

# INTERNATIONAL STANDARD

**IEC**  
**61189-2**

Second edition  
2006-05

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**Test methods for electrical materials, printed  
boards and other interconnection structures  
and assemblies –**

**Part 2:  
Test methods for materials  
for interconnection structures**



Reference number  
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## **Test methods for electrical materials, printed boards and other interconnection structures and assemblies –**

### **Part 2: Test methods for materials for interconnection structures**

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# INTERNATIONAL ELECTROTECHNICAL COMMISSION

## TEST METHODS FOR ELECTRICAL MATERIALS, PRINTED BOARDS AND OTHER INTERCONNECTION STRUCTURES AND ASSEMBLIES –

### Part 2: Test methods for materials for interconnection structures

#### FOREWORD

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International Standard IEC 61189-2 has been prepared by IEC technical committee 91: Surface mounting technology, in cooperation with technical committee 52: Printed circuits (now disbanded), and technical committee 50: Environmental testing.

This second edition replaces the first edition, published in 1997, and its Amendment 1 (2000). It constitutes a technical revision.

The document 91/564/FDIS, circulated to the National Committees as Amendment 2, led to the publication of this new edition.



The text of this standard is based on the first edition, its amendment 1 and on the following documents:

FDIS	Report on voting
91/564/FDIS	91/572/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

The significant technical changes with respect to the previous edition concern the addition of several new tests in the following categories:

- C: Chemical test methods
- D: Dimensional test methods:
- E: Electrical test methods
- M: Mechanical test methods
- N: Environmental test methods
- X: Miscellaneous test methods

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

This standard forms part of a series and should be used in conjunction with other parts in the same series, all under the main title *Test methods for electrical materials, interconnection structures and assemblies*:

- Part 1: General test methods and methodology
- Part 2: Test methods for materials for interconnection structures
- Part 3: Test methods for interconnection structures (printed boards)
- Part 4: Test methods for electronic components assembling characteristics<sup>1</sup>
- Part 5: Test methods for printed board assemblies
- Part 6: Test methods for materials used in electronic assemblies

It should also be read in conjunction with IEC 60068: Environmental testing.

The committee has decided that the contents of this publication will remain unchanged until the maintenance result date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

<sup>1</sup> Under consideration.

## INTRODUCTION

IEC 61189 relates to test methods for printed boards and printed board assemblies, as well as related materials or component robustness, irrespective of their method of manufacture.

The standard is divided into separate parts, covering information for the designer and the test methodology engineer or technician. Each part has a specific focus; methods are grouped according to their application and numbered sequentially as they are developed and released.

In some instances test methods developed by other TCs (e.g. TC 50) have been reproduced from existing IEC standards in order to provide the reader with a comprehensive set of test methods. When this situation occurs, it will be noted on the specific test method; if the test method is reproduced with minor revision, those paragraphs that are different are identified.

This part of IEC 61189 contains test methods for materials used to produce interconnection structures (printed boards) and electronic assemblies. The methods are self-contained, with sufficient detail and description so as to achieve uniformity and reproducibility in the procedures and test methodologies.

The tests shown in this standard are grouped according to the following principles:

- P: preparation/conditioning methods
- V: visual test methods
- D: dimensional test methods
- C: chemical test methods
- M: mechanical test methods
- E: electrical test methods
- N: environmental test methods
- X: miscellaneous test methods

To facilitate reference to the tests, to retain consistency of presentation, and to provide for future expansion, each test is identified by a number (assigned sequentially) added to the prefix (group code) letter showing the group to which the test method belongs.

The test method numbers have no significance with respect to an eventual test sequence; that responsibility rests with the relevant specification that calls for the method being performed. The relevant specification, in most instances, also describes pass/fail criteria.

The letter and number combinations are for reference purposes, to be used by the relevant specification. Thus "2D01" represents the first dimensional test method described in this publication.

In short, for this example, 2 is the part of IEC standard (61189-2), D is the group of methods, and 01 is the test number.

A list of all test methods included in this standard, as well as those under consideration is given in Annex B. This annex will be reissued whenever new tests are introduced.

## TEST METHODS FOR ELECTRICAL MATERIALS, PRINTED BOARDS AND OTHER INTERCONNECTION STRUCTURES AND ASSEMBLIES –

### Part 2: Test methods for materials for interconnection structures

#### 1 Scope

This part of IEC 61189 provides a catalogue of test methods representing methodologies and procedures that can be applied to test materials used for manufacturing interconnection structures (printed boards) and assemblies.

#### 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60068-1:1988, *Environmental testing – Part 1: General and guidance*

IEC 60068-2-2:1974, *Environmental testing – Part 2: Tests – Tests B: Dry heat*

IEC 60068-2-78:2001, *Environmental testing – Part 2-78: Tests – Test Cab: Damp heat, steady state*

IEC 60093:1980, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials*

IEC 60243-1:1998, *Electrical strength of insulating materials – Test methods: Tests at power frequencies*

IEC 61189-3:1997, *Test methods for electrical materials, interconnection structures and assemblies – Part 3: Test methods for interconnection structures (printed boards)*

ISO 3274:1996, *Geometrical Products Specifications (GPS) – Surface texture: Profile method – Nominal characteristics of contact (stylus) instruments*

ISO 9001:2000, *Quality systems – Model for quality assurance in production, installation and servicing*

ANSI/UL-94:1993, *Standard for tests for flammability of plastic materials for parts in devices and appliances, Tests for*

#### 3 Accuracy, precision and resolution

Errors and uncertainties are inherent in all measurement processes. The information given below enables valid estimates of the amount of error and uncertainty to be taken into account.

Test data serve a number of purposes which include:

- monitoring a process;
- enhancing confidence in quality conformance;
- arbitrating between customer and supplier.

In any of these circumstances, it is essential that confidence can be placed upon the test data in terms of:

- accuracy: calibration of the test instruments and/or system;
- precision: the repeatability and uncertainty of the measurement;
- resolution: the suitability of the instruments and/or system for the test.

### 3.1 Accuracy

The regime by which routine calibration of the test equipment is undertaken shall be clearly stated in the quality documentation of the supplier or agency conducting the test, and shall meet the requirements of ISO 9001:2000.

The calibration shall be conducted by an agency having accreditation to a national or international measurement standard institute. There should be an uninterrupted chain of calibration to a national or international standard.

Where calibration to a national or international standard is not possible, round robin techniques may be used, and documented, to enhance confidence in measurement accuracy.

The calibration interval shall normally be one year. Equipment consistently found to be outside acceptable limits of accuracy shall be subject to shortened calibration intervals. Equipment consistently found to be well within acceptable limits may be subject to relaxed calibration intervals.

A record of the calibration and maintenance history shall be maintained for each instrument. These records should state the uncertainty of the calibration technique (in  $\pm$  % deviation) in order that uncertainties of measurement can be aggregated and determined.

A procedure shall be implemented to resolve any situation where an instrument is found to be outside calibration limits.

### 3.2 Precision

The uncertainty budget of any measurement technique is made up of both systematic and random uncertainties. All estimates shall be based upon a single confidence level, the minimum being 95 %.

Systematic uncertainties are usually the predominant contributor, and will include all uncertainties not subject to random fluctuation. These include:

- calibration uncertainties;
- errors due to the use of an instrument under conditions which differ from those under which it was calibrated;
- errors in the graduation of a scale of an analogue meter (scale shape error).

Random uncertainties result from numerous sources but can be deduced from repeated measurement of a standard item. Therefore, it is not necessary to isolate the individual contributions. These may include:

- random fluctuations such as those due to the variation of an influence parameter. Typically, changes in atmospheric conditions reduce the repeatability of a measurement;
- uncertainty in discrimination, such as setting a pointer to a fiducial mark, or interpolating between graduations on an analogue scale.

Aggregation of uncertainties: Geometric addition (root-sum-square) of uncertainties may be used in most cases. Interpolation error is normally added separately and may be accepted as being 20 % of the difference between the finest graduations of the scale of the instrument.

$$U_t = \pm \sqrt{(U_s^2 + U_r^2)} + U_i$$

where

$U_t$  is the total uncertainty

$U_s$  is the systematic uncertainty

$U_r$  is the random uncertainty

$U_i$  is the interpolation error

Determination of random uncertainties: Random uncertainty can be determined by repeated measurement of a parameter, and subsequent statistical manipulation of the measured data. The technique assumes that the data exhibits a normal (Gaussian) distribution.

$$U_r = \frac{t \times \sigma}{\sqrt{n}}$$

where

$U_r$  is random uncertainty

$n$  is the sample size

$t$  is the percentage point of the “ $t$ ” distribution (from 3.5), statistical tables

$\sigma$  is the standard deviation ( $\sigma_{n-1}$ )

### 3.3 Resolution

It is paramount that the test equipment used is capable of sufficient resolution. Measurement systems used should be capable of resolving 10 % (or better) of the test limit tolerance.

It is accepted that some technologies will place a physical limitation upon resolution (e.g. optical resolution)

### 3.4 Report

In addition to requirements detailed in the test specification, the report shall detail:

- the test method number and revision;
- the identity of the sample(s);
- the test instrumentation;
- the specified limit(s);
- an estimate of measurement uncertainty, and resultant working limit(s) for the test;
- the detailed test results;
- the test date, and operators' signature.

### 3.5 Student's “ $t$ ” distribution

Table 1 gives values of the factor “ $t$ ” for 95 % and 99 % confidence levels, as a function of the number of measurements. It is sufficient to use 95 % limits, as in the case of the worked examples shown in Annex A.

**Table 1 – Student's "t" distribution**

Sample size	t value 95 %	t value 99 %		Sample size	t value 95 %	t value 99 %
2	12,7	63,7		14	2,16	3,01
3	4,3	9,92		15	2,14	2,98
4	3,18	5,84		16	2,13	2,95
5	2,78	4,6		17	2,12	2,92
6	2,57	4,03		18	2,11	2,9
7	2,45	3,71		19	2,1	2,88
8	2,36	3,5		20	2,09	2,86
9	2,31	3,36		21	2,08	2,83
10	2,26	3,25		22	2,075	2,82
11	2,23	3,17		23	2,07	2,81
12	2,2	3,11		24	2,065	2,8
13	2,18	3,05		25	2,06	2,79

### 3.6 Suggested uncertainty limits

The following target uncertainties are suggested:

- a) Voltage < 1 kV:  $\pm 1,5 \%$
- b) Voltage > 1 kV:  $\pm 2,5 \%$
- c) Current < 20 A:  $\pm 1,5 \%$
- d) Current > 20 A:  $\pm 2,5 \%$

#### Resistance

- e) Earth and continuity:  $\pm 10 \%$
- f) Insulation:  $\pm 10 \%$
- g) Frequency:  $\pm 0,2 \%$

#### Time

- h) Interval < 60 s:  $\pm 1 \text{ s}$
- i) Interval > 60 s:  $\pm 2 \%$
- j) Mass < 10 g:  $\pm 0,5 \%$
- k) Mass 10 g to 100 g:  $\pm 1 \%$
- l) Mass > 100 g:  $\pm 2 \%$
- m) Force:  $\pm 2 \%$
- n) Dimension < 25 mm:  $\pm 0,5 \%$
- o) Dimension > 25 mm:  $\pm 0,1 \text{ mm}$
- p) Temperature < 100 °C:  $\pm 1,5 \%$
- q) Temperature > 100 °C:  $\pm 3,5 \%$
- r) Humidity 30 to 75 % RH:  $\pm 5 \%$  RH

#### Plating thicknesses

- s) Backscatter method:  $\pm 10 \%$
- t) Microsection:  $\pm 2 \mu\text{m}$
- u) Ionic contamination:  $\pm 10 \%$

## 4 Catalogue of approved test methods

This standard provides specific test methods in complete detail to permit implementation with minimal cross-referencing to other specific procedures. The use of generic conditioning exposures is accomplished in the methods by reference, for example IEC 61189-1 and IEC 60068, and when applicable, is a mandatory part of the test method standard.

Each method has its own title, number and revision status to accommodate updating and improving the methods as industry requirements change or demand new methodology. The methods are organized in test method groups and individual tests.

## 5 P: Preparation/conditioning test methods

### 5.1 Test 2P01: Dry heat (under consideration)

### 5.2 Test 2P02: Solder float stress (under consideration)

## 6 V: Visual test methods

## 7 D: Dimensional test methods

### 7.1 Test 2D01: Thickness of base materials and rigid boards

#### 7.1.1 Object

This test method covers the procedure for the determination of the thickness of base materials, clad or unclad.

#### 7.1.2 Test specimens

Standard sheet sizes of metal-clad or unclad base materials.

Standard panel sizes of metal-clad or unclad base materials.

#### 7.1.3 Test apparatus and material

A suitable micrometer having a resolution of 0,01 mm or better shall be used.

#### 7.1.4 Procedure

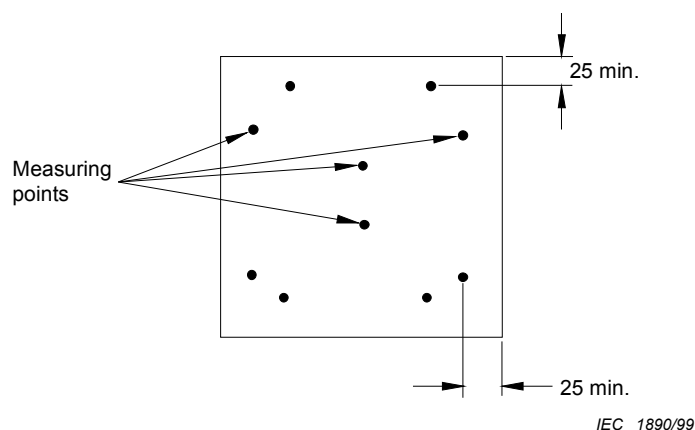
##### a) General conditions

- Test specimens shall be placed between the two faces of the micrometer, so that the whole face of the pressure-foot will fall within the area of the material. The pressure-foot shall be lowered gently, slowly and with great care onto the test specimen so that all punching effect is avoided.
- No stress shall be imposed by hand on the instrument or the material when a reading is being taken. The reading shall be taken as soon as the pointer has ceased to move. It is necessary to take care in avoiding parallax errors and vibrations which may significantly affect the results.

##### b) Method 1

- This procedure is intended for the thickness measurement of the sheets of metal-clad or unclad base materials.
- The specimen shall be held vertically or horizontally.

- Thickness to the nearest 0,01 mm at two points 25 mm or more inside each edge, at eight points, and additionally at two points in the middle parts, so that a total of 10 points, shall be measured as shown in Figure 1.
- The measurement shall be made twice at each point and the mean value shall be determined as the thickness of each point.
- For automatic thickness inspection, continuous measuring shall be performed in three measuring tracks parallel to the longitudinal axis of the sheet, two at least 25 mm from the longitudinal edges and the third near the midline.



**Figure 1 – Thickness measuring points**

**c) Method 2**

- This procedure is intended for the thickness measurement of panels of metal-clad or unclad base materials. The thickness of the specimens held vertically or horizontally shall be measured at the places which are agreed between the interested parties.

**7.1.5 Report**

The report shall include:

- a) the test method number and revision;
- b) the date of the test;
- c) the identification of the material tested;
- d) a statement certifying that the test was carried out for as-received metal-clad or unclad base materials;
- e) the thicknesses measured and the nominal thickness with its tolerance;
- f) any deviation from this test method;
- g) the name of the person conducting the test.

**7.1.6 Additional information**

The use of a micrometer with a damping device, or controlled rate of movement of the pressure-foot, is advantageous.



## 8 C: Chemical test methods

### 8.1 Test 2C01: Resistance to sodium hydroxide of base materials

#### 8.1.1 Object

The purpose of this test method is to provide a procedure for determining the alkaline resistance of base materials by exposure to a sodium hydroxide solution.

#### 8.1.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge of the sheet.
- b) Specimens shall be prepared from a sample of metal-clad base material from which the metal has been completely removed by any appropriate method reflecting usual practice.
- c) Specimen size is  $(50 \pm 2)$  mm in both length and width, and shall be cut out using a fine saw to give the edges a smooth finish.
- d) A minimum of three specimens shall be used.

#### 8.1.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) an appropriate alkaline-proof container which contains an analytical grade sodium hydroxide solution maintained at a temperature of  $(40 \pm 2)$  °C at a concentration by weight of  $(3 \pm 0,2)$  %. In order to ensure that the concentration remains within the tolerance, the solution must be prepared daily. The number of specimens tested per litre of solution shall not be more than 50;
- b) a rack to hold specimens upright in the container. The design of the rack shall allow maximum exposure of the specimen surfaces to the solution;
- c) a clean dry gauze, cloth or paper to wipe off the water from the specimen surfaces;
- d) a fine blade saw for the sample preparation.

#### 8.1.4 Procedure

Place the specimens in the rack then in the sodium hydroxide solution for  $3 \text{ min} \pm 20 \text{ s}$ .

Take the rack out of the sodium hydroxide solution and quickly rinse the specimens under running water for a minimum of 5 min.

Wipe the water from the specimen surfaces completely with a clean dry gauze, cloth or paper.

Immediately make a visual check for colour change, swelling, blistering and/or delamination.

#### 8.1.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the changes in surface appearance, if any;
- e) any deviation from this test method.

### 8.1.6 Additional information

Sodium hydroxide is a powerful alkaline chemical. It shall be handled with care, avoiding eye and skin contact by wearing protective glasses and chemically resistant gloves.

## 8.2 Test 2C02: Gel time of epoxy based prepreg materials

### 8.2.1 Object

The purpose of this test method is to provide a means for determining the gel time of epoxide resin impregnated reinforcement cured to the B-stage used in the manufacturing of laminate and printed boards.

### 8.2.2 Test specimens

A number of pieces approximately 100 mm square or another convenient size, in order to yield approximately 1 g of dry resin, shall be cut from areas uniformly distributed across the width of the sheet or roll, but excluding the area within 25 mm of each edge or selvage.

### 8.2.3 Test apparatus and material

The following test apparatus and materials shall be used:

- a) heating plate capable of maintaining a temperature of  $(170 \pm 0,5) ^\circ\text{C}$ ;
- b) timer, capable of determining time within  $\pm 1$  s;
- c) wooden stick, pointed, approximately 3 mm in diameter;
- d) a measure of capacity for 0,3 g to 0,4 g resin powder;
- e) sieve, 50 mesh per inch.

### 8.2.4 Procedure

Detach the dry resin from the prepreg (B-stage) by folding or crushing. Remove any glass fibre present by sieving alternatively, in the case of materials too soft to detach dry resin by crushing, the resin required may be obtained by pressing the folded stack of material in contact with the heating plate and squeezing out the melted resin.

Remove any glass fibre present by sieving.

Adjust the heating plate or equivalent to  $170 ^\circ\text{C}$  and allow to stabilize at that temperature.

Using the measure of capacity a quantity of 0,3 g to 0,4 g resin powder shall be taken.

Pour the measured dry resin in the form of a small cone on one spot of the heating plate and start the timer immediately. If the alternative method given above is used, the timer shall be started at that moment when the folded stack is brought in contact with the heating plate.

Stir the resin, using a wooden stick approximately 3 mm in diameter, holding the stick as near vertical as possible and mixing the centre as well as the edges of the melted resin. While stirring, the diameter of the pool of melting resin shall not exceed 25 mm.

At the approach of the gel point the resin becomes tacky and forms strings when pulling the stick out. The gel point is reached when it no longer forms strings when pulling the stick out, and is no longer tacky but still elastic. At this point, the timer is stopped and the elapsed time measured in seconds is taken as the gel time. When used as a reference, three separate measurements shall be carried out and the average recorded as gel time.

### 8.2.5 Report

The report shall include:

- a) the test method number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the gel time in seconds (average);
- e) any deviation from this test method.

### 8.2.6 Additional information

The determination of gel time may also be carried out using a sample of the resin contained in a rotational viscometer, in which case more information about the flow characteristics of the resin may be obtained. If this method is used, the viscosity value corresponding to the gel point shall be defined by determination of the correlation with the test described above.

## 8.3 Test 2C03: Resin content of prepreg materials by treated weight

### 8.3.1 Object

The purpose of this test method is to provide a means for measuring the resin content of resin impregnated reinforcement cured to the B-stage, only if the weight of uncoated reinforcement is known. This method is applicable to both organic and inorganic reinforcements.

### 8.3.2 Test specimens

- a) Specimens shall be taken from the roll or sheet in such a way that they are at least 25 mm from the edge.
- b) Four specimens  $(100 \pm 0,2) \text{ mm} \times (100 \pm 0,2) \text{ mm}$  shall be taken at equal spacing across the web for rolls or from different areas of sheeted material.

### 8.3.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) analytical balance with a 0,001 g or better resolution;
- b) desiccator (stabilization chamber) capable of maintaining 25 % relative humidity (RH) or less at room temperature.

### 8.3.4 Procedure

#### 8.3.4.1 Determination of weight of reinforcement

The weight of 1 dm<sup>2</sup> reinforcement may be determined by one of the two methods described below:

##### 8.3.4.1.1 Method 1

Determine the weight of the reinforcement from the actual length, width and weight of roll of reinforcement material:

$$W_B = 10 \times \frac{W_R}{L \times D}$$

where

$W_B$  is the weight of 1 dm<sup>2</sup> reinforcement (g)

$W_R$  is the roll weight (kg)

$L$  is the roll length (m)

$D$  is the roll width (m)

#### 8.3.4.1.2 Method 2

Determine the weight from median statistical or typical reinforcement weight in grams per metre:

$$W_B = 0,01 W$$

where

$W_B$  is the weight of 1 dm<sup>2</sup> reinforcement (g)

$W$  is the weight of 1 m<sup>2</sup> (g)

#### 8.3.4.2 Determination of total weight of prepreg

Determine actual reinforcement weight at the beginning of the roll, using the same measurement technique as described in this method.

All the above methods consider any finishes applied to the reinforcement as part of the reinforcement.

The specimen shall be desiccated unless the prepreg material is tested within 10 min of manufacture to prevent the absorption of moisture.

Determine and record the total weight of the four specimens to the nearest 0,001 g.

#### 8.3.4.3 Resin content

The resin content shall be calculated as follows:

$$C_R = \left( 1 - \frac{W_B}{W_T} \right) \times 100$$

where

$C_R$  is the resin content (%)

$W_B$  is the weight of the reinforcement (g/dm<sup>2</sup>)

$W_T$  is the weight of the treated prepreg (g/dm<sup>2</sup>)

#### 8.3.5 Report

The report shall include:

- the test method number and revision index;
- the testing date;
- the identification of the material tested;
- the weight of the treated specimen to the nearest 0,001 g and the resin content to the nearest 0,1 %;
- any deviation from this test method.

### 8.3.6 Additional information

The volatile content of the prepreg (excluding moisture) is considered as part of the treated product using this method. If it is desired to exclude volatile content the specimen should be baked at an appropriate time and temperature.

The accuracy of this test method for determination of resin content is dependent primarily upon the accuracy of the basic weight of the reinforcement.

The effect of static charges may present a serious problem in weighing material which has been stored if the specimen is larger than the sample pan of the analytical balance.

If it is desired to check variation across the width of the roll, four specimens may be cut down the roll at each location to be tested.

In most cases, the amount of organic material from the reinforcement is negligible; however, special attention should be paid to materials such as reinforcement containing finishes, which have a substantial amount of organic content (5 % or more), and for organic reinforcements which may have significant moisture content.

The resin content test by sublimation, Test 2C10, is a more accurate measurement of resin content and shall be used for reference purposes.

## 8.4 Test 2C04: Volatile content of prepreg materials

### 8.4.1 Object

The purpose of this test method is to provide a means for measuring the volatile content of resin impregnated B-stage reinforcement in the manufacturing of laminate and multilayer printed boards.

