sided laminate less than 0.5 mm [0.020 in] shall be laminated together with the clad surfaces facing out. Two plies minimum of prepreg shall be used. The prepreg shall be of the same basic resin type

IPC-TM-650		
Number 2.3.4.2	Subject Chemical Resistance of Laminates, Prepreg, and Coated Foil Products, by Solvent Exposure	Date 12/94
Revision A		

and of a thickness when cured not greater than 0.13 mm [0.005 in] total, using the prepreg manufacturer's recommended press cycle. Laminate over 0.5 mm [0.020 in] shall be treated like double clad laminate without lamination.

5.1.2 Resin-coated Foil Resin-coated foil samples shall be laminated together using at least two plies of prepreg between the coated sides of the foil. The prepreg shall be of the same basic resin type and of a thickness when cured not greater than 0.127 mm [0.005 in] total, using a press cycle and any subsequent post cure which meets the manufacturer's recommendations for both the coated foil and prepreg. (See 6.2.1.1)

For qualification and referee tests, a 0.50 ± 0.10 mm [0.02 in] composite core is to be built.

5.1.3 Laminate with Coated Foil Surfaces Single-sided laminate shall be tested after lamination as in 5.1.1. Double-sided laminate with coated foil on one side shall have the foil etched from the uncoated side and shall subsequently be laminated as 5.1.1. Double-sided laminate with coated foil on both sides shall be tested as is.

5.1.4 Prepreg Prepreg shall be laminated to a thickness of 0.50 ± 0.10 mm [0.020 in] using treated copper foil on both sides and following the prepreg manufacturer's recommended press and post cure cycle. (See 6.2.1.1) For prepreg less than 0.13 mm [0.005 in] thick when cured, at least two plies shall be bonded to either side of an etched laminate of a thickness such that the pressed sample satisfies the required thickness.

5.1.5 Double-sided Laminate Double-sided laminate shall be submitted for testing as is.

5.2 Specimen Preparation

5.2.1 Etching Samples, as specified in 3.0, shall be etched in accordance with standard industry practices. For referee purposes, 2.3.6, 2.3.7, 2.3.7.1, or 2.3.7.2 shall be used.

5.2.2 Cut the specimens to size per 3.2. Cutting shall be performed using apparatus and in such a manner that prevents edge damage. Edge smoothing is recommended to prevent excessive absorption.

5.2.3 Markings Each specimen shall be identified by marking with a lead pencil or equivalent technique which is not removed by the solvent and which doesn't affect the results.

5.3 Preconditioning

5.3.1 Measure and record nominal thickness of the test specimen using IPC-TM-650, Method 2.2.18.

5.3.2 Place the etched specimens vertically in the metal rack and dry the specimens in the oven for 60 ± 5 minutes at 105°C [221°F]. Air flow around each specimen must be assured.

5.3.3 Remove the specimens from the oven and immediately place in the desiccator to cool for 60 ± 30 minutes.

5.4 Test Condition The test shall be performed at standard laboratory conditions: $23 \pm 2^\circ\text{C}$ [$73 \pm 0.9^\circ\text{F}$] and $50 \pm 5\%$ R.H.

5.5 Measurement

5.5.1 Fill the battery jar with 750 ml of solvent and maintain the solvent temperature at $23 \pm 0.5^\circ\text{C}$ [$73 \pm 0.9^\circ$] using the water bath.

5.5.2 Remove the specimens individually from the desiccator and weigh to the nearest milligram on the analytical balance. Samples must be weighed within 60 seconds of removal from the desiccator. Record these weights as W_1 .

5.5.3 Place the rack into the beaker of solvent, start the 60 minute timer and using tongs place the specimens in the rack at suitable intervals (recommend approximately 2 minutes). Insure that the specimens are completely immersed.

5.5.4 Place the metal clip on the balance and tare it out.

5.5.5 After 10 minutes $+15, -0$ seconds, using the tongs remove the first specimen from the solvent and start the 60 second timer.

5.5.6 Slowly wag the specimen in the air. As soon as the specimen is free of surface wetness, but no longer than 30 seconds, place the specimen on the balance supporting it on the metal clip such that both surfaces of the specimen are exposed (placing the specimen flat on the balance pan is not permitted since evaporation from the side next to the pan will be reduced.)

IPC-TM-650		
Number 2.3.4.2	Subject Chemical Resistance of Laminates, Prepreg, and Coated Foil Products, by Solvent Exposure	Date 12/94
Revision A		

5.5.7 After 60 +5, -0 seconds from the time of removal from the solution, note the weight of the specimen and record it as W_2 .

5.5.8 Repeat 5.5.5 – 5.5.7 for the remaining specimens insuring the immersion time of each specimen is kept within the tolerance.