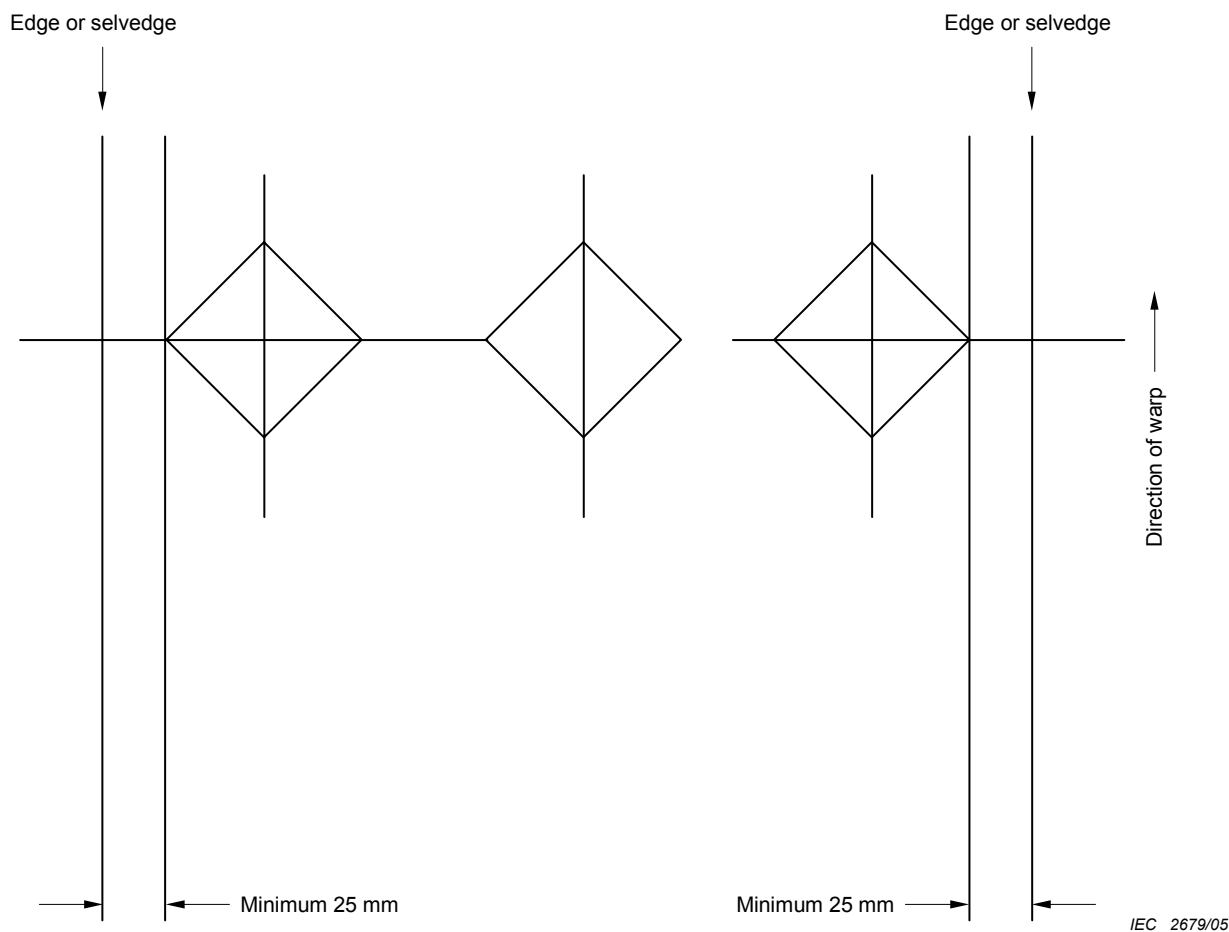
### 8.4.2 Test specimens

Three specimens  $(100 \pm 0,2) \text{ mm} \times (100 \pm 0,2) \text{ mm}$  shall be cut from positions along a line perpendicular to the machine direction of reinforcement, with the diagonals of the specimens parallel to the machine (warp) and cross-machine directions (weft), and no closer than 25 mm from the edge of the prepreg sheet or roll, see Figure 2. A hole approximately 3 mm in diameter shall be punched in the corner of each specimen.

### 8.4.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) analytical balance with a 0,001 g or better resolution;
- b) an air circulating oven capable of achieving a temperature of up to 250 °C and controllable within  $\pm 3 \text{ °C}$ ;
- c) a desiccator (stabilization chamber) capable of maintaining 25 % RH or less at room temperature;
- d) appropriate hanging metal hooks to hang specimens.



**Figure 2 – Position of specimens**

#### 8.4.4 Procedure

Weigh each specimen including a hanging metal hook to the nearest 0,001 g and record as  $M_1$ .

Hang each specimen with the metal hook in the circulating air oven at  $(163 \pm 3)^\circ\text{C}$  for  $(15 \pm 1)$  min except for unmodified polyimide prepreg. For unmodified polyimide prepreg, the drying condition in the oven shall be at  $(225 \pm 3)^\circ\text{C}$  for  $(15 \pm 1)$  min.

Remove each specimen with hook from the chamber, one at a time, and cool to room temperature in a desiccator. Reweigh the specimen including the hanging metal hook within 2 min after removal from the desiccator to the nearest 0,001 g and record as  $M_2$ .

The volatile content of each specimen shall be calculated as follows:

$$C_V = \frac{(M_1 - M_2)}{M_1} \times 100$$

where

$C_V$  is the volatile content (%)

$M_1$  is the weight before oven conditioning (g/100 cm<sup>2</sup>)

$M_2$  is the weight after oven conditioning (g/100 cm<sup>2</sup>)

### 8.4.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the average of the three volatile content values in per cent (%);
- e) the temperature of the circulating air oven;
- f) any deviation from this test method.

### 8.4.6 Additional information

The 163 °C oven temperature for epoxide and 225 °C oven temperature for unmodified polyimide are for these resin systems only. For other resin systems, the temperature shall be determined upon agreement between the purchaser and supplier.

## 8.5 Test 2C05: Blistering during heat shock

### 8.5.1 Object

To determine the ability of the copper-clad laminate to withstand specified heat shocks, simulating soldering processes, without blistering.

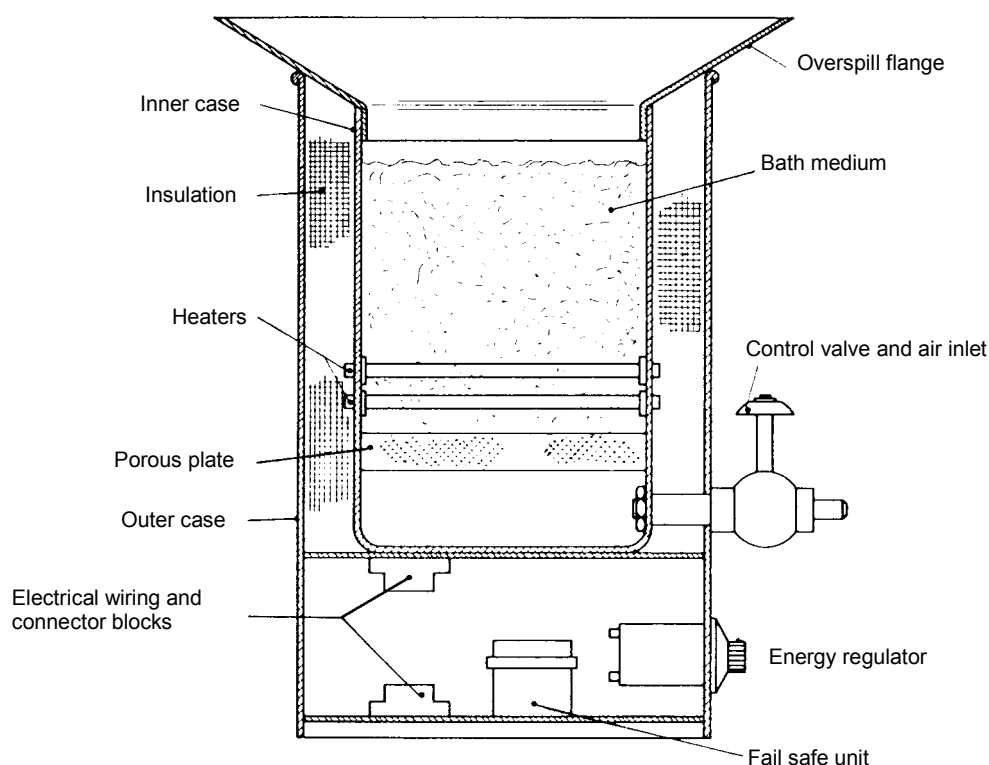
### 8.5.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be a square of  $(50 \pm 5)$  mm cut from a sample of the copper-clad base material and shall be unetched. For double-sided metal-clad base material, each face shall be tested separately with separate test specimens. For each side of the laminate tested, three specimens shall be used.

### 8.5.3 Test apparatus and materials

A bath meeting one of the following requirements:

- a) a liquid bath of well-stirred silicone liquid or equivalent fluid kept throughout the test at  $(260^{+5}_0)$  °C. The temperature shall be measured at  $(25 \pm 2,5)$  mm below the surface;
- b) a fluidized sand bath (see Figure 3) of suitable design kept at a temperature of  $(260^{+5}_0)$  °C measured in approximately the same location that will be occupied by the specimen;
- c) a suitable solder bath of depth not less than 40 mm shall be used. If circular, the bath shall be not less than 120 mm in diameter, and if rectangular, not smaller than 100 mm × 75 mm. The bath shall be protected from draughts. The temperature of the solder shall be kept at  $(260^{+5}_0)$  °C throughout the test. The temperature shall be measured at a depth of  $(25 \pm 2,5)$  mm below the surface. The bath shall contain solder of nominal 60/40 or 63/37 tin-lead composition. The characteristics of the solder shall be as follows:
  - completely solid 183 °C;
  - completely liquid 188 °C.



IEC 2680/05

#### PRINCIPLES OF OPERATION

Small solid particles can be readily "fluidized" by means of a suitable gas (air) stream. A diagrammatic cross-section of a fluidized sand bath is shown above. Clean, dry air at a constant pressure of about 2 N/cm<sup>2</sup> from a pump or from an air line is supplied via a control valve to a chamber beneath the diffuser (porous plate). This porous plate ensures a uniform flow of air across the full section of the container and acts also as a support plate for the solid sand bed.

As the control valve is slowly opened, the solid sand bed remains undisturbed and the air finds its way between the particles; under such conditions the pressure drop is proportional to the rate of flow of air. As the valve is opened further, the air drag on the particles will cause them to separate and the whole mass of the bed can be seen to have expanded. The bed now behaves as a fluid and is said to be "fluidized". Further opening of the valve is not accompanied by an increase in pressure drop, which remains constant at a value corresponding to the head of the column of particles, but the bed becomes more turbulent and will have the appearance of boiling liquid. The best heat transfer and most uniform temperatures are obtained when the bath is in this "boiling" state.

**Figure 3 – Fluidized sand bath**

### 8.5.4 Procedure

#### 8.5.4.1 Liquid bath procedure

The specimen shall be held in a horizontal position, at a depth of (25 ± 2,5) mm in a holder of heat capacity low enough to avoid the temperature of the fluid falling below 260 °C. The specimen shall be totally immersed in the fluid for the time given in the relevant specification and, immediately after removal, shall be inspected visually for blistering and delamination of the base material.

#### 8.5.4.2 Fluidized sand bath procedure

The specimen shall be immersed edgewise, that is with its surface at a right angle to the bath surface, for the time given in the relevant specification and, immediately after removal, shall be inspected for blistering and for delamination of the base material.



#### 8.5.4.3 Solder bath procedure

The specimen shall be floated on the surface of the clean molten solder with the metal-clad face of the specimen down for the time given in the relevant specification and, immediately after removal, shall be inspected for blistering and for delamination of the base material. The solder bath procedure shall be the method.

#### 8.5.5 Report

The report shall include:

- a) the test number and revision index;
- b) the test date;
- c) the identification of the material tested;
- d) the heat shock method used;
- e) the time during which the specimen was exposed to the heat shock;
- f) whether the specimens have blistered or delaminated; a border of 5 mm around the edge of the specimen is excluded from the requirement;
- g) any deviation from this test method.

#### 8.5.6 Additional information

Safety precautions: avoid skin contact with molten solder.

Cleaning of the surface of the molten solder can easily be done using a strip of unclad laminate.

### 8.6 Test 2C06: Flammability, vertical burning test for rigid materials

#### 8.6.1 Object

This test method is intended as a laboratory quality control technique using a low energy source of ignition. Results from this test should not be used to attempt to predict the behaviour of materials in a large-scale fire.

This test should be used for materials having good resistance to ignition. The test is carried out using a small test flame having an intensity similar to that of an actual source of fire.

Timings measured by this test are an indication of the ability of the material(s) to self-extinguish. There is no correlation with other properties of the material(s), such as the oxygen index.

Materials suitable for testing in accordance with this technique include rigid substrates and rigid substrates in combination with any surface coating(s).

#### 8.6.2 Test specimens

The test specimens shall be prepared from a sample of the metal-clad base material under test. The metal shall be completely removed using any etching method of commercial practice.

The specimen strip shall be  $(125 \pm 5)$  mm long and  $(13 \pm 0,3)$  mm wide. The edges shall be smooth. The corners of the specimens shall be rounded with a radius not exceeding 1,3 mm. The thickness of the sample will prejudice the results obtained.

A minimum of 10 specimens shall be tested. However, it is usual to take a total of 20 specimens for conditioning and testing to cover the eventuality of a failure during the test of the first set of specimens.

### 8.6.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) A draught-free room, test chamber or enclosure which provides a means of venting the fumes from burning specimens. A hood may be used, but its exhaust fan shall be disabled during the tests and allowed to operate only between tests in order to clear fumes. Subdued light is advantageous.
- b) The igniting source consisting of a blue flame,  $(20 \pm 2)$  mm high, produced using a laboratory burner (Bunsen or Tirril burner) having a tube with a length of 100 mm and an inside diameter of  $(9,5 \pm 0,5)$  mm. The tube shall not be equipped with end attachments such as stabilizers.
- c) A supply of technical grade methane gas with a suitable regulator and meter to produce a uniform gas flow. If natural gas is used as an alternative to methane, it should have a heat content of approximately 37 MJ/m<sup>3</sup>. This has been found to produce similar results.
- d) The required flame shall be obtained by adjusting the gas supply and air inlets of the burner until a yellow-tipped blue flame of the specified height is produced, and then by increasing the air supply until the yellow tip has just disappeared. The height of the flame shall then be measured again and corrected if necessary.
- e) A test fixture shall be comprised of a ring stand with two clamps or similar apparatus which is adjustable for vertical positioning of the specimen. Each specimen is to be held by clamping the upper 6 mm of the specimen with the long dimension oriented vertically, so that the lower end of the specimen is 10 mm above the top of the burner tube and 300 mm above a horizontal layer of dry tissue paper (50 mm × 50 mm swatch). An adjustable, movable holder maintains the burner tube centrally under the lower end of the specimen to an angle of 5° and the 10 mm distance between the lower end of the specimen and the top of the burner is to be maintained during the flame applications.
- f) A hand-operated timing device with a resolution of 1 s or better.

### 8.6.4 Procedure

Ten specimens shall be preconditioned in accordance with the requirements of 5.3 of IEC 60068-1 for a period of 48 h as a referee and 24 h for normal quality conformance prior to testing. The detail requirements are

- a) a temperature of 15 °C to 35 °C;
- b) a humidity of 25 % RH to 75 % RH;
- c) an air pressure of 86 kPa to 106 kPa.

Fluctuations shall be kept to a minimum.

The remaining 10 specimens shall be preconditioned in a circulating air oven for 24 h at  $(125 \pm 2)$  °C. They shall then be allowed to cool in a desiccator until specimens reach room temperature prior to testing.

Each specimen shall be held in the test fixture by clamping the upper 6 mm of the specimen with the long direction oriented vertically so that the lower end of the specimen is 10 mm above the top of the burner tube and 300 mm above a horizontal layer of dry tissue papers (50 mm × 50 mm swatch).

The burner, in a remote position from the specimen, shall be adjusted by controlling the gas supply and air inlets of the burner until a yellow-tipped blue flame  $(20 \pm 2)$  mm in height is produced. The air supply is then increased until the yellow tip has disappeared. The height of the flame shall then be measured again and corrected if necessary.

The burner shall be placed centrally beneath the lower end of the specimen and allowed to remain for 10 s. The burner shall then be moved at least 150 mm away from the specimen, and the time taken by the specimen to self-extinguish shall be measured. This shall be defined as the time from removal of the test flame from the specimen until the time when the specimen ceases to burn. Record the burn time on the laboratory pro forma in Annex C.

When the specimen ceases to burn, the burner shall immediately be replaced in its original position beneath the specimen. After 10 s, the test flame shall again be withdrawn and the duration of flaming shall again be measured. Record the burn time on the laboratory pro forma in Annex C.

If the test flame is extinguished during either application, it shall be reignited immediately and reapplied so that the total time of application is still 10 s. There shall be no more than three applications of the test flame during any 10 s ignition period, otherwise the material cannot be evaluated by this technique.

If the specimen drips molten or flaming material during either application of the test flame, the burner may be tilted to an angle of up to 45° and also slightly withdrawn from one of the 13 mm sides of the specimen during the flame application to avoid material dripping into the tube of the burner.

If the specimen drips molten or flaming material, or is consumed during the test, the burner shall be hand-held and the 10 mm distance between the bottom of the specimen and the top of the burner tube shall be maintained throughout the flame application. Any molten strings of material shall be ignored, and the flame shall be applied to the major part of the specimen. Record observed dripping or other significant observations on the laboratory pro forma in Annex C.

If the total of the ten burn times meets the requirements of the relevant specification but individual burning times exceed the relevant requirements, a further set of five specimens shall be tested. If the second set meets all the requirements, these requirements shall be deemed to be satisfied.

If the total of ten burning times for any set of five specimens exceed the specified requirements by no more than 5 s, a second set of five specimens shall be tested, and if the requirements for total and individual burning times are met, these requirements shall be deemed to be satisfactory.

### 8.6.5 Report

In addition to the general requirements for reporting, the report shall include

- a) test number and revision;
- b) identification of the material tested;
- c) testing date;
- d) the thickness of the specimen;
- e) the duration of flaming of each specimen after the first removal of the test flame;
- f) the duration of flaming of each specimen after the second removal of the test flame;
- g) whether the specimen burns up to the holding clamp;
- h) whether the specimen drips flaming particles which ignite the tissue paper;
- i) any deviation from this test method;
- j) the name of the person performing the test;
- k) the type of combustion. Flaming combustion is the combustion of the specimen in the gaseous phase with the emission of light. Glowing combustion of the specimen is the combustion without flame;
- l) the evaluated results.

### 8.6.6 Additional information

Annex C shows a suggested pro forma for reporting.

There are obvious hazards associated with flammability testing. Training of test operators, and familiarity with laboratory safety procedures is of paramount importance.

All fire effluent should be considered to be toxic, for the purposes of safety if not in fact.

Uncertainty of measurement calculations for burn times, although a variable, prove to be impractical. The result of the test is an attribute; the FV-0, FV-1 rating etc.

It is understood that a nominal substrate thickness of 1,6 mm will be used throughout the industry. Differences in thickness will prejudice test results.

American industry requirements (Underwriter's Laboratory Specification ANSI/UL-94) detail a specimen width of 12,7 mm to 13,2 mm.

The specimen width of  $(13 \pm 0,3)$  mm has therefore been chosen since this will accommodate ANSI/UL-94.

The smoothness of the specimen edges can be critical to the performance of the specimen. A polished finish is recommended. A rough finish (for example blanked) will significantly degrade performance due to the increase in surface area available to the flame.

Small-scale flammability tests, such as the one described herein, are an indicator of the behaviour of the material(s) tested. Fire integrity of equipments in which printed boards are used can only be assessed by equipment level testing.

Materials in combination may produce results that are different to those of the separate materials.

A material that is rated FV-1 or FV-2 when bonded to an inert substrate may produce an FV-0 performance (for example rigid polyimide/glass constrained with copper-invar). A FV-0 material in combination with a surface coating (for example solder resist) may be degraded to FV-1.

## 8.7 Test 2C07: Flammability; horizontal burning test for rigid materials

### 8.7.1 Object

This test method is intended as a laboratory quality control technique using a low energy source of ignition. Results from this test should not be used to attempt to predict the behaviour of materials in a large-scale fire.

This test is significantly less onerous than the similar vertical burn test and is intended to be used for materials having a limited resistance to ignition. The test is carried out using a small test flame having an intensity similar to that of an actual source of fire. This method does have an obvious application for printed board assemblies used in a horizontal configuration. Otherwise, due consideration should be given to its applicability.

Timings measured by this test are an indication of the ability of the material(s) to self-extinguish. There is no correlation with other properties of the material(s), such as the oxygen index.

Materials suitable for testing in accordance with this technique include rigid substrates and rigid substrates in combination with any surface coating(s).

### 8.7.2 Test specimen

The test specimens shall be prepared from a sample of the metal-clad base material under test. The metal shall be completely removed using any etching method of commercial practice.

The specimen strip shall be  $(125 \pm 5)$  mm long and  $(13 \pm 0,3)$  mm wide. The edges shall be smooth. The corners of the specimens shall be rounded with a radius not exceeding 1,3 mm. The thickness of the sample will prejudice the results obtained.

The specimens shall be marked with an indelible line (for example by scribing) which is perpendicular to the longitudinal axis, and which is  $(25 \pm 0,5)$  mm away from the end which is to be ignited.

A minimum of four specimens shall be tested.

### 8.7.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) A draught-free room, test chamber or enclosure which provides a means of venting the fumes from burning specimens. A hood may be used, but its exhaust fan shall be disabled during the tests and allowed to operate only between tests in order to clear fumes. Subdued light is advantageous.
- b) The igniting source consisting of a blue flame,  $(25 \pm 1)$  mm high, produced using a laboratory burner (Bunsen or Tirril burner) having a tube with a length of 100 mm and an inside diameter of  $(9,5 \pm 0,5)$  mm. The tube shall not be equipped with end attachments such as stabilizers.
- c) A supply of technical grade methane gas with a suitable regulator and meter to produce a uniform gas flow. If natural gas is used as an alternative to methane, it should have a heat content of approximately 37 MJ/m<sup>3</sup>. This has been found to produce similar results.

The required flame shall be obtained by adjusting the gas supply and air inlets of the burner until a yellow-tipped blue flame of the specified height is produced, and then by increasing the air supply until the yellow tip has just disappeared. The height of the flame shall then be measured again and corrected if necessary.

- d) A test fixture comprised of a ring stand with two clamps, adjustable for horizontal positioning of the specimen, and of a wire gauze. This shall enable the test specimen to be fixed with its long dimension horizontally, and with its transverse axis inclined at 45° to the horizontal line.
- e) A wire gauze (100 mm × 100 mm, 8 meshes per cm or 20 meshes per inch, 0,043 mm diameter steel wire) shall be clamped horizontally beneath the test specimen. An adjustable, movable holder maintains the burner tube in the same vertical plane as the lower longitudinal edge of the specimen and at an angle of approximately 45° to the horizontal line.
- f) A hand-operated timing device with a resolution of ±1 s or better.

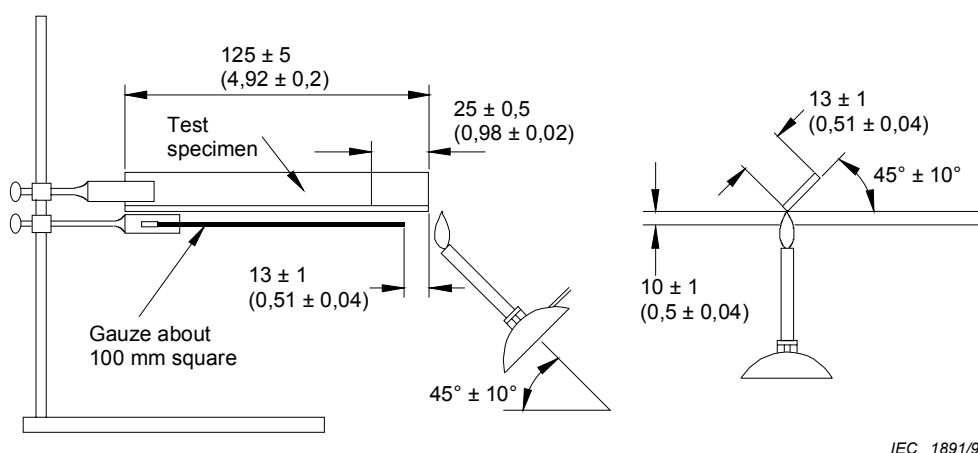
### 8.7.4 Test procedure

The specimens shall be preconditioned in accordance with the requirements of 5.3 of IEC 60068-1 for a period of 48 h as a referee or 24 h as normal quality conformance prior to testing. The detail requirements are

- a) a temperature of 15 °C to 35 °C;
- b) a humidity of 25 % RH to 75 % RH;
- c) an air pressure of 86 kPa to 106 kPa.

Fluctuations shall be kept to a minimum.

The test specimen shall be mounted in the test fixture such that the distance between the lowest edge of the specimen and gauze shall be 10 mm, with  $(13 \pm 2)$  mm of the unsupported end of the specimen projecting beyond the edge of the gauze as shown in Figure 4.



**Figure 4 – Test fixture**

The burner, in a remote position from the specimen, shall be adjusted by controlling the gas supply and air inlets of the burner until a yellow-tipped blue flame  $(20 \pm 2)$  mm in height is produced. The air supply is then increased until the yellow tip has disappeared. The height of the flame shall be measured again and corrected if necessary.

The burner shall be placed beneath the free end of the specimen so that a length of approximately 6,5 mm is subjected to the flame. The centre axis of the burner shall be in the same vertical plane as the lower horizontal edge of the specimen and at an angle of  $(45 \pm 10)^\circ$  to the horizontal line. Its position shall remain unchanged whilst the flame is applied.

The flame shall be applied to the specimen for 30 s and then removed. The burn time, in seconds, shall be measured from the instant of removal of the burner flame until the specimen extinguishes. Observation shall be made as to whether the burning proceeds beyond the indelible line.

The burn times and other observations shall be recorded on the laboratory pro forma as shown in Annex D.

### 8.7.5 Report

In addition to the general requirements for reporting, the report shall include:

- the test method;
- the average of the four burning times;
- the identification and description of the specimens;
- the thickness of the specimen;
- whether the burning of any of the specimens proceeds past the indelible line;
- whether the specimen material melts or produces burning drips;
- any deviation from this test method;
- the name of the person performing this test.

### 8.7.6 Additional information

Annex D comprises a suggested pro forma for reporting.

There are obvious hazards associated with flammability testing. Training of test operators, and familiarity with laboratory safety procedures is of paramount importance. All fire effluent should be considered to be toxic, for the purposes of safety, if not in fact.

Uncertainty of measurement for burn times, although a variable, prove to be impractical. The result of the test is an attribute; the HB rating, etc.

It is understood that a nominal substrate thickness of 1,6 mm will be used throughout the industry. Differences in thickness will prejudice test results.

American Industry requirements (Underwriter's Laboratory Specification ANSI/UL-94) detail a specimen width of 12,7 mm to 13,2 mm.

The specimen width of  $(13 \pm 0,3)$  mm has therefore been chosen since this will accommodate ANSI/UL-94.

The smoothness of the specimen edges can be critical to the performance of the specimen. A polished finish is recommended. A rough finish (for example, blanked) will significantly degrade performance due to the increase in surface area available to the flame.

Small-scale flammability tests, such as the one described, herein are an indicator of the behaviour of the material(s) tested. Fire integrity of equipment in which printed boards are used can only be assessed by equipment level testing.

## 8.8 Test 2C08: Flammability, flex material

### 8.8.1 Object

The purpose of this test method is to evaluate the flammability and to determine the degree of flame resistance of thin sheets or films in thickness less than 0,5 mm.

The test is carried out using a small test flame whose intensity is of similar order to that of a fire being started by the accidental overheating of a single electronic component.

### 8.8.2 Test specimens

#### 8.8.2.1 Choice of specimen shape

Either specimen shown in Figure 5, Flat rectangle for V method, vertical flammability, or Figure 6, wrapped cylindrical for VTM method, vertical flammability for un-reinforced laminate, will be chosen. The latter one is intended especially when the thickness of sample sheet is less than 0,2 mm and test results with former one are not stable due to being too thin or exhibiting excessive distortion or shrinkage of the specimens.

#### 8.8.2.2 Size and quantity of specimens

- a) Figure 5: The test specimen shall be 13 mm wide by 125 mm long by the thickness of the material. Five specimens shall be used for each set of measurements. Two sets shall be required for one test.
- b) Figure 6: The test specimen is wrapped tightly around the longitudinal axis of a 13 mm diameter mandrel to form a lapped cylinder 200 mm long with the 125 mm line exposed. The overlapping ends of the specimen are to be secured within 75 mm portion above the 125 mm mark (upper tube section) by means of pressure sensitive tape. The mandrel is then to be removed.

If the material is prone to developing static charges which make the formation of a cylinder difficult, the unformed specimen is to be discharged by a device or material intended for that purpose.

Different generic materials, although capable of being wrapped and taped around the mandrel, may exhibit varying degrees of flaring out of the untaped end, some of which may result in a nonlapped "U" type specimen. These various forms are considered acceptable to test if the upper end can be formed into the cylinder.

### 8.8.3 Test apparatus and materials

- a) Test chamber: A laboratory hood, totally enclosed, with a heat-resistant glass window for observing the test, shall be used. The exhaust fan shall be turned off during the test, but may be turned on to clear out the fumes between tests.
- b) Specimen holder: Clamping device shall be provided within the test chamber so that the specimen will hang with its length in a vertical position approximately coincident with the central vertical axis of the test chamber.
- c) Laboratory burner and gas supply: A Bunsen or Tirril burner shall be used having a tube length of 100 mm and an inside diameter of 9,5 mm. The gas supply shall be regulated and metered for uniform flow. The standard gas shall be technical grade methane. Natural gas having a normal heat content of 37 MJ/m<sup>3</sup> may be substituted.
- d) Timer: Stopwatch or other suitable timing device with a precision of 1,0 s minimum.
- e) Cotton: A supply of dry absorbent 100 % surgical cotton, cut by 50 mm by 50 mm with normal thickness 6 mm.
- f) Desiccator: Desiccation chamber, containing anhydrous calcium chloride, or other drying agent, capable of maintaining an atmosphere of less than 30 % RH at 23 °C.
- g) Conditioning room or chamber: Capable of being maintained at (23 ± 2)°C and RH of (50 ± 5) %.
- h) Air circulating oven type capable of maintaining (70 ± 2) °C.
- i) Mandrel for wrapping specimens formed from a rod of 12,5 mm diameter.
- j) Thin steel scale or template for gauging flame height.
- k) Etching system capable of complete removal of the metal cladding.
- l) Cutting apparatus such as shears or equivalent equipment.
- m) Tape: Pressure-sensitive adhesive.

### 8.8.4 Procedure

#### 8.8.4.1 Specimen preparation

Metal-clad laminates shall be completely etched using standard industry practices. Unclad laminate shall be tested in the "as is" condition. Specimens shall be cut and formed in accordance with 8.8.2.2.

#### 8.8.4.2 Conditioning

- a) Before testing, one set (five specimens) of specimens shall be kept for 48 h in the conditions in accordance with 8.8.3 g).
- b) The other set (five specimens) of specimens shall be kept for 168 h in the conditions in accordance with 8.8.3 h) and then allowed to stabilize in the desiccator noted in 8.8.3 f) for at least 4 h at room temperature, prior to testing.
- c) Once removed from the desiccator, specimens shall be tested within 30 min.
- d) All specimens are to be tested in a laboratory atmosphere of (15 to 35) °C and (45 to 75) % RH.



#### 8.8.4.3 Specimen mounting

- a) Clamp the specimen from the upper 6 mm of the specimen, with the longitudinal axis vertical, so that the lower end of the specimen is 300 mm above a horizontal layer of dry absorbent surgical cotton noted in 8.8.3 e).
- b) If the VTM method mentioned in 8.8.2.1 is applied, a heavy spring clamp should be used so that the upper end of the tube is closed to prevent any chimney effects during the test.

#### 8.8.4.4 Adjustment of test flame

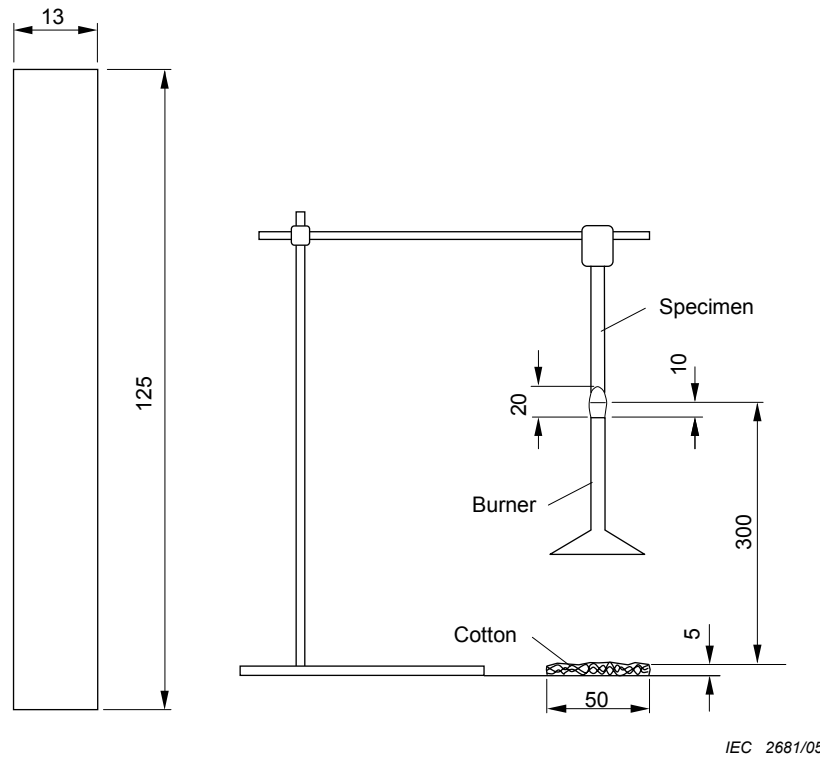
Adjust the burner to produce a blue flame 20 mm high. The flame is obtained by adjusting the gas supply and air ports of the burner until a 20 mm yellow-tipped blue flame is produced. Increase the air supply until the yellow tip just disappears. Measure the height of the flame again and readjust it if necessary.

#### 8.8.4.5 Test procedure for Figure 5

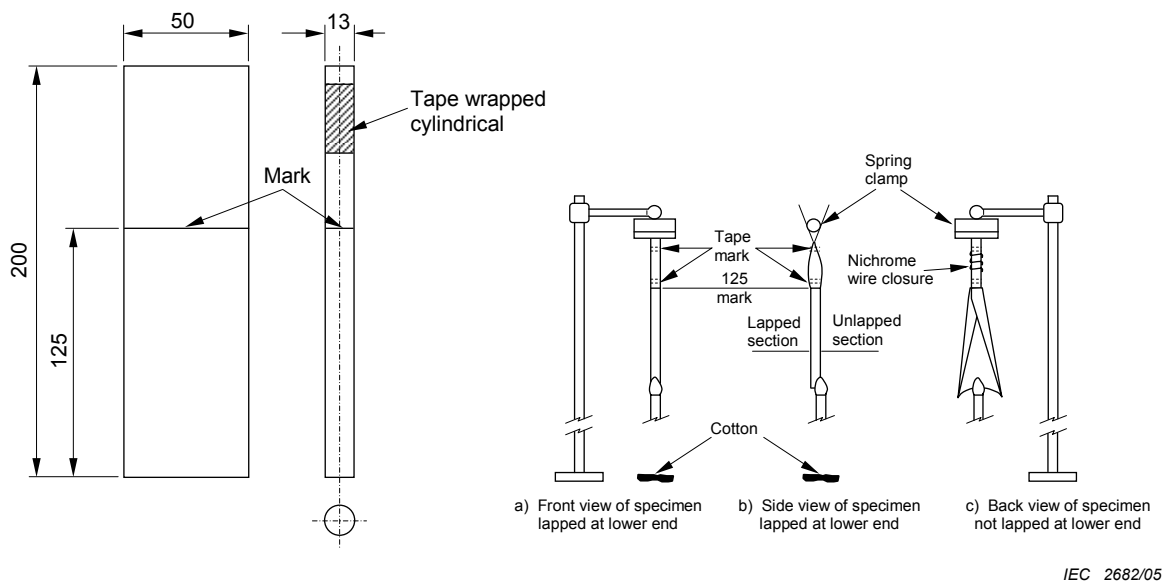
- a) Apply the flame centrally to the middle point of the bottom edge of the specimen so that the top of the burner is 10 mm below the lower end of the specimen and maintain it at that distance for 10 s, moving the burner as necessary in response to any changes in the length or position of the specimen. If the specimen drips molten or flaming material during the flame application, tilt the burner at an angle of up to 45° and withdraw it just sufficiently from beneath the specimen to prevent material from dropping into the barrel of the burner while maintaining the 10 mm spacing between the centre of the top of the burner and the remaining portion of the specimen, ignoring any strings of molten material. After the application of the flame to the specimen for 10 s, immediately withdraw the burner to a distance at least 150 mm away from the specimen and simultaneously commence measurement of the afterflame time,  $t_1$ , in seconds. Record  $t_1$ .
- b) After flaming of the specimen ceases, immediately place the burner again under the specimen and maintain the burner at a distance of 10 mm from the remaining portion of the specimen for additional 10 s, keeping the burner so that it is clear of dropping material. After this application of the flame to the specimen, remove the burner to a distance of at least 150 mm from the specimen and simultaneously commence measurement of the afterflame time,  $t_2$ , and the afterglow time,  $t_3$ . Record  $t_2$  and  $t_3$ .

#### 8.8.4.6 Test procedure for Figure 6

- a) Apply the flame centrally to the middle of the bottom edge of the specimen that is unlapped so that the top of the burner is 10 mm below that point of the lower end of the specimen, and maintain it at that distance for 3 s, moving the burner as necessary in response to any changes in the length or position of the specimen.
- b) For specimens that are not lapped at their lower end and that are suspended from the pinched upper end, the flame is to be applied in line with the longitudinal axis of the specimen.
- c) If the specimen drips molten or flaming material during the flame application, tilt the burner at an angle of up to 45° and withdraw it just sufficiently from beneath the specimen to prevent material from dropping into the barrel of the burner while maintaining the 10 mm spacing from the centre of the top of the burner and after the application of the flame to the specimen for 3 s, immediately withdraw the burner to a distance at least 150 mm away from the specimen and simultaneously use the timing device to commence measurement of the afterflame time  $t_1$  in seconds. Record  $t_1$ .
- d) When after-flaming of the specimen ceases, immediately place the burner under the specimen and maintain the burner at a distance of 10 mm from the remaining portion of the specimen for an additional 3 s, while moving the burner clear of dropping material as necessary. After the application of the flame to the specimen, remove the burner to a distance of at least 150 mm from the specimen and simultaneously commence measurement of the afterflame time,  $t_2$ , and the afterglow time,  $t_3$ , of the specimen. Record  $t_2$  and  $t_3$ .



**Figure 5 – V method (Vertical flammability method)**



**Figure 6 – VTM method (vertical flammability method for recalcitrant specimens)**

### 8.8.5 Report

The report shall include:

- number of the test method and revision;
- procedure used;
- identification of the specimens;
- afterflame time after first flame application,  $t_1$ .
- afterflame time after second flame application,  $t_2$ .

- f) afterglow time after second flame application,  $t_3$ .
- g) Figure 5: whether or not specimens burn up to the holding clamp;
- h) Figure 6: whether or not specimens burn up to the 125 mm mark;
- i) whether or not specimens drip flaming particles ignite the cotton indicator;
- j) date of the test;
- k) name of the person conducting the test

## 8.9 Test 2C09: Melting viscosity of prepreg materials

### 8.9.1 Object

This test method covers the procedure for the determination of the isothermic melting viscosity of prepreps at elevated temperatures using a rotating cone-and-plate viscosimeter.

### 8.9.2 Test specimens

The test specimens shall be cut not less than 25 mm from the edge or selvage of the prepreg.

The test specimens shall be prepared from a sample of the prepreg material under test by cutting rectangular pieces of approximately 200 mm × 300 mm, separating the resin from the reinforcement material by folding and crushing the prepreg and collecting it in a plastic bag. Any glass fibres present shall be removed by sieving with a wire mesh of 0,5 mm mesh width. (200 ± 20) mg resin powder shall be taken with the measuring scoop.

A single specimen should suffice.

### 8.9.3 Test apparatus and materials

The following apparatus and test material shall be used.

- a) A plate-and-cone rotating viscometer with a heatable plate and an adjustable revolution speed.

Cone material: Stainless steel type X12CrNiS18;

Cone diameter: (20 ± 0,5) mm;

Cone height: 2 mm;

Cone angle: 2°;

Cone surface: Roughness  $R = (6,3 \pm 0,1) \mu\text{m}$ ;

Distance between cone and plate:

- if spring-supported, the cone shall touch the surface of the plate; it can be lifted off with spring force (2 ± 1) mm;
- if fixed, the cone shall not touch the surface of the plate; the gap between cone and plate shall be constant during all measurements and is an item to be negotiated between customer and supplier.

- b) An X/Y chart recorder adjusted to the viscometer.

The zero of the chart recorder shall coincide with the zero of the viscosimeter. The adjustment shall be made with the rotating cone not resting on the plate.

The revolution speed of the cone shall be selected suitable to the plot width. If the total width is 250 mm, the plot width measured shall be between 50 mm and 100 mm.

For the defined cones 1 mm plot width corresponds to the following viscosity values at different revolution speeds:

Revolution speed r/min	Viscosity factor Pa × s
6	2,048
12	1,024
24	0,512
48	0,256

- c) A measuring scoop whose volume represents approximately 200 mg of resin powder.
- d) A wire mesh screen of 0,5 mm mesh width.
- e) A timer.
- f) A ruler with a millimetre scale (overall length 30 cm).
- g) Plastic bags large enough to hold prepreg sample.

#### 8.9.4 Procedure

The surface temperature of the viscosimeter plate shall be adjusted and preheated to  $(140 \pm 0,3) ^\circ\text{C}$ . The viscosimeter plate shall be preheated for a minimum of 30 min. The measuring cone, which is not heated, shall be lowered onto the preheated plate and shall be held in contact with it for  $2 \text{ min} \pm 10 \text{ s}$ . Then the cone shall be lifted, and the specimen of resin powder shall be placed on the heated plate concentric under the cone within 5 s.

As soon as the main amount of the specimen is placed on the plate and the revolution speed required is adjusted, the rotation of the viscosimeter and the timer shall be started. The recording head shall be lowered onto the recording paper and the paper transport shall be started with a speed of 20 mm/min.

$(30 \pm 3) \text{ s}$  after placing the resin powder on the heated plate, the rotating cone shall be lowered onto the molten resin.

The graph of viscosity versus time is recorded automatically. The measurement is completed, when a point of greatest inflection of the curve has been observed (see Figure 7) after the curve has been stabilized.

Then the cone is lifted from the plate, and the rotation is stopped.

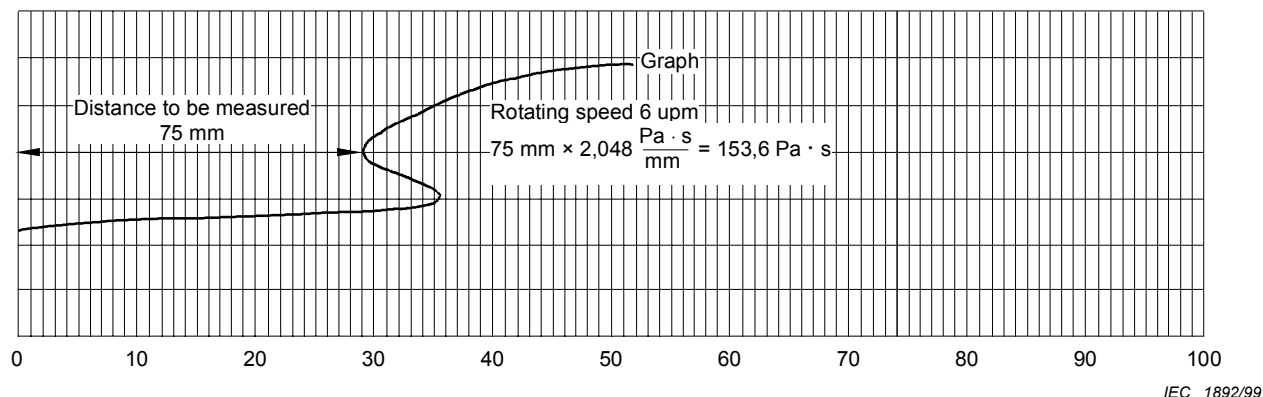
In order to evaluate the graph the chart recording paper shall be taken from the X/Y chart recorder, the distance between the time base line and the minimum of the curve measured with the ruler, and this value multiplied with the viscosity factor for the selected revolution speed (see 8.9.3). The resulting product is the melting viscosity in Pascal seconds.

#### 8.9.5 Report

The report shall include:

- a) the test number and revision;
- b) the identification of the sample prepreg tested;
- c) the date of the test;
- d) identification and description of the specimens of resin powder;
- e) the distance between cone and plate, if fixed;

- f) the revolution rate of the cone;
- g) the melting viscosity;
- h) any deviations from the parameters defined (for example plate temperature or cone type).



**Figure 7 – Example of prepreg melting viscosity**

#### 8.9.6 Additional information

As several cones may exhibit small differences of their geometry, it is necessary to calibrate each cone to be used with the viscosimeter. This can be carried out:

- either by the manufacturer, who gives the corresponding calibration factors;
- or by the user with a qualified calibration oil.

Cleaning of the cone and the heating plate. The following steps are recommended:

- rough cleaning with a bronze scraper;
- afterwards fine cleaning with an appropriate solvent.