5.5.9 A fresh batch of solvent shall be used for no more than 18 specimens or for a period of time no longer than 8 hours.

5.6 Evaluation

5.6.1 Weight Gain

5.6.1.1 Subtract the dry weight of each specimen, W_1 , from the final weight, W_2 of each specimen after immersion. Record the weight gain in mg.

5.6.1.2 Calculate the average weight gain in mg.

5.6.2 Appearance Examine the specimens using 20/20 vision for any changes to the appearance of the material, such as hazing, whitening or swelling.

5.7 Report

5.7.1 Report the average weight gain in milligrams for the material tested.

5.7.2 Report the measured specimen thickness.

5.7.3 Report actual test conditions for temperature and humidity and the solvent temperature.

5.7.4 Report any anomalies in the test or any variations from the specified procedures or tolerances.

5.7.5 Report any changes in the visual quality of the specimens as determined in 5.6.2.

6.0 Notes

6.1 Safety Considerations See the MSDS sheet for the solvent used before running this test.

6.1.1 During test, the beaker with the test solvent should be covered or under a ventilation hood.

6.1.2 Dispose of the spent solvent in accordance with local regulations.

6.2 Factors Affecting Tests

6.2.1 Sample

6.2.1.1 Same Sample The results obtained on the same sheet of material may be significantly affected by the degree of cure of the material and the thermal history of the sample.

6.2.1.2 Variation Due to Thickness of Sample There is a very small difference due to sample thickness. This is under 10% on typical epoxy materials for 0.2 mm [0.008 in] to 1.5 mm [0.060 in] and under 5% on more chemical resistant material from 0.13 mm [0.005 in] to 1.5 mm [0.060 in].

6.2.2 Accuracy Since there are no standards for this type of test, the accuracy of the method cannot be established.

6.2.3 Precision the reproducibility of this test depends on the degree of control over the controlled variables and some other variables such as atmospheric pressure, which are generally not controlled in industrial laboratories. Precision between laboratories with 20% to 50% RH was 10% at the 90 mg level and 16% at the 25 mg level.

6.2.4 Desiccants Desiccants capable of 0.002 mg moisture levels include P_{205} , Mg (C10₄)₂, anhydrous BaO and fused KOH.

Some other desiccants such as CaCl₂ result in considerably higher moisture levels and may not be used in this test. (See Weast R.C., CRC Handbook of Chemistry and Physics, 65th edition, E-37, 1984.)

6.2.5 Humidity Considerations

6.2.5.1 Effect of Humidity Higher humidity will produce higher solvent pickup due to retarded solvent evaporation.

6.2.5.2 Control of Humidity Control of the critical environment can be obtained in uncontrolled laboratories using an enclosed balance with a saturated salt solution of Mg(NO₃)₂·6H₂O (for 50% RH).

All significant openings in the enclosure are sealed with tape and an inner door is fabricated with a small opening for sample entry and exit. A shallow pan is fabricated to occupy most of the balance floor without contacting the weighing

IPC-TM-650		
Number 2.3.4.2	Subject Chemical Resistance of Laminates, Prepreg, and Coated Foil Products, by Solvent Exposure	Date 12/94
Revision A		

pan. If the balance door is closed immediately after sample entry and exit, 50 ± 5% RH can be maintained.

Verification of conditions in the enclosure during a simulated test can be made using a rapid response humidity probe; however, this should be done with unconditioned specimens since solvents will affect the response of many humidity probes.

6.2.6 Temperature Considerations The effect of the solvent temperature on results is severe. A variation of 5°C [9°F] can result in an error as high as 50% relative to values determined at 23°C [73°F].

6.3 Consideration of Other Test Parameters

6.3.1 Using Same Solvent Shorter dwell times, e.g., 3 minutes, generally result in better discrimination between material of varying chemical resistance. However, test variability is generally increased. Shorter tests are excellent for side by side comparisons of materials.

Longer tests, e.g., 30 minutes, often do not differentiate adequately between materials, and while useful on homogeneous material, variability on material with surface coatings may be excessive.

6.3.2 Other Solvent Blends Other solvents and various methylene chloride based combinations have been commonly used in the industry. However, results and precision of the test may vary significantly and the added problem of variability in solution make-up is introduced.

6.4 Supersession This test method supersedes 2.3.4.2, dated 5/86, and 2.3.4.3, dated 5/86.

6.5 Desiccator Conditions The Test Methods Task Group determined that a great majority of test laboratories are unable to consistently hold the Relative Humidity in a desiccator to less than 20%. Based on data from participating company lab management, the lowest practically feasible RH for use with the affected IPC Test Methods is 30% maximum.