### 8.10 Test 2C10: Resin content of prepreg materials by sublimation

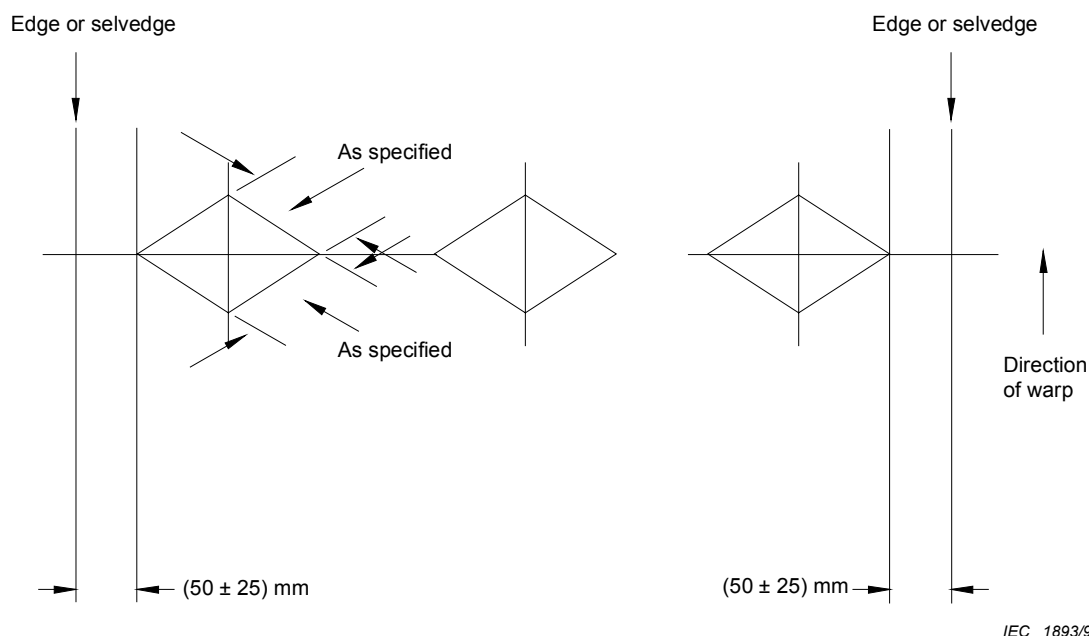
#### 8.10.1 Object

The purpose of this test method is to provide a means for measuring the resin content of resin impregnated B-stage glass fabric for use as bonding sheet material for base materials and printed boards using the resin sublimation method.

#### 8.10.2 Test specimens

Each specimen shall be comprised of three squares  $(100 \pm 10)$  mm by  $(100 \pm 10)$  mm as taken from positions along a line normal to the warp of the fabric with the diagonals of the squares parallel to the warp and weft threads. One square shall be taken from the position equidistant from the edges, and the other two squares from positions on opposite sides of the first with their outer extremities  $(50 \pm 25)$  mm from each edge or selvage (see Figure 8).

All loose particles and projecting fibres shall be removed from the squares.



**Figure 8 – Position of specimens for resin content**

### 8.10.3 Test apparatus and materials

The following test apparatus and material shall be used.

- An analytical balance with an accuracy of 0,001 g.
- A muffle furnace capable of maintaining a temperature between 550 °C and 800 °C. The muffle furnace must be placed in a ventilation hood.
- A ceramic crucible of sufficient size to hold the specimen.
- A stabilization chamber (drying cabinet desiccator) capable of maintaining less than 20 % RH at room temperature.

### 8.10.4 Procedure

For a referee, the crucible shall be heated in a muffle furnace between 550 °C and 800 °C for 15 min, and allowed to cool to room temperature in a desiccator and weighed to the nearest 0,001 g ( $M_1$ ). For normal conformance testing, the crucible may simply be weighed to the nearest 0,001 g.

The test specimen shall be placed in the crucible. It is permissible to cut the 100 mm × 100 mm square into pieces to allow it to fit into the crucible.

The specimen and crucible shall then be weighed to the nearest 0,001 g ( $M_2$ ).

The specimen and crucible shall be heated in the muffle furnace at a temperature of between 550 °C and 800 °C for 1 h and placed in the desiccator and allowed to cool to room temperature.

The specimen and crucible shall again be weighed to the nearest 0,001 g ( $M_3$ ).

The heating, cooling, and weighing shall be repeated until two consecutive weighings ( $M_3$ ) agree within 0,002 g.

The per cent resin content shall be calculated as follows:

$$C_r = \frac{100 (M_2 - M_3)}{M_2 - M_1} - C_v$$

where

- $C_r$  is the percentage resin content (%);
- $M_1$  is the weight of crucible (g);
- $M_2$  is the initial weight of crucible and specimen (g);
- $M_3$  is the final weight of crucible and specimen (g);
- $C_v$  is the percentage volatile content (%).

#### 8.10.5 Report

The report shall include:

- a) the test number and revision;
- b) the date of the test;
- c) the identification of the material tested;
- d) the resin content for each of the three specimens;
- e) any deviation from this test method;
- f) the name of the person conducting the test.

#### 8.10.6 Additional information

The resin content of prepreg may also be determined by the treated weight method as described in Test 2C03.

The fumes from burning resin are toxic, so the muffle furnace shall be placed in a ventilation hood.

### 8.11 Test 2C11: UV blocking characteristics of laminates

#### 8.11.1 Object

The purpose of this test method is to establish a technique of measuring the ultraviolet blocking ability of base materials. Laminates which more effectively block the transmission of UV light are less prone to a "print through" defect for UV cured solder mask.

#### 8.11.2 Test specimens

- a) Three specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge of the sheet or panel.
- b) The specimens shall be approximately 100 mm × 100 mm in both the length and width as taken from the sample of metal-clad base material under test.
- c) The metal cladding shall be completely removed by etching or other suitable means which does not affect the surface of the laminate. The surface of the laminate shall be cleaned thoroughly.

### 8.11.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) an exposure machine consisting of a colourless transparent glass platform or a transparent film which constitutes a support platform and a 3 kW ultraviolet lamp whose peak wavelength is 365 nm;
- b) an ultraviolet irradiance meter capable of measuring a maximum intensity of 40 mW/cm<sup>2</sup>. The peak wavelength shall be measurable with a sensitivity of 0,1 mW/cm<sup>2</sup>.

### 8.11.4 Procedure

- a) Switch on the power of the exposure machine and preheat the lamp for at least 10 min but not more than 30 min.
- b) Shift the UV irradiance meter to the maximum measuring range. Put the UV probe on the exposure machine transparent platform with the probe centred over the UV lamp.
- c) Adjust the distance between the UV lamp and the glass platform so that the UV intensity shown by the UV irradiance meter is within  $(25 \pm 5)$  mW/cm<sup>2</sup>. This value is recorded as "A".
- d) Remove the UV probe and put the specimen on the glass platform. The probe shall then be centred over the test specimen. Record the UV intensity shown by the UV irradiance meter as "B".
- e) The other two specimens shall be measured and recorded in the same fashion.

### 8.11.5 Report

In addition to the general requirements for reporting, the report shall include:

- a) the test method number and revision;
- b) the date of the test;
- c) the identification and description of the specimen;
- d) the average and maximum of the three recorded UV irradiance values;
- e) any deviation from this test method;
- f) the name of the person conducting the test.

## 8.12 Test 2C12: Total halogen content in base materials

### 8.12.1 Object

The purpose of this test method is to establish the amount of chlorine and bromine compounds in base materials. This test method is applicable to reinforced base materials with a minimum thickness of 0,3 mm and to un-reinforced base materials with a minimum thickness of 0,08 mm.

A combustion flask is used to extract ionic and covalent halogen from the specimen, and ion exchange chromatography is used for the quantitative analysis of halogen content.

### 8.12.2 Test specimens

- a) Rigid or flexible base materials shall be used for the test in accordance with the minimum thickness requirements described in the object. The copper foil (if applicable) shall be removed from the test specimens by etching, by any industry acceptable etching method or by mechanical peeling before test.
- b) For reinforced base materials, the number of test specimens shall not be less than 5 with a minimum size of 1 cm × 1 cm. For un-reinforced base materials, the number of test specimens shall not be less than 5 with a minimum size of 1 cm x 1 cm.



- c) Wash the specimens thoroughly in distilled or de-ionized water. Reference shall be made for the size and number of the specimens.
- d) For reinforced base materials, dry the specimen at  $(105^{+5}_0)$  °C for  $(1^{+0,25}_0)$  h.
- e) For un-reinforced base materials, wipe off the water with a lint-free cloth or paper wiper and leave them to dry at room temperature for a minimum of 1 h.

### 8.12.3 Apparatus and materials

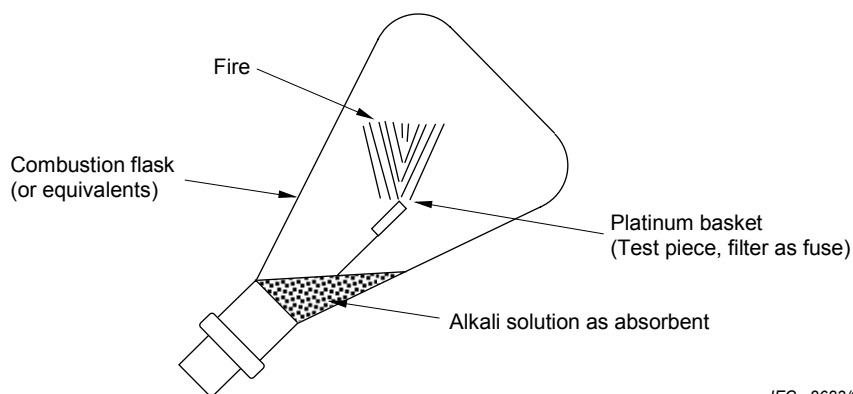
The following test apparatus and materials shall be used:

- a) ion exchange chromatograph with a detection limit of  $10^{-6}$  or better;
- b) analytical balance with an accuracy of approximately 1 mg or better;
- c) knife;
- d) tweezers;
- e) vinyl gloves;
- f) lint-free cloth, paper wipers or equivalent;
- g) quantitative filter paper;
- h) combustion flask, or equivalent;
- i) oxygen (99,9 % purity or better);
- j) gas pressure regulator;
- k) flint striker, or another ignition device;
- l) wash bottle;
- m) beaker;
- n) micro-pipette;
- o) flasks (various sizes);
- p) potentiometric titrator for silver nitrate, if necessary;
- q) platinum basket for holding specimen;
- r) alkali solution;
- s) ethyl alcohol;
- t) chlorine ion standard solution for chromatograph;
- u) bromine ion standard solution for chromatograph;
- v) silver nitrate titrant if potentiometric titration using silver nitrate is used.

### 8.12.4 Procedure

#### 8.12.4.1 Combustion procedure

- a) Weigh the specimen using the analytical balance and record the result.
- b) Place approximately 50 ml alkali solution in a combustion flask to act as an absorbent of combustion gas.
- c) Fill the combustion flask with oxygen.
- d) Insert a test specimen into the combustion flask as shown in Figure 9 below.
- e) Insert a piece of filter paper into the combustion flask as shown in Figure 9 to act as a flame starter.
- f) Apply several drops of ethyl alcohol on the test specimen for improving ignition of the flame. Ignite fire to the filter paper.



IEC 2683/05

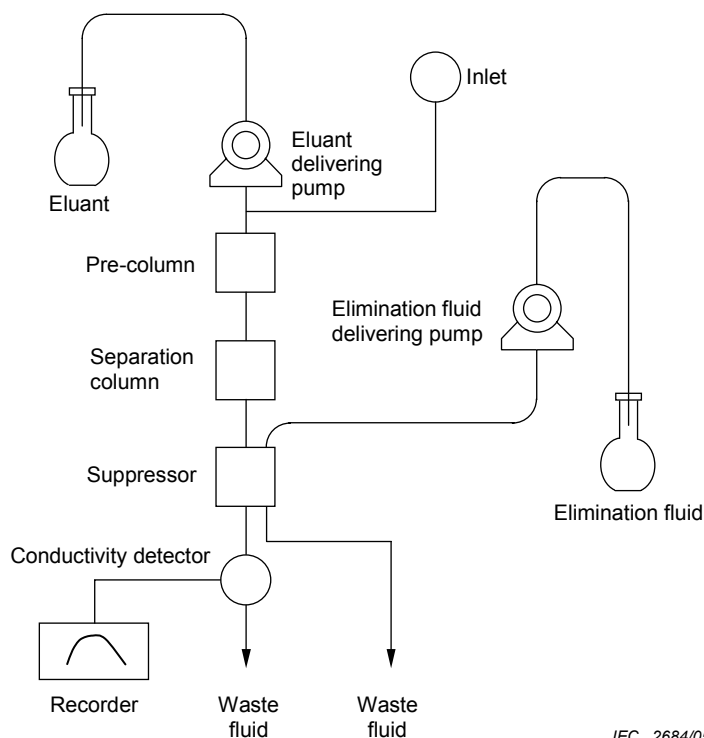
**Figure 9 – Absorption of combustion gas using a combustion flask set-up**

- g) Leave the flask at room temperature for  $(30 \pm 2)$  min after combustion. The generated gas is absorbed into the alkali solution to produce  $\text{Cl}^-/\text{Br}^-$  ion solution.
- h) Transfer the solution from the combustion flask to a volumetric flask. Add de-ionized water in the flask until the total amount of the test solution becomes 100 ml.
- i) Remove the suspended particles from the test solution by filtration or centrifugation if necessary.
- j) Follow the same procedure outlined above without the test specimen to prepare a reference test solution (blank) without combustion of a test specimen.

NOTE Chlorine and bromine contents shall be measured first without any test specimen in the combustion flask.

#### 8.12.4.2 Chemical analysis

- a) Inject the test fluid gathered from the flask into the inlet of an ion exchange chromatograph shown in Figure 10 below.



IEC 2684/05

**Figure 10 – Composition of ion exchange chromatograph**

- b) Analyse the peak area/height of the  $\text{Cl}^-/\text{Br}^-$  ions conductivity on the recorder and obtain  $\text{Cl}^-/\text{Br}^-$  ions concentration from a calibration curve.
- c) Obtain  $\text{Cl}^-/\text{Br}^-$  ions concentration of the reference by comparing its conductivity using the standard solution. The test solution is compared to the reference solution (blank). Any contaminant in the reference solution will be subtracted from the test solution.
- d) Titrate test solution using silver nitrate if concentration of  $\text{Cl}^-/\text{Br}^-$  ions is high (higher than 1 wt %).
- e) An example of analysing conditions for the ion exchange chromatography is given in Table 2 below.
- f) Analysing conditions depends on the test devices, test specimens, their composition and environment.

**Table 2 – Example of analysing conditions for the ion exchange chromatography**

Item/material	Definition/quantity
Eluant	Alkali solution
Eluant delivering rate	1,0 ml/min to 2,0 ml/min
Elimination fluid	$\text{H}_2\text{SO}_4$
Delivering rate of elimination fluid	1,0 ml/min to 2,0 ml/min
Column	Pre-column, separation column
Suppressor	Suppression for anion
Detector	Conductivity meter

**8.12.4.2.1 Calculation of halogen content**

Insert the concentration of halogen ion ( $\text{Cl}^-/\text{Br}^-$ ) obtained in the following formula to obtain halogen contents in the specimens.

$$\text{Chlorine (wt \%)} = (\text{Cl}^- \text{ concentration in the test fluid (}^{-6} \text{ ppm)} - \text{Cl}^- \text{ concentration in the reference (}^{-6} \text{ ppm)}) \times \{\text{quantity of test solution (ml)/mass of the test specimen}\} \times 10^{-7}$$

NOTE 1 For the bromine content, use the same equation but  $\text{Br}^-$  values in the place of  $\text{Cl}^-$ .

**8.12.5 Report**

In addition to the general requirements for reporting, the report shall include:

- a) the test method number and revision;
- b) the date of the test;
- c) the identification and description of the specimen;
- d) the average chloride content of the 5 specimens in ppm;
- e) the average bromide content of the 5 specimens in ppm;
- f) the average total halogen content of the 5 specimens in ppm;
- g) any deviation from this test method, and
- h) the name of the person conducting the test.

## 9 M: Mechanical test methods

### 9.1 Test 2M01: Test method for bow and twist

#### 9.1.1 Object

This test method covers the procedure for determination of the deviation from flatness of the metal-clad sheet in a direction parallel to its edges or diagonal.

This test is not applicable to sheets thinner than 0,8 mm or with copper thickness differences side to side of more than 70  $\mu\text{m}$  (610  $\text{g/m}^2$ ).

#### 9.1.2 Test specimens

Test specimens shall be taken from the panel or sheet in such a manner that they are at least 25 mm from the edge.

Specimens shall be prepared from a sample of the metal-clad base material under test.

Specimen size shall be  $(300 \pm 5)$  mm in both length and width. Other specific panel sizes may be tested upon agreement between customer and supplier.

For referee, the specimen will be conditioned at  $(23 \pm 2) ^\circ\text{C}$  and  $(50 \pm 5) \% \text{ RH}$  for a minimum of 18 h prior to measurement of bow or twist.

A minimum of three specimens shall be tested.

#### 9.1.3 Test apparatus and materials

A taper gauge or feeler gauge shall be used.

#### 9.1.4 Procedure

##### a) Bow measurement

- Bow shall be measured by placing the specimen unrestrained on a flat horizontal surface with its predominantly convex surface upward. The maximum vertical distance from the flat surface to the bottom side of the laminate shall be determined using a taper gauge or a feeler gauge.
- The result shall be expressed as a percentage of the length of the side where the vertical displacement was measured.

##### b) Twist measurement

- Twist shall be measured by placing the specimen unrestrained on a flat horizontal surface with its predominantly convex surface upward and with three corners in contact with the flat surface. The maximum vertical distance from the flat surface to the bottom side of the remaining corner of the laminate shall be determined using a taper gauge or a feeler gauge.
- The result shall be expressed as a percentage of the diagonal length of the specimen.
- The bow and twist shall be reported as the average of the three specimens tested and the highest measurement for both bow and twist.

#### 9.1.5 Report

The report shall include:

- a) the test number and revision;
- b) the testing date;

- c) the identification of the material tested;
- d) the average and highest measurement of bow;
- e) the average and highest measurement of twist;
- f) any deviation from the test method, including panel size if not 300 mm × 300 mm;
- g) the name of the person conducting the test.

#### 9.1.6 Additional information

None.

### 9.2 Test 2M02: Bow/twist after etching and heating

#### 9.2.1 Object

This test method covers the procedure for the determination of the deviation from flatness of the metal-clad sheet in a direction parallel to its edges, or diagonal after etching and heating, simulating two process steps of printed board fabrication.

This test is not applicable to sheets thinner than 0,8 mm or with copper thickness differences side to side of more than 70 µm (610 g/m<sup>2</sup>).

#### 9.2.2 Test specimens

Specimens shall be taken from the panel or sheet in such a manner that they are at least 25 mm from the edge.

Specimens shall be prepared from a sample of the metal-clad base material under test.

Specimen size shall be (300 ± 5) mm in both length and width. Other specific panel sizes may be tested upon agreement between user and supplier.

The specimens shall be maintained at standard laboratory conditions of (23 ± 2) °C and (50 ± 2) % RH during the test. As a referee the specimen will be conditioned for a minimum of 18 h prior to measurement of bow or twist.

A minimum of three specimens shall be tested.

#### 9.2.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) Any etching method of commercial practice shall be used. In case of dispute between customer and supplier, the etching shall be carried out with a spray, or equivalent method with an aqueous solution of ferric chloride of density 1,32 g/cm<sup>3</sup> to 1,41 g/cm<sup>3</sup>, as measured at room temperature. The temperature of the etching solution shall not exceed 37 °C.
- b) A suitable chamber (air-circulating oven) capable of maintaining the temperature as called for in the relevant material specification with a tolerance of ±2 °C.
- c) A taper gauge or feeler gauge.

## 9.2.4 Procedure

### 9.2.4.1 Etching

The metal of the specimen shall be completely removed by etching. Immediately after it is etched, the specimens shall be washed with cold running water of resistivity of not less than 10  $\Omega$  for as long as necessary to remove surface contamination (normally 15 min to 30 min).

### 9.2.4.2 Heating

The etched specimen shall be placed unrestrained on a flat horizontal surface and shall be subjected to dry heat as specified in 3.1 of IEC 60068-2-2:1974 in a test chamber with the temperature indicated in the relevant material specification, a temperature tolerance of  $\pm 2$  °C and for a duration of  $(30 \pm 5)$  min.

### 9.2.4.3 Conditioning

After the process steps of etching and heating, for referee purposes the specimens shall be preconditioned at  $(23 \pm 2)$  °C and  $(50 \pm 5)$  % RH for 18 h as the conditioning step. As a quality conformance procedure, at least 2 h of conditioning shall be used.

### 9.2.4.4 Bow measurement

Bow shall be measured by placing the specimen unrestrained on a flat horizontal surface with its predominately convex surface upward. The maximum vertical distance from the flat surface to the bottom side of the laminate shall be determined using a taper gauge or feeler gauge.

The result shall be expressed as a percentage of the length of the side where the vertical distance was measured.

The bow value after etching and heating reported shall be the average of the percentages of the three test specimens as well as the highest measurement.

### 9.2.4.5 Twist measurement

Twist shall be measured by placing the specimen unrestrained on a flat horizontal surface with its predominantly convex surface upward and with three corners on the bottom side of the specimens in contact with the flat surface. The maximum vertical distance from the flat surface to the bottom side of the remaining corner of the laminate shall be determined using a taper gauge or feeler gauge.

The result shall be expressed as a percentage of the diagonal of the specimen.

The twist value after etching and heating reported shall be the average of the percentages of the three test specimens as well as the highest measurement.

## 9.2.5 Report

The report shall include:

- a) the test number and revision;
- b) the date of the test;
- c) the identification of the material tested;
- d) the average and highest measurement of bow;
- e) the average and highest measurement of twist;
- f) any deviation from this test method;
- g) the name of the person conducting the test.

### 9.2.6 Additional information

The etching solution described in 9.2.3 of this test method is a powerful acid chemical. It shall be handled with care preventing eye and skin contact by wearing protective glasses and chemically resistant gloves respectively.

## 9.3 Test 2M03: Cure factor of base materials by differential scanning calorimetry (DSC) or thermomechanical analysis (TMA)

### 9.3.1 Object

This test method covers the procedure for the determination of the cure factor of the base material resin by differential scanning calorimetry or thermomechanical analysis.

The cure factor is calculated as the cured portion of the resin.

### 9.3.2 Test specimens

- a) The test specimens shall be prepared as described in 9.10.2 of test method 2M10 or 9.11.2 of 2M11.
- b) A minimum of three specimens shall be used.

### 9.3.3 Test apparatus and materials

The test apparatus and materials shall be that as described in 9.10.3 of test method 2M10 or 9.11.3 of 2M11.

### 9.3.4 Procedure

#### 9.3.4.1 Process step 1

The procedure for measuring the glass transition temperature of the material shall be as follows. When the heating is completed the test specimen and reference shall be maintained at the final temperature for  $(10 \pm 5)$  min. After this time the test specimen and reference material shall be immediately cooled to  $30^\circ\text{C}$  and be allowed to stabilize for  $(10 \pm 5)$  min without removing them from the chamber.

The glass transition temperature shall be determined as described in 9.10.4 of test method 2M10 or 9.11.4 of 2M11.

#### 9.3.4.2 Process step 2

The test specimen and reference shall be re-subjected to process step 1.

The glass transition temperature shall be measured again.

#### 9.3.4.3 Evaluation

The cure factor shall be determined from the following formula:

$$C_F = 1 - \frac{T_{g,2} - T_{g,1}}{T_{g,1}}$$

where

$C_F$  is the cure factor,

$T_{g,1}$  is the glass transition temperature [K] determined by process step 1,

$T_{g,2}$  is the glass transition temperature [K] determined by process step 2.

The cure factor determined shall be the average value measured from three determinations.

### 9.3.5 Report

The report shall include:

- a) the test number and revision;
- b) the date of test;
- c) the identification of the material tested;
- d) the quantity, form and weight of the specimen;
- e) the reference (whether empty crucible etc.) and weight;
- f) the method of preparation of the specimen and any heat treatment;
- g) the heating rate if other than 10 °C/min;
- h) the cure factor as an average of the three specimens;
- i) the name of the person conducting the test.

### 9.3.6 Additional information

- a) Consideration must be given to the effects of water absorption of the test specimen when determining cure factor by means measuring glass transition temperature.
- b) Prolonged exposure to elevated temperature can thermally degrade the resin structure and lead to incorrect cure factors of greater than 1,0.

## 9.4 Test 2M04: Twist after heating (under consideration)

## 9.5 Test 2M05: Pull-off strength

### 9.5.1 Object

To determine the force, normal to the specimen, required to separate a land from the base material after a specified hand soldering operation. The test is performed on the specimen as manufactured and without conditioning. The test is not applicable to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier

### 9.5.2 Test specimens

Specimens shall be removed from the panel in such a way that they are at least 25 mm from the edge of the laminate.

Specimens may be of any convenient size and shall be printed by any appropriate method in usual practice with a test pattern consisting of a number of lands of diameter  $(4 \pm 0,1)$  mm. It is convenient for each land to have a central etched hole of a diameter of about 0,6 mm for locating the drill.

A hole of diameter  $(1,3 \pm 0,1)$  mm shall be drilled through the centre of each land.

A minimum of at least 10 lands shall be tested. For a double-sided metal-clad laminate, at least five lands on each side shall be tested.

Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique for example by sticking the sample to a rigid material.



### 9.5.3 Test apparatus and materials

The following test apparatus and test material shall be used:

- a) a temperature controlled soldering iron with a copper bit about 30 mm long and 5 mm in diameter, with its end forming an angle of  $45^\circ \pm 10^\circ$ . The temperature of the bit shall be  $(270 \pm 10)^\circ\text{C}$  throughout the test;
- b) the solder to be used shall be a 60/40, or 63/37 tin-lead wire with a diameter not greater than 1,5 mm and a rosin core flux core;
- c) single-strand copper wire of diameter 0,9 to 1,0 mm;
- d) a jig to prevent the wire moving during soldering and subsequent cooling;
- e) a suitable support in the form of a rigid plate with a central hole of 6 mm diameter;
- f) a tensile testing machine.

### 9.5.4 Procedure

The land shall be evenly tinned for  $(4 \pm 1)$  s with the soldering tool and a minimum amount of solder.

A piece of single strand copper wire, previously tinned with the solder, shall be passed at a right angle through the hole in the centre of the land and soldered into position for  $(4 \pm 1)$  s so that a continuous fillet is formed between the wire and the land. For soldering the heat supply shall be made to the wire only. The wire shall not be moved during the soldering and subsequent cooling, and to ensure that it is not, the wire and specimen may be held in a jig.

The specimens shall be allowed to cool to room temperature for at least 10 min before testing.

By means of a tensile testing machine and support described in 9.5.3 e), a force shall be applied in tension, by pulling the wire at right angles to the test specimen at a crosshead speed of  $(50 \pm 2)$  mm/min until the land separates from the base material or the wire breaks. The force in N required to detach the land, or to break the wire, shall be noted.

A test shall be invalidated by the breaking of a wire before the specified minimum force is reached, or by wire pull-out, and in no case shall the same wire and the same land be resoldered and pulled again. The test shall be repeated until 10 valid measurements have been made.

The minimum force required to detach any of the 10 lands from the base material shall be reported as the pull-off strength of the sample under test. Each side of the laminate shall be reported separately.

### 9.5.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the measured pull-off strength for each side of the laminate;
- e) any deviation from this test method.

### 9.5.6 Additional information

None.

## 9.6 Test 2M06: Peel strength after exposure to solvent vapour

### 9.6.1 Object

To determine the resistance to peeling under specified conditions and after exposure to solvent vapour. The test is performed on the sample as manufactured and without conditioning. The test does not apply to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier.

### 9.6.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be prepared from a sample of the copper-clad base material under test and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup>, the foil mass per unit area may be increased to not more than 335 g/m<sup>2</sup> by any procedure in which ductile copper is deposited. The specimen shall be (50 ± 1) mm wide, and at least 75 mm long, and it shall be printed by any appropriate method in usual practice with the test pattern of Figure 11.
- c) The number of specimens used for one test shall be sufficient for the peeling of at least 25 mm from each of four strips of copper foil.
- d) Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique for example by sticking the sample to a rigid material.

### 9.6.3 Test apparatus and materials

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of (50 ± 5) mm/min;
- b) a clamp to grip the detached end of the foil over its entire width;
- c) a solvent to be agreed between purchaser and supplier.

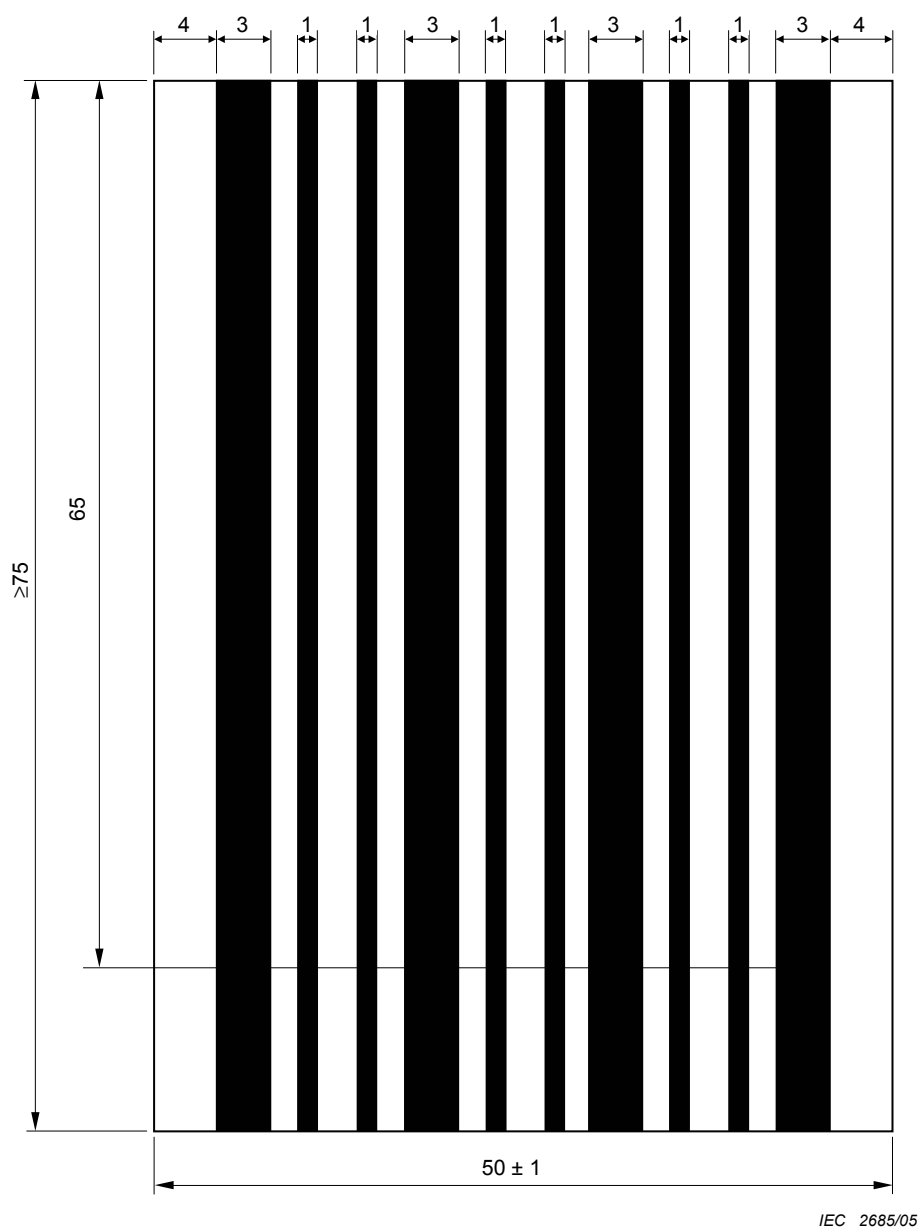
### 9.6.4 Procedure

The specimen shall be suspended for (120 ± 5) s in the vapour of the solvent chosen, boiling at atmospheric pressure. The samples shall be removed from the solvent vapour and placed in standard laboratory conditions of (23 ± 2) °C and (45 ± 5) % RH. Immediately after removal from the solvent and again after 24 h, the specimen shall be examined for blistering or delamination. If it has not blistered or delaminated, it shall then be used for the measurement of peel strength after exposure to solvent vapour. By agreement between purchaser and supplier the test may be carried out with other solvent vapours.

One end of the strip of the metal foil shall be detached from the base material for a distance of at least 10 mm, and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width in the jaws of the clamp on the tensile testing machine, and a steady increasing pull shall be applied in a direction (90 ± 5)° to the plane of the base material until the metal foil is peeled off at a rate of (50 ± 5) mm/min. The peel force shall be recorded. A length of at least 25 mm shall be peeled at this rate from each of four strips.

Should copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup> break during the test, or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described in the method to a total mass per unit area not less than 275 g/m<sup>2</sup> but not more than 335 g/m<sup>2</sup>.

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in newtons per millimetre.



NOTE 1 For at least 65 mm at one end of each strip, the strip should be  $(1 \pm 0,1)$  mm wide or  $(3 \pm 0,2)$  mm wide. Line spaces between adjacent lines may be approximately 2 mm to 3 mm.

NOTE 2 For reference purposes,  $(3 \pm 0,2)$  mm line width is recommended.

**Figure 11 – Specimen for peel strength measurement**

### 9.6.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the solvent vapour to which the specimen was exposed;
- e) the measured peel strength;
- f) whether additional copper has been added;

- g) whether there was blistering or delamination in the specimen immediately after exposure to solvent vapour and/or after 24 h recovery;
- h) any deviation from this test method.

#### 9.6.6 Additional information

Environmentally damaging solvents shall not be used. But if it is required to use such solvents, the vapour should not be released into the environment. A good way to prevent this is to use a cooler above the vapour in such a way that, after condensation, the solvent drips back in the heated vessel. The environmental protection decrees of the national authorities shall be observed.

### 9.7 Test 2M07: Peel strength after immersion in solvent

#### 9.7.1 Object

To determine the resistance to peeling under specified conditions and after immersion in solvent. The test is performed on the sample as manufactured and without conditioning. The test does not apply to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier.

#### 9.7.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be prepared from a sample of the copper-clad base material under test and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup>, the foil mass per unit area may be increased to not more than 335 g/m<sup>2</sup> by any procedure in which ductile copper is deposited. The specimen shall be (50 ± 1) mm wide, and at least 75 mm long, and it shall be printed by any appropriate method of usual practice with the test pattern of Figure 11.
- c) The number of specimens used for one test shall be sufficient for the peeling of at least 25 mm from each of four strips of copper foil.
- d) Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique, for example by sticking the sample to a rigid material.

#### 9.7.3 Test apparatus and materials

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of (50 ± 5) mm/min;
- b) a clamp to grip the detached end of the foil over its entire width;
- c) the solvent as stated in the relevant specification.

#### 9.7.4 Procedure

Use for this test the solvent as stated in the relevant specification. Additional solvents may be used by agreement between purchaser and supplier. The test shall be carried out on separate test specimens for each solvent.

The specimen shall be immersed for (10 <sup>+1</sup><sub>0</sub>) min in the solvent at room temperature.

The samples shall be removed from the solvent and placed in standard laboratory conditions of (23 ± 2) °C and (45 ± 5) % RH. Immediately after removal from the solvent vapour and again after 24 h, the specimen shall be examined for blistering, delamination, surface roughness, tackiness or colour change. If it has not blistered, delaminated or experienced other surface defects, it shall be used for the measurement of peel strength after immersion in solvent.

One end of the strip of the metal foil shall be detached from the base material for a distance of at least 10 mm, and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width in the jaws of the clamp on the tensile testing machine, and a steady increasing pull shall be applied in a direction  $(90 \pm 5)^\circ$  to the plane of the base material until the metal foil is peeled off at a rate of  $(50 \pm 5)$  mm/min, the peel force shall be recorded. A length of at least 25 mm shall be peeled at this rate from each of four strips.

Should copper foil of nominal mass less than  $305 \text{ g/m}^2$  break during the test or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described above to a total mass not less than  $275 \text{ g/m}^2$ , but not more than  $335 \text{ g/m}^2$ .

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in newtons per millimetre.

### 9.7.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the solvent used;
- e) the measured peel strength;
- f) whether additional copper has been added to the test specimen;
- g) whether there was blistering, delamination, surface roughness, tackiness or colour change immediately after the immersion and/or after 24 h recovery;
- h) any deviation from this test method.

### 9.7.6 Additional information

Environmentally damaging solvents shall not be used. But if it is required to use such solvents the vapour should not be released into the environment. A good way to prevent this is to use a cooler above the vapour in such a way that, after condensation, the solvent drips back in the heated vessel. The environmental protection decrees of the national authorities shall be observed.

## 9.8 Test 2M08: Flexural strength (under consideration)

## 9.9 Test 2M09: Resin flow of prepreg material

### 9.9.1 Object

The purpose of this test method is to provide a means for measuring the resin flow of impregnated reinforcement cured to the B-stage.

### 9.9.2 Test specimens

- a) The test specimens shall be taken in such a way that they are at least 25 mm from the edge of the prepreg sheet or roll.
- b) For rolls of prepreg, four specimens  $(100 \pm 0,2) \text{ mm} \times (100 \pm 0,2) \text{ mm}$  shall be cut from positions equally spaced across the width of the roll. For sheeted prepreg, the four specimens shall be cut randomly from two different sheets. The diagonals of the specimens shall be parallel to the machine and cross-machine directions, see Figure 2.
- c) All loose particles and projecting fibres shall be removed from the material.

### 9.9.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) laminating press maintained at a temperature of  $(170 \pm 3) ^\circ\text{C}$  and capable of providing a pressure of  $(1,5 \pm 0,15)$  MPa on the test specimen;
- b) analytical balance with a 0,001 g or better resolution;
- c) release material polyvinyl-fluoride (PVF) or equivalent;
- d) plates of stainless steel  $(1,6 \pm 0,8)$  mm thick and 150 mm square minimum, but not larger than the press platen size;
- e) a punch and die set, circular disk of  $(79,8 \pm 0,1)$  mm in diameter or square, whose sides are  $(70,7 \pm 0,4)$  mm;
- f) desiccator (stabilization chamber) capable of maintaining less than 25 % RH at room temperature.

### 9.9.4 Procedure

If specimens are stored for over 24 h, specimens should be desiccated for a minimum of 4 h prior to testing.

The specimens shall be stacked together with their edges in careful alignment. They may be fastened together with suitable means. The stack of specimens shall then be weighed to the nearest 0,005 g ( $M_1$ ).

Place the stack of specimens between the release films, then place them all between plates of stainless steel.

Insert the assembly of plates and test specimen in the preheated laminating press maintained at  $(170 \pm 3) ^\circ\text{C}$  and immediately apply a pressure of  $(1,5 \pm 0,15)$  MPa within a maximum of 5 s elapsed time. The pressure shall be held until there is no further flow of resin, or for 10 min, whichever is longer.

After lamination, open the press, remove the specimen and allow to cool to room temperature.

A piece having an area of 5 000 mm<sup>2</sup> shall be removed from the central area of the laminate in the form of a square whose sides are  $(70,7 \pm 0,4)$  mm or a circular disk  $(79,8 \pm 0,4)$  mm in diameter using the punch and die set, and weighed to the nearest 0,005 g ( $M_2$ ) without release films.

The resin flow shall be calculated to the nearest 0,1 % as follows:

$$RF = \frac{100 (M_1 - 2M_2)}{M_1}$$

where

$RF$  is the resin flow (%)

$M_1$  is the original weight in (g)

$M_2$  is the weight of square or disk in (g)

When the test is used as a reference, a second set of specimens shall be tested, and the average of the two reported.

### 9.9.5 Report

The report shall include:

- a) the test method number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) the resin flow to the nearest 0,1 %;
- e) any deviation from this test method.

### 9.9.6 Additional information

If necessary, before punching the specimen, post cure the test specimen in accordance with the manufacturer's post-cure method.

The 170 °C temperature for the laminating press is suitable for epoxide resin systems only. For other resin systems, the temperature shall be determined upon agreement between purchaser and supplier.

## 9.10 Test 2M10: Glass transition temperature of base materials by differential scanning calorimetry (DSC)

### 9.10.1 Object

This test method covers the procedure for the determination of the glass transition temperature of base materials by differential scanning calorimetry. The glass transition temperature is indicated by an endothermic shift in the differential heat flow resulting from a change in the heat capacity of the material at that temperature.

### 9.10.2 Test specimens

The specimen shall be taken from the sheet in such a manner that it is not less than 25 mm from the edge.

The test specimens shall be of suitable size and shape for the specimen holder of the measuring system and weigh  $(0,010 \pm 0,030)$  g. The specimens may be prepared by sawing or punching.

For referee testing, a minimum of three specimens shall be tested.

### 9.10.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) Differential scanning calorimeter or differential thermal analyser capable of heating (cooling) at rates up to at least  $(20 \pm 1)$  °C/min, and of automatically recording differential heat flow or differential temperature between the specimen and a reference, to the required sensitivity and precision.
- b) Aluminum or other metal pans of high thermal conductivity specimen holders.
- c) An empty specimen pan or a reference pan filled with an inert reference material with a heat capacity approximately equivalent to the specimen (for example aluminum oxide).
- d) Recording charts for temperature-recording apparatus, with suitable graduations for recording of differential heat flow or differential temperature as a function of temperature. Instruments with digital data processing require an appropriate plotter or printer plotter.

- e) Nitrogen of 99,9 % purity or other inert gas supply, for blanketing specimen oxidation. If oxidative reactions are excluded, inert gas supply is not necessary. The dew point of the selected gas must be below the lowest operating temperature.

#### 9.10.4 Procedure

Use a specimen mass appropriate to the material to be tested. In most cases, a specimen mass of 0,010 g to 0,030 g specimen is satisfactory. As a reference, material with a heat capacity closely matched to that of the specimen or an empty pan may be used.

The specimen should be placed in the pan. After closing it with the relevant top by squeezing together the lower part and the top (cramping), a small hole shall be made in the top to allow vapour to escape. The crucible is then inserted into the test chamber of the instrument. The same procedure is followed for the reference as applicable.

Initiate flow of purge gas, as applicable.

Perform an initial thermal cycle from 30 °C to a temperature 15 °C above the expected  $T_g$  to erase previous thermal history at a rate of  $(20 \pm 2)$  K/min.

Hold temperature until a steady state is achieved (usually 5 min to 10 min).

Quench cool to  $(30 \pm 2)$  °C as rapidly as possible.

Hold temperature until a steady state is achieved (usually 5 min to 10 min).

As a referee, reheat at a rate of  $(10 \pm 2)$  K/min and record the heating curve until a temperature 25 °C higher than the observed  $T_g$ . For normal quality conformance testing,  $(20 \pm 2)$  K/min may be used.

Determine the midpoint temperature  $T_m$  (°C) as described in Figure 12, and report it as  $T_g$ .

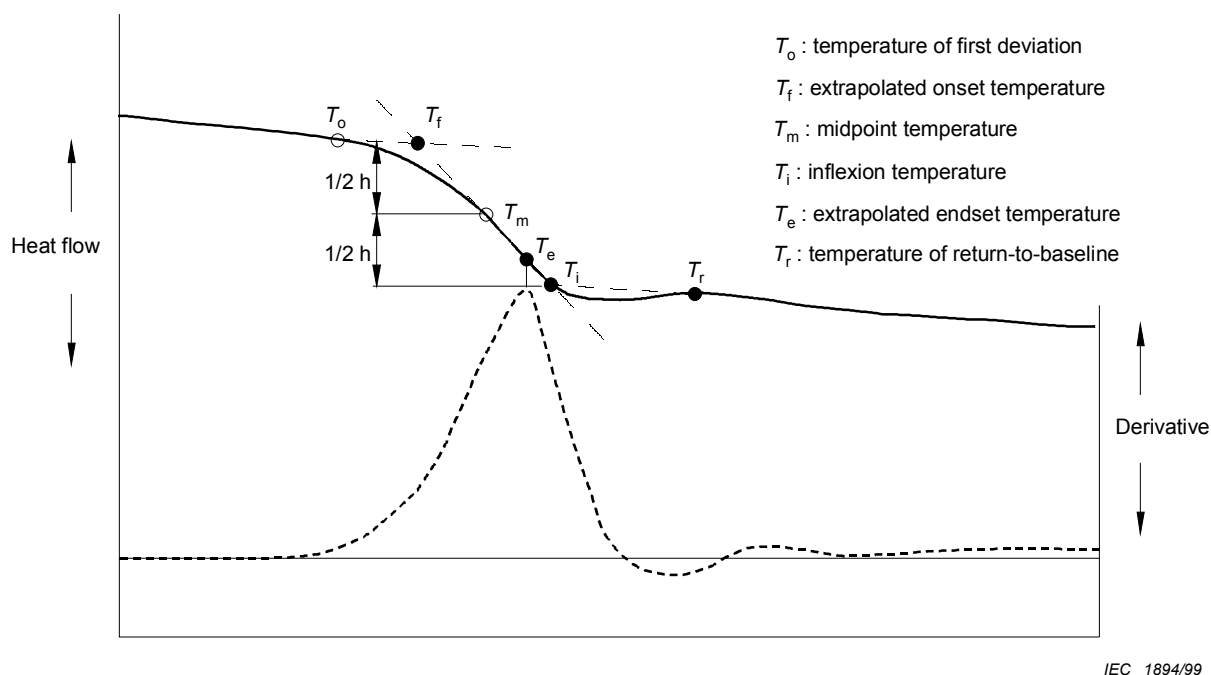


Figure 12 – Differential scanning calorimetry

(Characteristic transition points associated with glass transition)



### 9.10.5 Report

The report shall include:

- a) the test number and revision;
- b) the identification of the material tested;
- c) the quantity, form and weight of the specimens;
- d) the date of testing;
- e) the reference (i.e. empty crucible, etc.) and weight;
- f) the method of preparation of the specimens;
- g) the reheating rate if other than 10 °C per minute;
- h) the glass transition temperature (including information about the number of determinations used);
- i) additional information about test apparatus.

### 9.10.6 Additional information

The calibration of the DSC instrument shall be performed according to the recommendations of the manufacturer with a standard reference material appropriate to the temperature regions of interest.

## 9.11 Test 2M11: Glass transition temperature of base materials by thermomechanical analysis (TMA)

### 9.11.1 Object

The test method covers the procedure for the determination of the glass transition temperature of base materials by thermomechanical analysis (TMA). The glass transition temperature is indicated by the change in slope of the probe displacement curves in expansion mode resulting from a change in the rate of Z-axis expansion at that temperature.

### 9.11.2 Test specimens

The specimens shall be taken from the sheet in such a manner that they are not less than 25 mm from the edge.

The specimens shall be of suitable size and shape for the specimen holder of the measuring system and weigh (7,5 to 10) mg. The specimens shall be flat and parallel sided cross to direction of measurement and shall be free from burrs and fibres. The specimen may be prepared by sawing or punching.

For referee testing, a minimum of three specimens shall be tested.

### 9.11.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) A thermomechanical analyser or similar device consisting of a specimen holder into which the specimen can be placed. Changes in the length or in the compressive modulus of the specimen are sensed by the movement of a probe.
- b) A probe whose shape and size shall be such that the load applied to the specimen by the probe shall not cause indentation of the specimen. Flat, circular probes whose diameters are (2 to 5) mm are used.

- c) Means for sensing movement of the probe resulting from changes in length or compressive modulus of the specimen and for translating these movements into signals suitable for input to a chart recorder or data processing system. The sensing element should be capable of producing an electrical output of at least 1 mV per micrometer of probe movement with provision for less sensitive ranges when needed.
- d) A means of recording changes on specimen length or probe position as a function of specimen temperature. X-Y chart or strip chart recorders that have sensitivities of 1  $\mu\text{m}$  of probe deflection per centimetre of chart width or greater are acceptable. Instruments with digital data processing require an appropriate plotter or printer plotter.
- e) A means for uniformly heating the specimen at a predetermined rate over the temperature range of interest. Provisions should be made for pre-cooling the furnace and specimen where near ambient or sub-ambient temperature measurements are to be made. Heating and cooling rates of up to at least 10 K/min are required;
- f) Means for measuring the temperature of the test specimen.
- g) Means of purging the specimen environment with a dry inert gas, such as nitrogen or helium (the latter preferred due to its higher thermal conductivity). The dew point of the selected gas must be below the lowest operating temperature.

#### 9.11.4 Procedure

After mechanical removal of the metal, the specimens shall be dried for a minimum of 1 h at a temperature 20 °C below the expected  $T_g$ , but at a maximum of 110 °C.

Place a specimen having a thickness of 1 mm to 3 mm in the specimen holder under the probe. The specimen temperature sensor is placed in contact with the specimen or as near to the specimen as possible (whichever is recommended by the instrument manufacturer).

Move the furnace to enclose the specimen holder. Start the dry inert purge gas before cooling or heating the specimen. If measurements near ambient or sub-ambient temperatures are to be made, cool the specimen and furnace to at least 30 °C below the lowest temperature of interest. The refrigerant used for cooling should not come into direct contact with the specimen.

A load of 2 g shall be used.

Select an appropriate sensitivity setting on the recorder. Pre-analysis on a similar specimen may be run to provide this information.

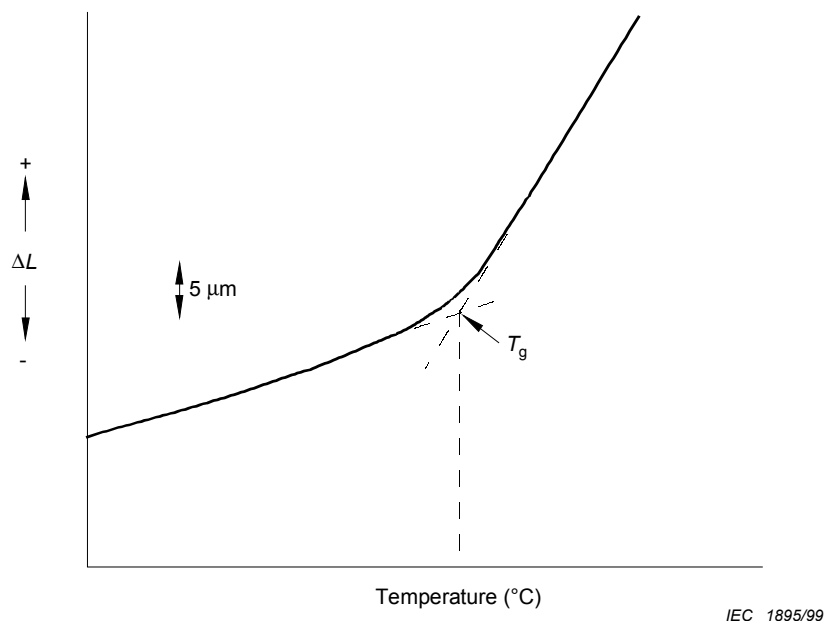
Heat the specimen at a constant heating rate of  $(10 \pm 2)$  K/min over the desired temperature range. Other rates may be used but must be noted in the report.

An abrupt change in the slope of the displacement curve indicates a transition of the material from one state to another. The projected temperature from the intersection of the extrapolated linear portions of the curve is used as the transition temperature  $T_g$ .

If residual stresses are evident (a sudden irreversible deflection at the glass transition) the heating should be stopped about 20 °C above this temperature. The temperature is then returned to the initial conditions and the run is repeated. The glass transition determined on this second run is reported along with the supplied heat treatment.

Determination of the glass transition temperature shall be made as follows (see Figure 13).

- 1) Construct tangents to the curve below and above the sharp bend. The intersection point of the tangents is the glass transition temperature of a specimen. For referee measurements the average of three determinations shall be reported as the  $T_g$  (°C).
- 2) Results obtained by retesting some specimens shall not be treated as an independent test of a new specimen.



Size: 1,50 mm

Rate: K

Atmosphere: inert gas

**Figure 13 – Thermomechanical analysis (expansion mode)**

#### 9.11.5 Report

The report shall include:

- a) the test number and revision;
- b) the identification of the material tested;
- c) the dimensions of the test specimen;
- d) the method of preparation of the test specimen;
- e) the specimen orientation with respect to the machine direction of the original base material;
- f) the specimen orientation in the holder;
- g) the temperature range;
- h) the heating range if other than 10 K/min;
- i) the glass transition temperature, with information about the number of determinations used;
- j) additional information about test apparatus;
- k) the testing date;
- l) any deviation from this test method;
- m) the name of the person conducting the test.

#### 9.11.6 Additional information

Calibration of the thermomechanical analyser shall be conducted in the temperature region of interest against appropriate standard reference materials. The calibration procedure shall employ the same heating rate, purge gas and purge gas flow as the test specimens.

## 9.12 Test 2M12: Surface waviness

### 9.12.1 Object

The test method covers the procedure for the determination of the surface waviness of metal-clad base materials essentially caused by the warp and fill threads of the glass fabric reinforcement.

### 9.12.2 Test specimens

The test specimens shall be cut not less than 25 mm from the edge of the sheet.

The test specimens shall be prepared from a sample of the metal-clad base material under test. They shall be of suitable size and shape for the measuring apparatus.

A single specimen should suffice for testing.

### 9.12.3 Test apparatus and materials

The following test apparatus and materials shall be used.

A contact profile meter for the measurement of surface roughness parameters by the profile method described in ISO 3274 shall be used, but with the following properties.

- a) The profile meter shall be able to record the wave spectrum of the surface roughness with a low pass filter characteristic. (The transmission of the instrument shall be in the range of low frequencies or long wave lengths.)
- b) The tip radius of the stylus shall be 5  $\mu\text{m}$  or 10  $\mu\text{m}$ .

### 9.12.4 Procedure

The procedure shall be carried out according to the instructions of the test equipment manufacturer using the following parameters:

- measuring length  $L_{\text{MW}} = 4 \text{ mm}$ ;
- cut off  $\lambda_{\text{B}} = 0,8 \text{ mm}$  (for low pass filter).

During the measurement the specimen shall be held stable in horizontal position.

Five measurements shall be made in machine (warp) and transverse (fill) direction each.

The average of the five measurements in either direction shall be recorded as surface waviness in the relevant direction.

In the case of materials clad on both sides, tests have to be carried out on each of them.

### 9.12.5 Report

The report shall include:

- a) the test number and revision;
- b) the identification of the material tested;
- c) the date of the test;
- d) the surface waviness in the machine direction (first side);
- e) the surface waviness in the transverse direction (first side);

- f) the surface waviness in the machine direction (second side);
- g) the surface waviness in the transverse direction (second side);
- h) any deviation from this test method;
- i) the name of the person conducting the test.

### 9.12.6 Additional information

Surface waviness is essentially caused by the type of reinforcement of the base material, for example the warp and fill threads of the glass fabric.

If no profile meter with low pass filter is available, an instrument with high pass filter can be used with the parameters

$$L_{MW} = 12,5 \text{ mm}$$

$$\lambda_B = 2,5 \text{ mm}$$

The resulting error in measurement is negligible; but, in this case, the information that an instrument with high pass filter is used shall be included in the report.

## 9.13 Test 2M13: Peel strength as received

### 9.13.1 Object

To determine the resistance to peeling under specified conditions. The test is performed on the sample as laminated and without conditioning. The test does not apply to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier.

### 9.13.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be prepared from a sample of the copper-clad base material under test and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup>, the foil mass per unit area may be increased to not more than 335 g/m<sup>2</sup> by any procedure in which ductile copper is deposited. The specimen shall be (50 ± 1) mm wide, and at least 75 mm long, and it shall be printed by any appropriate method in usual practice with the test pattern of Figure 11.
- c) The number of specimens used for one test shall be sufficient for the peeling of at least 25 mm from each of four strips of copper foil.
- d) Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique for example by sticking the sample to a rigid material.

### 9.13.3 Test apparatus and materials

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of (50 ± 5) mm/min;
- b) a clamp to grip the detached end of the foil over its entire width.

#### 9.13.4 Procedure

One end of the strip of the metal foil shall be detached from the base material for a distance of at least 10 mm, and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width in the jaws of the clamp on the tensile testing machine, and a steady increasing pull shall be applied in a direction  $(90 \pm 5)^\circ$  to the plane of the base material until the metal foil is peeled off at a rate of  $(50 \pm 5)$  mm/min, the force to do this being measured. A length of at least 25 mm shall be peeled at this rate from each of four strips.

Should copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup> break during the test or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described as above to a total mass per unit area not less than 275 g/m<sup>2</sup>, but not more than 335 g/m<sup>2</sup>.

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in newtons per millimetre.

#### 9.13.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) a statement certifying that the test was carried out "as received";
- e) the measured peel strength;
- f) whether additional copper has been added to the test specimens;
- g) any deviation from this test method.

#### 9.13.6 Additional information

None.

### 9.14 Test 2M14: Peel strength after heat shock

#### 9.14.1 Object

To determine the resistance to peeling under specified conditions and after specified heat shocks simulating soldering processes. The test is performed on the sample as manufactured and without conditioning. The test does not apply to the outer 25 mm of the laminate sheet as manufactured and provided by the supplier.

#### 9.14.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be prepared from a sample of the copper-clad base material under test and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup>, the foil mass per unit area may be increased to not more than 335 g/m<sup>2</sup> by any procedure in which ductile copper is deposited. The specimen shall be  $(50 \pm 1)$  mm wide, and at least 75 mm long, and it shall be printed by any appropriate method in usual practice with the test pattern of Figure 11.

- c) The number of specimens used for one test shall be sufficient for the peeling of at least 25 mm from each of four strips of copper foil.
- d) Test specimens prepared from samples of metal-clad polymeric films may require further preparation before being tested for peel strength, to avoid the peeling of any adhesive layer from the base film over an area wider than the test strip. If necessary, the base material shall be inscribed with a sharp blade on both sides of each test strip ensuring that any adhesive layer has been severed, but with insufficient force to cut the base film.
- e) Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique for example by sticking the sample to rigid material.

### 9.14.3 Test apparatus and material

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of  $(50 \pm 5)$  mm/min;
- b) a clamp to grip the detached end of the foil over its entire width;
- c) a desiccator (stabilization chamber) capable of maintaining 25 % RH or less at room temperature;
- d) a bath meeting one of the following requirements:
  - 1) a liquid bath of well-stirred silicone or equivalent fluid, kept at  $(260^{+5}_0)$  °C, throughout the test shall be used. The temperature shall be measured  $(25 \pm 2,5)$  mm below the surface used.
  - 2) a fluidized sand bath of suitable design, kept at a temperature of  $(260^{+5}_0)$  °C. The temperature shall be measured in approximately the same location that will be occupied by the specimen;
  - 3) a suitable solder bath of depth not less than 40 mm shall be used. If circular, the bath shall be not less than 120 mm in diameter, and if rectangular, not smaller than 100 mm × 75 mm. The bath shall be protected from draughts. The temperature of the solder shall be kept at  $(260^{+5}_0)$  °C throughout the test. The temperature shall be measured at a depth of  $(25 \pm 2,5)$  mm below the surface. The bath shall contain a solder of nominal 60/40 or 63/37 tin/lead composition with a melting temperature range as follows:
    - completely solid: 183 °C;
    - completely liquid: 188 °C.

### 9.14.4 Procedure

If specified in the relevant specification for the material under test, the specimen shall be preconditioned, allowed to cool to room temperature in a desiccator and subjected to heat shock within 30 min of removal from the preconditioning atmosphere.

#### 9.14.4.1 Heat shock

The specimen shall be subjected to heat shock by one of the following three methods, the choice between them to be subject to agreement between purchaser and supplier.

##### 9.14.4.1.1 Liquid bath method

Bath as described in 9.14.3 d)1). The specimen shall be held in a horizontal position, at a depth of  $(25 \pm 2,5)$  mm in a holder of heat capacity low enough to avoid the fluid temperature falling below 260 °C. The specimen shall be totally immersed in the fluid for the time given in the relevant specification.

#### **9.14.4.1.2 Fluidized sand bath method**

Bath as described in 9.14.3 d) 2). The specimen shall be immersed edgewise, that is with its surface at right angles to the bath surface, for the time given in the relevant specification.

#### **9.14.4.1.3 Solder float method**

Bath as described in 9.14.3 d) 3). Specimens of metal-clad polymeric film may be attached to the lower surface of a block of cork or other thermal insulation using drawing pins or other low mass holding devices.

The conductive pattern on the specimen shall be prepared by a treatment which prevents the adhesion of solder to the metal, and which has a negligible effect on heat transfer.

The specimen shall then be dropped, etched side downwards, onto the surface of the clean molten solder for the time given in the relevant specification.

#### **9.14.4.2 Visual examination**

After removal from whichever source of heat shock has been used, the specimen shall be examined for blistering or delamination. If it has not blistered or delaminated, it shall be allowed to cool to between 15 °C and 35 °C and then be used for the measurement of peel strength after heat shock.

#### **9.14.4.3 Peeling**

One end of the strip of the metal foil shall be detached from the base material for a distance of at least 10 mm, and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width in the jaws of the clamp on the tensile testing machine, and a steady increasing pull shall be applied in a direction  $(90 \pm 5)^\circ$  to the plane of the base material until the metal foil is peeled off at a rate of  $(50 \pm 5)$  mm/min, the peel force shall be recorded. A length of at least 25 mm shall be peeled at this rate from each of four strips.

Should copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup> break during the test or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described above to a total mass per unit area not less than 275 g/m<sup>2</sup>, but not more than 335 g/m<sup>2</sup>.

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in N per millimetre.

#### **9.14.5 Report**

The report shall include:

- a) the test number and revision index;
- b) the test date;
- c) the identification of the material tested;
- d) a statement certifying that the test has been carried out "after heat shock";
- e) the heat shock method used;
- f) the time during which the specimen was exposed to the heat shock;
- g) the measured peel strength;
- h) whether additional copper has been added to the test specimen;
- i) whether there was blistering or delamination after the heat shock;
- j) any deviation from this test method.



#### 9.14.6 Additional information

#### 9.14.7 Safety precautions

Avoid eye and skin contact with hot materials by using suitable protection.

### 9.15 Test 2M15: Peel strength after dry heat

#### 9.15.1 Object

To determine the resistance to peeling under specified conditions and after exposure to high temperature. The test is performed on the specimen as manufactured and without conditioning. The test does not apply to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier.

#### 9.15.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be prepared from a sample of the copper-clad base material under test and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup>, the foil mass per unit area may be increased to not more than 335 g/m<sup>2</sup> by any procedure in which ductile copper is deposited. The specimen shall be (50 ± 1) mm wide, and at least 75 mm long, and it shall be printed by any appropriate method in usual practice with the test pattern of Figure 11.
- c) The number of specimens used for one test shall be sufficient for the peeling of at least 25 mm from each of four strips of copper foil.
- d) Test specimens prepared from samples of copper-clad polymeric films may require further preparation before being tested for peel strength, to avoid the peeling of any adhesive layer from the base film over an area wider than the test strip. If necessary, the base material shall be inscribed with a sharp blade on both sides of each test strip ensuring that any adhesive layer has been severed, but with insufficient force to cut the base film.
- e) Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique, for example by sticking the sample to a rigid material.

#### 9.15.3 Test apparatus and materials

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of (50 ± 5) mm/min;
- b) a clamp to grip the detached end of the foil over its entire width;
- c) circulating air chamber capable of maintaining (200 ± 2) °C in the working space. Forced air circulation may be used to maintain homogenous conditions. In order to limit radiated heat problems, the temperature of the walls may not differ by more than 3 % from the specified ambient temperature of the test. The absolute humidity inside the test chamber shall not exceed 20 g of water vapour per cubic metre of air (corresponding to approximately 50 % RH at 35 °C).

#### 9.15.4 Procedure

The specimen shall be exposed to high temperature using the chamber described in 9.15.3c, for 500 h, unless otherwise specified. The temperature of the air in the heating chamber shall be that given in the relevant specification. The air in the chamber shall circulate during the entire heating period, and the large surfaces of the specimen shall be parallel with the air flow.

The specimen shall be removed from the air chamber and placed in standard laboratory conditions of  $(23 \pm 2) ^\circ\text{C}$  and  $(45 \pm 5) \% \text{RH}$ . After a minimum of 1 h, the specimen shall be examined for blistering or delamination. If it has not blistered or delaminated, it shall then be used for the measurement of peel strength after dry heat.

One end of the strip of the metal foil shall be detached from the base material for a distance of at least 10 mm, and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width, in the jaws of the clamp on the tensile testing machine, and a steady increasing pull shall be applied in a direction  $(90 \pm 5)^\circ$  to the plane of the base material until the metal foil is peeled off at a rate of  $(50 \pm 5) \text{ mm/min}$ , the force to do this being measured. A length of at least 25 mm shall be peeled at this rate from each of four strips.

Should copper foil of nominal mass per unit area less than  $305 \text{ g/m}^2$  break during the test or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described in 9.15.2 b) to a total mass per unit area not less than  $275 \text{ g/m}^2$ , but not more than  $335 \text{ g/m}^2$ .

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in N per millimetre.

#### 9.15.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) a statement certifying that the test has been carried out "after dry heat";
- e) the time during which the specimen was exposed to the dry heat;
- f) the temperature of the heating chamber;
- g) the recovery if not standard;
- h) the measured peel strength;
- i) whether additional copper has been added to the test specimen;
- j) whether there was blistering or delamination after the dry heat;
- k) any deviation from this test method.

#### 9.15.6 Additional information

None.

### 9.16 Test 2M16: Peel strength after simulated plating

#### 9.16.1 Object

To determine the resistance to peeling under specified conditions and after simulated plating. The test is performed on the sample as manufactured. The test does not apply to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier.

#### 9.16.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.

- b) The test specimen shall be prepared from a sample of the copper-clad base material under test, and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than  $305 \text{ g/m}^2$ , the foil mass per unit area may be increased to not more than  $335 \text{ g/m}^2$  by any procedure in which ductile copper is deposited. The specimen shall be  $(50 \pm 1) \text{ mm}$  wide, and at least  $75 \text{ mm}$  long, and it shall be printed by any appropriate method in usual practice with the test pattern in Figure 11.
- c) The number of specimens used for one test shall be sufficient for the peeling of at least  $25 \text{ mm}$  from each of four strips of copper foil.
- d) Test specimens prepared from samples of copper-clad polymeric films may require further preparation before being tested for peel strength, to avoid the peeling of any adhesive layer from the base film over an area wider than the test strip. If necessary, the base material shall be inscribed with a sharp blade on both sides of each test strip ensuring that any adhesive layer has been severed, but with insufficient force to cut the base film.
- e) Specimens with a thickness less than  $0,8 \text{ mm}$  shall be built up to sufficient rigidity by a suitable technique for example by sticking the sample to a rigid material.

### 9.16.3 Test apparatus and materials

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of  $(50 \pm 5) \text{ mm/min}$ ;
- b) a clamp to grip the detached end of the foil over its entire width;
- c) an equipment to use as an electrolytic cell consisting of:
  - a carbon rod to use as anode;
  - an electrolyte; a well-stirred aqueous solution of sodium sulphate, whose concentration is  $10 \text{ g}$  anhydrous sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) per litre of distilled water, and whose temperature is maintained at  $(70 \pm 2) ^\circ\text{C}$ .

### 9.16.4 Procedure

The specimen shall be made the cathode of the electrolytic cell as described under 9.16.3 c). The specimen shall be held with the strips of its test pattern vertical so that they are just fully submerged. They shall be connected electrically by any suitable means and a d.c. voltage of  $(5 \pm 1) \text{ V}$  shall be applied to the cell and adjusted so that the current has a steady value equivalent to approximately  $215 \text{ A/m}^2$  of the submerged foil. At the end of  $(20 \pm 1) \text{ min}$ , the test specimen shall be removed from the solution, wiped to remove surface moisture and left about  $1/2 \text{ h}$  to cool to room temperature.

The specimen shall be examined for non-adherence of the foil. If the foil has not become detached, it shall be used for the measurement of peel strength after simulated plating.

One end of the strip of the metal foil shall be detached from the base material for a distance of at least  $10 \text{ mm}$ , and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width, in the jaws of the clamp on the tensile testing machine, and a steady increasing pull shall be applied in a direction  $(90 \pm 5)^\circ$  to the plane of the base material until the metal foil is peeled off at a rate of  $(50 \pm 5) \text{ mm/min}$ , the peel force shall be recorded. A length of at least  $25 \text{ mm}$  shall be peeled at this rate from each of four strips.

Should copper foil of nominal mass per unit area less than  $305 \text{ g/m}^2$  break during the test or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described in 9.16.2 b) to a total mass per unit area not less than  $275 \text{ g/m}^2$ , but not more than  $335 \text{ g/m}^2$ .

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in newtons per millimetre.

### 9.16.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) a statement certifying that the test has been carried out "after simulated plating";
- e) the measured peel strength;
- f) whether additional copper has been added to the test specimen;
- g) whether there was non-adherence of the foil after the simulated plating treatment;
- h) any deviation from this test method.

### 9.16.6 Additional information

The simulated plating treatment may be carried out conveniently using 500 cm<sup>3</sup> of solution in a 600 cm<sup>3</sup> wide-necked flask, and a glass stirrer. A variable resistance of about 30 Ω capable of taking 0,2 A, and an ammeter to measure 0,2 A, will control and measure the current required.

## 9.17 Test 2M17: Peel strength at high temperature

### 9.17.1 Object

To determine the resistance to peeling under specified conditions and at a specified high temperature, after exposure to that specified temperature for a specified time. The test is performed on the sample as manufactured and without conditioning. The test does not apply to the outer 25 mm of the laminated sheet as manufactured and provided by the supplier.

### 9.17.2 Test specimens

- a) Specimens shall be taken from the panel or sheet in such a way that they are at least 25 mm from the edge.
- b) The test specimen shall be prepared from a sample of the copper-clad base material under test and before it is etched, its thickness shall normally be that of the material. However, in the case of materials clad with copper foil of nominal mass per unit area less than 305 g/m<sup>2</sup>, the foil mass per unit area may be increased to not more than 335 g/m<sup>2</sup> by any procedure in which ductile copper is deposited. The specimen shall be (50 ± 1) mm wide, and at least 75 mm long, and it shall be printed by any appropriate method in usual practice with the test pattern in Figure 11.
- c) The number of specimens used for one test shall be sufficient for the peeling of at least 25 mm from each of four strips of copper foil.
- d) Test specimens prepared from samples of copper-clad polymeric films may require further preparation before being tested for peel strength, to avoid the peeling of any adhesive layer from the base film over an area wider than the test strip. If necessary, the base material shall be inscribed with a sharp blade on both sides of each test strip, ensuring that any adhesive layer has been severed, but with insufficient force to cut the base film.
- e) Specimens with a thickness less than 0,8 mm shall be built up to sufficient rigidity by a suitable technique for example by sticking the sample to a rigid material.

### 9.17.3 Test apparatus and materials

The following test apparatus and material shall be used:

- a) a tensile testing machine that achieves a peel rate of (50 ± 5) mm/min;
- b) a clamp to grip the detached end of the foil over its entire width;

- c) a bath of well-stirred silicone or equivalent liquid/air media, kept within  $\pm 2$  °C of the temperature given in the relevant specification;
- d) a circulating air oven maintained within  $\pm 2$  °C of the temperature given in the relevant specification. As a minimum the chamber should completely surround the test specimen and clamping jaws.

#### 9.17.4 Procedure

One end of the strip of the metal foil shall be detached from the base material for a distance of at least 10 mm, and sufficient for the apparatus used. The detached end of the foil shall be gripped over its entire width, in the jaws of the clamp on the tensile testing machine, before immersion in the hot fluid as described in 9.17.3 c). The specimen shall be held in a horizontal position in the fluid, at a depth of  $(25 \pm 2,5)$  mm in a holder of heat capacity low enough to avoid the temperature of the fluid from falling below the range specified. The specimen shall be totally immersed in the fluid for 2 min minimum before a steady increasing pull shall be applied in a direction  $(90 \pm 5)^\circ$  to the plane of the base material until the metal foil is peeled off at a rate of  $(50 \pm 5)$  mm/min, the peel force shall be recorded. A length of at least 25 mm shall be peeled at this rate from each of four strips.

For the testing of peel strength four specimens shall be prepared and only one conductor of each specimen shall be tested. This test may alternatively be carried out in a circulating-air oven as described in 9.17.3 d).

In that case the test shall be carried out after the specimen has reached the required temperature, and shall be completed within 75 min after the specimen and apparatus have been introduced into the oven.

Should copper foil of nominal mass per unit area less than  $305 \text{ g/m}^2$  break during the test, or fail to meet the requirements of the relevant specification, a further set of four strips shall be tested from material to which additional copper has been added as described in 9.17.2 b) to a total mass per unit area not less than  $275 \text{ g/m}^2$ , but not more than  $335 \text{ g/m}^2$ .

The minimum load per unit width required to peel the metal foil during the test shall be taken as the peel strength. It shall be expressed in newtons per millimetre.

#### 9.17.5 Report

The report shall include:

- a) the test number and revision index;
- b) the testing date;
- c) the identification of the material tested;
- d) a statement certifying that the test has been carried out "at high temperature";
- e) the temperature at which the test has been carried out;
- f) the measured peel strength;
- g) whether additional copper has been added to the test specimen;
- h) any deviation from this test method.

#### 9.17.6 Additional information

None.

**9.18 Test 2M18: Surface quality** (under consideration)**9.19 Test 2M19: Punching** (under consideration)**9.20 Test 2M20: Flexural strength****9.20.1 Object**

To determine the flexural strength of rigid insulating base materials. The test is performed on the specimen as manufactured and under the condition defined. The test shall only be applied to specimens 0,80 mm or greater in thickness. It applies to a freely supported beam, loaded at midspan ( three-point loading test ).

**9.20.2 Test specimens**

- The test specimens shall be prepared from a sample of the metal-clad base material under test, from which the metal has been completely removed, its thickness shall be that of the base material.
- Dimensions of the specimens shall be as shown in Table 3. The specimens shall be free of fracture and delamination. The edges shall be smoothed by sanding or equivalent means (do not radius the edges).
- Unless otherwise specified, ten specimens shall be tested, five in the lengthwise and five in the crosswise direction of the sample sheet or panel.

**Table 3 – Specimen dimensions**

Specimen dimensions			Test parameters	
Nominal thickness $h$ mm	Width $w$ mm $\pm 0,5$	Length $L$ mm	Span $L_v$ mm $\pm 0,5$	Speed of testing $N$ – mm per min $\pm 0,2$
$0,8 < h \leq 3$	25,0	At least $20 h$	$16 h$	$h / 2$
$3 < h \leq 5$	10,0			
$5 < h \leq 10$	15,0			

**9.20.3 Test apparatus and materials**

The following test apparatus and material shall be used:

- A standard tension and compression test apparatus which can be operated at a constant rate of crosshead movement shown in Table 3. The error in the load measuring system shall not exceed  $\pm 1$  %. The loading nose and supports shall have cylindrical surfaces. The radius of nose ( $R_1$ ) and the radius of supports ( $R_2$ ) shall be as follows:  $R_1 = (5,0 \pm 0,1)$  mm,  $R_2 = (2,0 \pm 0,2)$  mm for thicknesses of the test specimen  $\leq 3$  mm, and  $R_2 = (5,0 \pm 0,2)$  mm for thickness of the test specimen  $> 3$  mm. The span  $L_v$  shall be adjustable.
- Etching system capable of complete removal of the metallic cladding.
- Measuring devices capable of determining specimen width  $w$  and supported beam span  $L_v$  to the nearest 0,025 mm and the specimen thickness  $h$  to the nearest 0,0025 mm.

**9.20.4 Procedure**

- Set tester for the required span and crosshead vertical speed as specified in Table 3.
- Align the loading nose and supports so that the axes of the cylindrical surfaces are parallel and the loading nose is midway between the supports.
- The test specimens shall be at room temperature. Centre the specimen on the support with the long axis of the specimen perpendicular to the loading nose and supports.

- d) Apply the load at the speed of testing from Table 3 until the specimen breaks. The load at breakage shall be recorded in newton ( $P$ ).

The flexural strength ( $\sigma_{fB}$ ) of each specimen shall be calculated as follows.

$$\sigma_{fB} = \frac{3PLv}{2wh^2} \left( \text{N/mm}^2 = \text{MPa} \right)$$

where

- $P$  is the applied force at breakage, in newtons;  
 $Lv$  is the span, in millimetres;  
 $w$  is the width of the specimen in millimetres;  
 $h$  is the thickness of the specimen in millimetres.

### 9.20.5 Report

The report shall include:

- the test number and revision index;
- the testing date;
- the identification of the material tested;
- the average of the calculated flexural strengths;
- the individual test results, if required;
- any deviation from this test method;
- the name of the person who conducted the test.

### 9.20.6 Additional information

None.

## 9.21 Test 2M21: Rolling fatigue of flexible base materials

### 9.21.1 Object

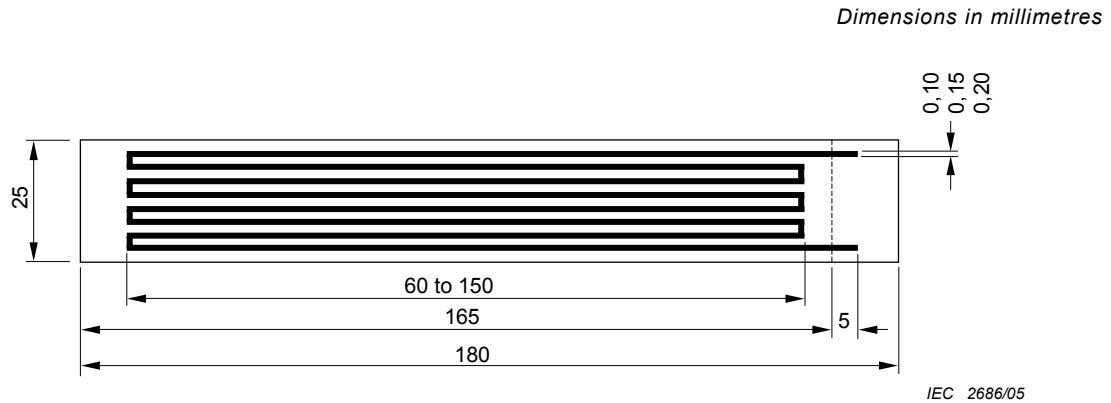
To determine the ability of a flexible metal-clad laminate to withstand repeated flexing without cracking or detachment of the metal cladding.

### 9.21.2 Test specimens

The test specimen shall be a piece of the flexible material clad material under test and, before it is etched, its thickness shall be that of the material. The specimen shall be made with the pattern shown in Figure 14. The details of the specimen are shown below:

- The pattern consists of a single conductor with the following widths and spacing (see Figure 14):
  - 0,1 mm/0,1 mm;
  - 0,15 mm/0,15 mm;
  - 0,2 mm/0,2 mm.
- Pattern segments 8 to 12 pattern (see Figure 14).
- Length of segments: 60 mm to 150 mm.
- Typical specimen:
  - conductor: machine direction (MD);
  - COVERLAY side: loop inside.

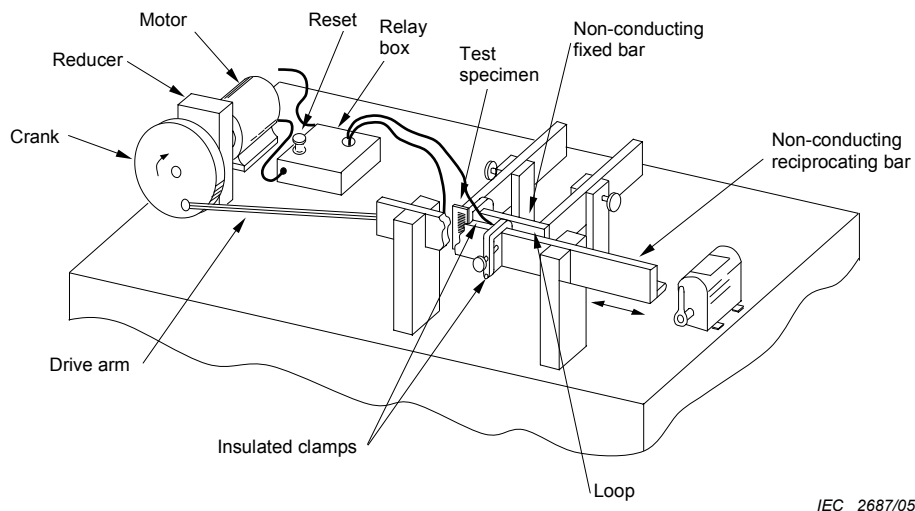
- e) Reference specimens:  
MD/loop outside;  
counter machine direction (CMD)/loop inside;  
CMD/loop outside.
- f) Number of specimen: 5 pieces for each test.



**Figure 14 – Test specimen pattern**

### 9.21.3 Test apparatus and materials

- a) The general arrangement of the apparatus is shown in Figure 15. This allows one end of the test specimen to be held with an insulating clamp against the face of a fixed non-conducting bar.
- b) The other end of the specimen can be similarly clamped to the face of the second non-conducting bar mounted parallel to the first so that 180° loop of the specimen is formed between the two bars and the distance between the bars can be adjusted to vary the diameter of the loop. The second bar slides in the direction of its major dimension and can be reciprocated over a travel of approximately 30 mm at a rate of not less than 50 cycles/min. A relay is included in the apparatus so that any discontinuity of 10 ms or longer in a circuit which includes the conductive pattern on the test specimen will cause the motor driving the reciprocating bar to stop. A counter indicates the number of cycles completed during a test.



**Figure 15 – General arrangement of apparatus**



#### 9.21.4 Procedure

- a) A short length of insulated wire shall be connected to the extreme ends of the conductive pattern.
- b) The test specimen shall be mounted between the parallel bars of the test apparatus so that the inside radius of the loop is 2,0 mm and the wires shall be connected to the relay. In the test of the material clad on both sides the two conductive patterns shall be connected in series to the relay.
- c) The reciprocating travel shall be such that the loop travels at 20 mm and the specimen does not bend at either clamp. The rate of reciprocation shall be approximately 10 cycles/min.
- d) The test shall be conducted by reciprocating the movable bar until electrical discontinuity in the conductive pattern on the test specimen causes the relay to stop the motor, or until the required number of cycles has been completed without failure (detachment of the copper foil from the flexible base material is also failure).
- e) The test shall be performed on five specimens in the machine direction with the conductive pattern on the inside of the loop as a typical case. It shall be repeated similarly using each five specimens of machine direction and loop outside, cross-machine direction on loop inside and cross-machine direction on loop outside if it is necessary.
- f) The minimum number of cycles needed to cause discontinuity shall be taken as the value of flexural fatigue.
- g) If the flexible material under test is metal clad on both sides, the test shall be performed on both sides; the reverse side shall be removed by etching before testing.

#### 9.21.5 Report

The report shall include:

- a) the test number and revision;
- b) the date of the test;
- c) the identification and description of the specimens;
- d) conductor width/pattern space (mm);
- e) length of conductor pattern (mm);
- f) diameter of loop (mm);
- g) the number of cycles to cause electrical discontinuity in the conductive pattern or the number of cycles completed without failure, or the fact that detachment of the copper foil of the flexible base material occurred. The fatigue number is the average number for 5 pieces;
- h) the name of the person conducting the test;
- i) any deviation from this test method.

### 9.22 Test 2M22: Weight of foil after lamination

#### 9.22.1 Object

To determine the mass per unit area of metal foils after lamination (by etching).

#### 9.22.2 Test specimens

Three specimens, approximately 100 mm × 100 mm, from a sample of the metal-clad base material under test.

### 9.22.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) Analytical balance with an accuracy of 0,002 g.
- b) Slide caliper.
- c) An oven capable of maintaining a temperature of  $(105 \pm 5) ^\circ\text{C}$ .
- d) Any suitable etching solution of commercial practice may be used. As referee solution ferrichloride with a density of  $(1,32 - 1,41) \text{ g/cm}^3$  room temperature shall be used.
- e) A desiccator or drying chamber with calcium chloride or similar as drying medium.

### 9.22.4 Procedure

Measure the dimensions of the specimens to an accuracy of  $\pm 0,1 \text{ mm}$ . Calculate the areas in  $\text{m}^2$  (A).

Precondition the specimens for 24 h at  $(23 \pm 2) ^\circ\text{C}$ ,  $(50 \pm 5) \%$  relative humidity, then weigh to an accuracy of 0,002 g ( $M_1$ ).

Remove the copper foil completely by etching. Rinse the specimens in cold running water until the water forms an intact film on the surface.

Dry the specimens for 1 h at  $(105 \pm 5) ^\circ\text{C}$ , Allow to cool down to room temperature in a desiccator and reweigh it ( $M_2$ ).

Materials clad with metal foils on both sides shall be tested as above unless it is necessary to determine the mass of the individual foils, for example, when different foils are used on opposite sides or in case of dispute. In these cases, separate specimens shall be used to determine the foil mass on each side. The foil on the reverse side shall be protected by a suitable resist applied before the copper foil on the upper side is removed.

Calculation:

Metal foil on one side, or different foils on opposite sides:

$$\text{Mass per unit area} = \frac{(M_1 - M_2)}{A} \text{ g/m}^2$$

An average of the three measurements is reported.

Metal foil on both sides, the same nominal thickness:

$$\text{Mass per unit area} = \frac{(M_1 - M_2)}{2A} \text{ g/m}^2$$

An average of the three measurements is reported.

### 9.22.5 Report

The report shall include:

- a) test method number and revision;
- b) identification of the test material;

- c) date of test;
- d) mass per unit area in g/m<sup>2</sup> for each side;
- e) any deviation from this test method;
- f) the name of the person conducting the test.

#### **9.22.6 Additional information**

None.

### **9.23 Test method 2M23: Rectangularity of cut panels**

#### **9.23.1 Object**

This test method covers the procedure for determination of the deviation from a 90° angle on corners of cut-to size panels.

The test is applicable to panels of maximum dimension less than or equal to 610 mm.

#### **9.23.2 Test specimen**

The tests shall be made on cut-to-size production panels. A minimum of three panels shall be used for the test.

#### **9.23.3 Test apparatus and materials**

The following test apparatus and materials shall be used:

- a) A square test fixture with a 90° angle of dimension large enough to encompass the maximum dimension of the panel.
- b) A taper or feeler gauge.

#### **9.23.4 Procedure**

- a) Each corner of the panel shall be checked by holding the panel against one face of the square test fixture with its corner in the corner of the fixture and the maximum distance between the free panel edge and the adjacent fixture edge is measured with a taper or feeler gauge.
- b) The deviation value reported shall be the maximum observed measurement of all corners in the three test specimens.

#### **9.23.5 Report**

The report shall include:

- a) test method number and revision;
- b) identification and description of the test materials;
- c) date of testing;
- d) dimensions of the panel;
- e) the maximum observed deviation from 90° in millimetres;
- f) the number of panels tested;
- g) any deviation from this method;
- h) the name of the person conducting the test.

### 9.23.6 Additional information

None.

### 9.24 Test 2M24: Coefficient of thermal expansion (under consideration)

### 9.25 Test 2M25: Time to delamination by thermomechanical analysis (TMA)

#### 9.25.1 Object

The time to delamination test method by TMA measures the resistance of a base material, printed board or printed board assembly to delaminate between dielectric layers by heating isothermally at 260 °C in a TMA cell. This test is not applicable to rigid multilayer printed boards with bonded heat sinks or heavy metal layers without modification to the heating rate.

This test is performed in support of the more common and more frequently used thermal shock tests to evaluate interlaminar adhesion under thermal stress.

#### 9.25.2 Test specimens

- a) Specimens shall be cut not less than 25 mm from the edge of the sheet.
- b) Specimens shall be a square of minimum  $(6 \pm 2)$  mm. Larger specimens can be used provided they do not exceed equipment constraints. The specimens shall be flat, uniform in thickness, and free from burrs and fibres. The sample may be prepared by sawing or punching.
- c) When used as a referee method, a minimum of three specimens shall be used. As a routine quality assurance activity, one specimen shall be sufficient.

#### 9.25.3 Test apparatus and material

The following test apparatus and materials shall be used:

A thermomechanical analyser (TMA) or similar device consisting of

- a) a specimen holder into which the specimens are placed;
- b) a flat, circular probe having a diameter of 2 mm to 5 mm. The shape and size of the probe shall be such that the load applied to the specimen shall not cause indentation of the specimen. The clearance between the probe and the specimen shall be within 1 mm;
- c) means for sensing movement of the probe resulting from changes in thickness of the specimen and for translating these movements into a signal suitable for input to a recorder or data processing system. The sensing element should be capable of producing an electrical output of at least 1 mV/ $\mu$ m of the probe movement with provision for less sensitive ranges when needed;
- d) a means for recording changes in the specimen thickness. X-Y or strip chart recorders that have sensitivities of 1  $\mu$ m of probe deflection per centimetre of chart deflection are acceptable. Instruments with digital and data processing require an appropriate plotter or printer plotter;
- e) a means for heating the specimen and holding it thermally at 260 °C.

#### 9.25.4 Procedure

As a referee, the specimen may be preconditioned in an air circulating chamber at 125 °C for a time designated in the appropriate specification, then allowed to cool down under standard atmospheric conditions until the temperature is below 35 °C. The recovery time shall not exceed 8 h. As a routine quality assurance activity, the sample shall be tested as received.

The specimen shall be placed in the specimen holder under the probe with the centre of the probe as close to the centre of the specimen as possible with a minimum clearance of 1 mm. The specimen temperature sensor shall be placed in contact with the specimen or as near to the specimen as possible (whichever is recommended by the manufacturer).

The specimen holder shall be enclosed in the furnace.

A load of 2 g shall be used.

An appropriate sensitivity setting on the recorder shall be selected.

The specimen shall be heated at the maximum heating rate of the instrument utilized not to exceed 100 °C/min until the specimen reaches 260 °C.

The specimen shall be held at 260 °C until the delamination end point is reached.

The delamination end point is a sudden, irreversible increase in thickness of the specimen due to layer-to-layer delamination.

The time to delamination at 260 °C is the time in minutes and seconds from the beginning of the heat-up, through isothermal conditions up until delamination occurs.

#### **9.25.5 Report**

The report shall include:

- a) the test number and revision index;
- b) the date of analysis;
- c) the identification of the material tested;
- d) the average of three test specimens or the single-point measurement;
- e) the actual rate utilized;
- f) any deviations from this test method.

#### **9.25.6 Additional information**

This test can also be applied to metal core boards or other special structures with modification to the heating rate.

### **9.26 Test 2M26: Scaled flow test for prepreg materials**

#### **9.26.1 Object**

This procedure defines a test method used to determine the pressed thickness of a prepreg comprised of epoxide resin and reinforcement. The test is appropriate for checking prepreg consistency, but is not intended to define the suitability of the prepreg for use in a specific printed board process or application.

#### **9.26.2 Test specimens**

The prepreg specimen size shall be  $(140 \pm 1) \text{ mm} \times (180 \pm 1) \text{ mm}$ . Specimens shall be cut with the 180 mm dimension parallel to the machine direction of the prepreg.

Each laminated specimen shall be comprised of the number of prepreg plies specified in Table 4.

### 9.26.3 Test apparatus and materials

The following test apparatus and materials shall be used.

- a) A lamination press with a minimum heating platen size of 200 mm × 200 mm, capable of maintaining uniform pressure of  $(21 \pm 1)$  N/cm<sup>2</sup>, and capable of maintaining a temperature of  $(150 \pm 2)$  °C. The heating platen shall be flat and parallel within 0,25 mm.
- b) Press plates. The press plates used shall be steel metal between 3,0 mm and 6,5 mm thick, and shall be  $(115 \pm 1)$  mm ×  $(150 \pm 1)$  mm in size. The press plate shall be flat and parallel within 0,025 mm.
- c) Release material. The release material shall be polyvinyl fluoride (PVF) or equivalent, at least 220 mm × 220 mm in size.
- d) Temperature-resistant adhesive tape. The tape shall be suitable for holding the specimens during lamination.
- e) Prepreg cutting equipment. The cutter shall be capable of maintaining the specimen tolerances outlined in 9.26.2.
- f) Micrometer. A measuring instrument with a resolution of 0,002 mm.
- g) Stabilization chamber. A stabilization chamber (dry cabinet) with suitable desiccant (calcium chloride or equivalent) capable of maintaining less than 10 % RH at  $(21 \pm 2)$  °C.

### 9.26.4 Procedure

The specimens shall be cut to size and then placed in the stabilization chamber for a period of 24 h. The laminating process shall be performed within 15 min of removal from the chamber. Specimens tested within 15 min after manufacture (impregnation) need not be stabilized.

Specimens shall be gathered into a stack for test purposes. The number of plies in the stack shall be determined from Table 4.

Close the press and allow the press platens to preheat to  $(150 \pm 2)$  °C.

**Table 4 – Number of plies per specimen as a function of glass thickness**

Glass thickness mm	Number of plies
≤0,065	10
>0,065	5

On the top of one press plate, place a release sheet, then a stack of prepreg. Use tape to hold the stack in place. Position the tape on opposing corners so that it does not interfere with the 115 mm × 150 mm working area to be tested. A second release sheet is placed on top of the stack to form a sandwich (see Figure 16). Then cover with the second press plate.

Open press and immediately place the prepreg stack and release sheets on the bottom press platen, making sure the plates are centred on the press plate.

NOTE Make sure the release material is in place.

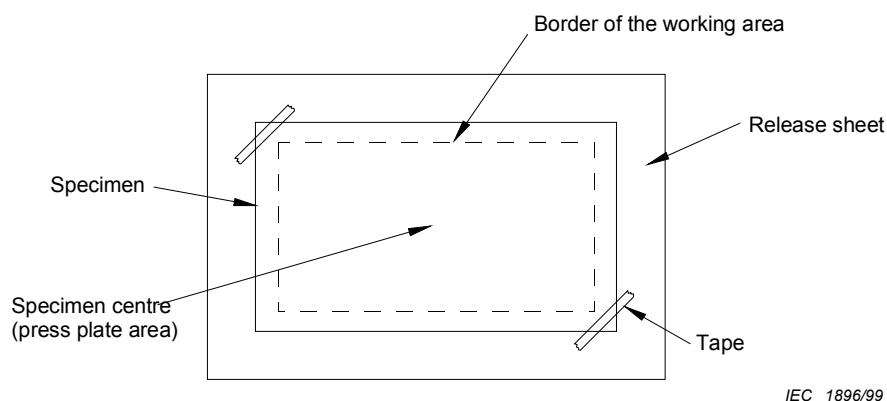
Press the specimens with a uniform pressure of  $(21 \pm 1)$  N/cm<sup>2</sup> for a minimum of 10 min. Full pressure is to be applied within 15 s after the package of prepreg and release films are placed on the press plates.

Open the press and carefully remove the laminated package, placing it onto a smooth flat surface and cool for 5 min before making measurements.

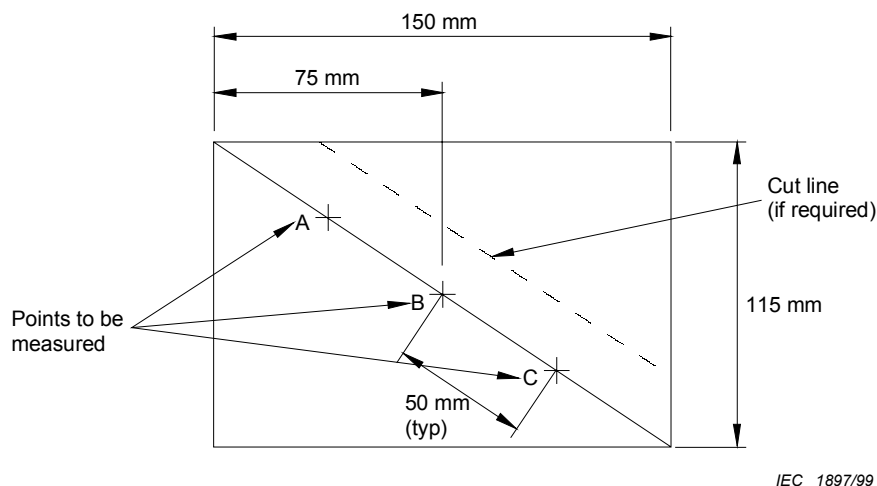
Remove release films from the laminated specimen. Using the template shown in Figure 17, mark the points to be measured. Cut the laminated specimen along the cut line shown in Figure 17, to facilitate access to measuring points.

Measure the thickness to the nearest 0,002 mm with the micrometer at the three points defined by Figure 17. Record all three measurements for each laminated specimen. Average the three measurements to determine the average measured thickness.

The thickness per ply is calculated by dividing the average measured thickness by the number of plies used in the laminated specimens.



**Figure 16 – Scaled flow test specimen before lamination**



**Figure 17 – Scaled flow test specimen measurement points**

### 9.26.5 Report

The report shall include:

- test method number and revision;
- the test date;
- the average thickness per ply of the prepreg in millimetres;
- identification and description of specimen(s);

- e) the number of plies and glass fabric style of the prepreg used for the laminated specimens;
- f) any deviation from this test method;
- g) the name of the person that conducted the test.

#### **9.26.6 Additional information**

A thickness variation between the three measurements of 0,06 mm or more is typically the result of prepreg shifting during the lamination. To verify the validity of the results, it may be necessary to repeat the test once.

### **9.27 Test 2M27: The resin flow properties of coverlay films, bonding films and adhesive cast films used in the fabrication of flexible printed boards**

#### **9.27.1 Object**

This process defines the test methods for resin flow properties of adhesive coated polyimide, or polyester (PET) films for either coverlay films, or bonding films and adhesive cast films supported on or between release materials used in the fabrication of flexible printed wiring boards.

#### **9.27.2 Test specimen**

##### **9.27.2.1 Squeeze-out of adhesive method**

From a sample of material under test, two specimens each 240 mm × 240 mm shall be cut. The specimens shall be taken from the sample sheet in such a manner that they are at least 25 mm from the edge.

##### **9.27.2.2 Filling-in clearance with adhesive method**

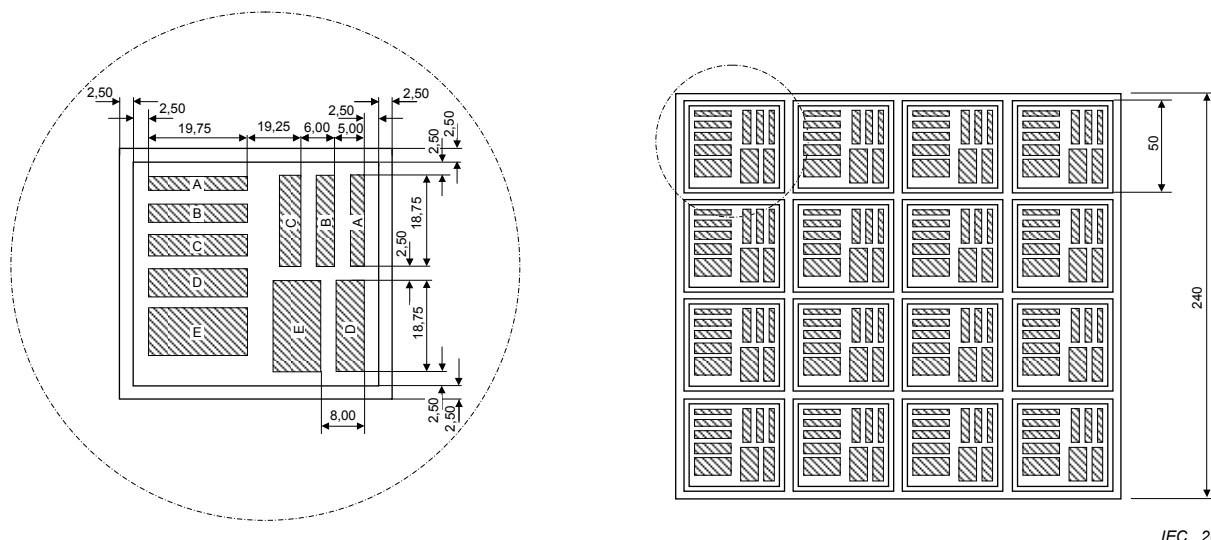
From a sample of film material under test, two specimens each 240 mm × 240 mm shall be cut. The specimens shall be taken from the sample sheet in such a manner that they are at least 25 mm from the edge.

#### **9.27.3 Test apparatus and materials**

The following test apparatus and materials shall be used:

- a) a measuring microscope or projection-type magnifier with cross-hair;
- b) copper foil of a thickness of 35 µm (305 g/m<sup>2</sup>) or 70 µm (610 g/m<sup>2</sup>), treated or untreated;
- c) a punching tool with circular punch sizes of 2 mm, 4 mm, and 6 mm;
- d) a laminating press which conforms to conditions in accordance with methods agreed upon between purchaser and supplier, or with the supplier's recommendations regarding pressure, temperature and press time;
- e) accessory materials used for lamination processes such as polypropylene film (approximately 60 µm thick), polyethylene film (approximately 100 µm thick), paper or plastic sheet (approximately 1 mm thick) for the cushion and press plates;
- f) two sheets of stainless steel press plates not less than 5 mm thick. Parallelism and roughness of the surfaces shall also be kept to produce a test specimen having thickness tolerances available for the required tests;
- g) a copper-clad laminated plastic sheet large enough to cover the specimen under test. The thickness of copper foil shall be 35 µm (305 g/m<sup>2</sup>) or 70 µm (610 g/m<sup>2</sup>) according to the requirement. The base film of the laminate shall be the same material and its thickness shall also be nearly the same as the sample under test;
- h) a set of machine tools and chemicals to be used for etching the patterns shown in Figure 18.





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Area	Conductor width mm	Clearance mm	Number of conductors
A	0,15	0,15	10
B	0,20	0,20	10
C	0,25	0,25	10
D	0,30	0,30	10
E	0,50	0,50	10

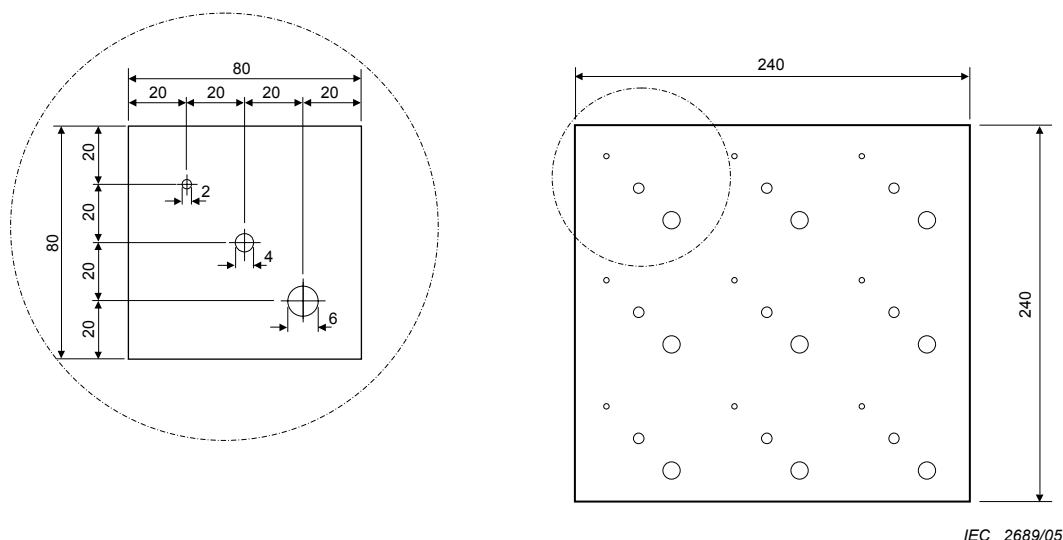
Figure 18 – Test patterns for clearance filling test

#### 9.27.4 Procedure

##### 9.27.4.1 Squeeze-out of adhesive method

This test provides a measure of the squeeze-out of adhesive around the terminal pads or soldering tabs during laminating processes. If this film is an adhesive cast, a sample of film material under test shall be transferred to a suitable polyimide film. This item effectively forms a coverlay film.

Twenty-seven holes, nine of each diameter respectively, shall be punched into the test specimens with attaching release or protective film as shown in Figure 19.



**Figure 19 – Punched test pattern for squeeze-out test**

After removal of release or protective material, the specimen shall be laid up for lamination in accordance with the method agreed upon between purchaser and supplier, or with the supplier's recommendations regarding stack, pressure, temperature and pressing time. Copper foils of a thickness of 35  $\mu\text{m}$  (305  $\text{g}/\text{m}^2$ ) shall be used and the untreated (shiny) side of the copper foil shall face the adhesive layer of the specimen.

In cases of dispute in relation to the laminating conditions, it is desirable to apply the standard procedures and conditions specified in 9.27.4.3 of this specification by agreement between customer and supplier.

After removal from the laminating press and cooling to room temperature for about 10 min at least, the maximum length of squeeze-out of the resin from the circumferences into the holes shall be measured by a measuring microscope or projection-type magnifier.

For each hole size, the average of nine measured squeeze-outs shall be calculated.

#### **9.27.4.2 Filling-in clearance with adhesive method**

This test provides a measure of compatibility of adhesive coated films to fill in clearances or gaps along the conductors and/or between the films under test and the base materials with adhesive. If this film is an adhesive cast, a sample of film material under test shall be transferred to a suitable polyimide film. This item effectively forms a coverlay film.

##### **9.27.4.2.1 Test procedure for coverlay and bonding films**

The test patterns shown in Figure 18 shall be produced by etching on the copper surface of the laminate.

After removal of release or protective materials, the specimen shall be laminated in accordance with 9.27.4.1.

The residual voids along the conductor lines and/or between the specimen and base laminate shall be inspected using the microscope or magnifier.

Copper foil to be laminated to adhesive coated film under test shall be an electrolytic foil of nominal thickness of 35  $\mu\text{m}$  (305  $\text{g}/\text{m}^2$ ).

Copper-clad flexible plastic film to be laminated to the sample under test shall have the same copper foil stated here and its base film shall have nearly the same thickness as the sample under test.

Other thicknesses and construction of both copper foil and copper-clad film may be used according to agreement between purchaser and supplier.

Cleaning procedures of copper surface which attaches to the sample under test will be provided in 9.27.4.4. Other procedures may be used according to agreement between purchaser and supplier.

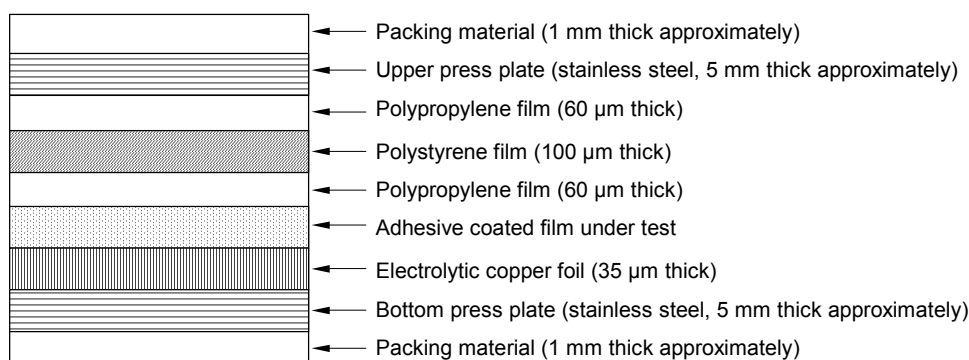
#### 9.27.4.3 Standard lamination procedure

When any inconsistency of test results is considered to be caused by the conditions for the preparation of test specimens, the referee pressing conditions specified in this subclause shall be used.

The procedures specified herein provide a standard method of lamination to produce the specimens for the tests of properties of cured coverlay films, bonding films or adhesive cast films used for the fabrication of flexible printed wiring boards.

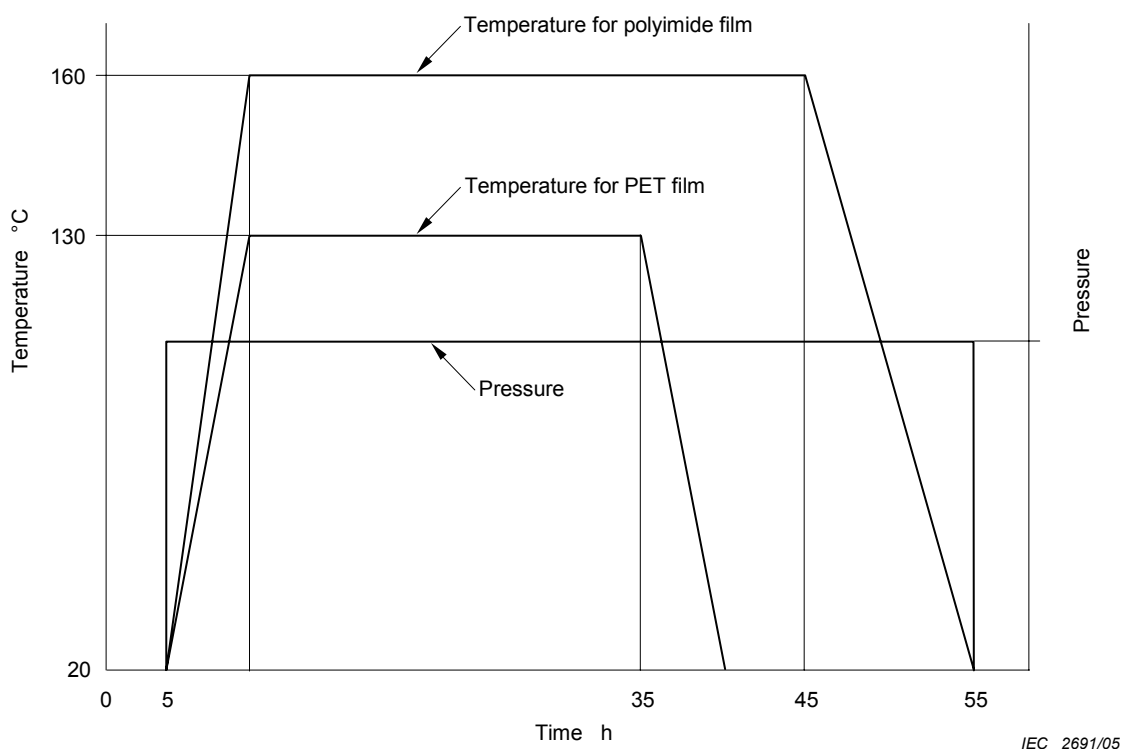
The stack of materials between the press plates to prepare the test specimen is shown in Figure 20. The surface of copper foil shall face the adhesive layer of the sample under test.

The profiles of parameters of press condition for the referee test laminates are shown in Figure 21.



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**Figure 20 – Standard lay-up of materials in the press to prepare the test specimens for referee tests**



**Figure 21 – Profiles of press conditions to prepare the test specimens for referee tests**

#### 9.27.4.3.1 Press plates

The press plates shall have enough area to produce a laminate of at least 250 mm × 250 mm after trimming. Parallelism of the plates shall be kept to produce a test specimen having tolerances of thickness available for the required tests.

#### 9.27.4.3.2 Laminating press

The temperature of the press shall be raised to 180 °C within nearly 10 min and the temperature of the plates can be maintained within ±3 °C of working temperature.

The press shall be capable of maintaining a pressure of 6 MPa on the specimen and the deviation of pressure caused by flowing of adhesive shall be kept within ±10 % of set values.

#### 9.27.4.4 Standard cleaning procedures of copper surfaces

When any inconsistency of test results is considered to be caused by any contamination on the copper surfaces used for the production of test laminate, the cleaning procedures stated in Table 5 shall be used.

**Table 5 – Cleaning procedures**

Step	Process	Chemicals	Temperature	Time s
1	Dip	Hydrochloric acid (1 020 ± 10) kg/m <sup>3</sup>	Room temperature	120 ± 5
2	Rinse	Clean running water	Room temperature	120 <sup>+60</sup> <sub>0</sub>
3	Dip	Ammonium persulphate (20 ± 0,5) %	Room temperature	120 ± 5
4	Rinse	Clean running water	Room temperature	120 <sup>+60</sup> <sub>0</sub>
5	Dip	Sulphuric acid (1 080 ± 10) kg/m <sup>3</sup>	Room temperature	60 ± 5
6	Rinse	Clean running water	Room temperature	120 <sup>+60</sup> <sub>0</sub>
7	Rinse (3 times)	Deionized water Use fresh each time	Room temperature	240 ± 60 (each step)
8	Dry	Wipe water off or dry in air	Room temperature	180 ± 60
9	Dip	Ethyl alcohol	Room temperature	60 ± 5
10	Dry	Hot air oven	(80 ± 2) °C	300 ± 60
11	Lamination shall be performed within 30 min after drying.			

### 9.27.5 Report

The report shall include:

- the test number and revision index;
- the test date;
- the identification of the material tested;
- the test method used;
- the average of nine measured squeeze-out values for each hole size given in millimetres;
- the details of press conditions;
- the numbers and detailed locations of voids and/or inclusions on the effective areas of the test specimen.

## 10 Electrical test methods

### 10.1 Test 2E01: Surface tracking, moisture condition (under consideration)

### 10.2 Test 10.2 2E02: Dielectric breakdown of base materials parallel to laminations

#### 10.2.1 Object

This test method covers the procedure for determination of the electrical strength of base materials parallel to laminations of the sheet being under test by applying an alternative high voltage increasing step by step on specimens immersed into transformer oil.

#### 10.2.2 Test specimens

Test specimens shall be according to Clause 4 of IEC 60243-1:1998, but with the following changes or additions:

- a) The test specimens shall be approximately 75 mm × 50 mm and of the same thickness as the material to be tested. They shall be prepared from a sample of the copper-clad base material under test by shearing or sawing. Two holes, 5 mm in diameter, shall be drilled along the longitudinal centre line and midway between the etches in the 50 mm dimension, with a spacing of 25 mm.
- b) After complete removal of the copper cladding by any industry acceptable practice, the specimens shall be conditioned as specified in the relevant specification. Unless otherwise specified, the specimens shall be conditioned for 48 h in distilled water of ambient temperature ( $23^{+2}_{-5}$ ) °C. Before testing, the specimens shall be wiped dry with a lint-free absorbent paper towel and stored for 30 min at ambient temperature.
- c) Four specimens shall be tested. Two specimens shall be cut in the machine direction and two in the transverse direction of the reinforcement.

### 10.2.3 Test apparatus and material

The following test apparatus and materials shall be used:

- a) electrical equipment according to Clause 7 of IEC 60243-1:1998, but with the following changes or additions:
  - a high voltage breakdown AC-tester (50-60 Hz) of 50 kV and of 5 kV above 10 kV voltage and with a motorized control capable of a rate of voltage increase of 1 kV/s;
  - suitable high voltage test leads;
  - a fixture containing two slightly tapered pin electrodes with spherical ends of 5 mm diameter and a centre distance of 25 mm;
- b) oil tank of sufficient size to take in the fixture, including the specimen and to ensure completely immersion of the specimen into the fluid; the tank shall be filled with transformer oil;
- c) a beaker or pan to be filled with water for conditioning of the specimens;
- d) racks for supporting specimens in the water bath;
- e) a timer 0–60 s;
- f) lintless towels of absorbent paper.

### 10.2.4 Procedure

The procedure shall be carried out according to Clause 8 of IEC 60243-1:1998, but with the following changes or additions:

- a) the first specimen shall be inserted into the fixture and immersed in oil;
- b) the electrical breakdown shall be determined by applying the high voltage, starting at 0 V, with a motorized control capable a rate of voltage increase of 1 kV/s;
- c) the breakdown voltage shall be recorded. If the breakdown occurs within the oil and no damaging of the specimen is obvious, the specimen may be tested again. If the breakdown occurs within the oil again, the oil should be filtered or replaced.

### 10.2.5 Report

The report shall include:

- a) the test number and revision;
- b) the date of the test;
- c) the identification and description of the material tested;
- d) the number and size of the specimens;

- e) the average value of the dielectric breakdown voltage of the step-by step method; rounded to the nearest kV; if the accuracy of the meter is not within 5 % for all obtained values, a correction of the readings with lower accuracy shall apply. The average value shall be reported with a "+" after the value, if there is one or two specimens pass the test without breakdown;
- f) the minimum value at which the oil broke down, if no specimen had a breakdown;
- g) any deviation from this test method;
- h) the name of the person conducting the test.

### 10.2.6 Additional information

- a) Use sharp, high-speed drills to prevent burning of the specimens or producing rough holes. These details may adversely affect the dielectric breakdown.
- b) The high voltage tester shall be installed and operated in accordance with the manufacturer's instructions and extreme care shall be used in performing the test. For criteria of breakdown, see Clause 10, IEC 60243-1:1998.

## 10.3 Test 2E03: Surface resistance after damp heat, steady state

### 10.3.1 Object

This test method covers the procedure for the determination of the surface resistance of the dielectric base material after storage at high humidity and recovery. The surface resistance is specified as the resistance between two electrodes of defined dimensions arranged on the surface of the dielectric base material.

### 10.3.2 Test specimens

- a) Specimens shall be cut not less than 25 mm from the edge of the sheet.
- b) Specimens shall be prepared from a sample of the base material under test.
- c) A minimum of three specimens shall be tested.

#### 10.3.2.1 Laminate thickness greater than or equal to 0,5 mm

Three specimens of dimensions  $(100 \pm 5)$  mm  $\times$   $(100 \pm 5)$  mm with the specified thickness shall be prepared for each test condition, unless otherwise specified.

#### 10.3.2.2 Laminate thickness less than 0,5 mm

Three specimens of dimensions  $(50 \pm 2)$  mm  $\times$   $(50 \pm 2)$  mm with the specified thickness shall be prepared for each test condition, unless otherwise specified.

### 10.3.3 Apparatus and materials

The following test apparatus and materials shall be used:

- a) Conditioning test chamber capable of programming and recording an environment of temperature ranging between  $(25 \pm 2)$  °C and  $(65 \pm 2)$  °C, and 85 % to 93 % RH.
- b) Air circulating oven capable of maintaining the specified test temperatures to within  $\pm 2$  °C.
- c) Resistance measuring instrumentation capable of measuring to  $10^{12}$  M $\Omega$ , minimum, with an accuracy of  $\pm 5$  % at its highest scale setting. The equipment shall have the capability of applying 500 V d.c. to the test specimen.

- d) A system/fixture for electrical connections into the temperature and humidity chambers. Three separate cables shall be provided to make connections to each specimen being conditioned/tested. The centre conductor of each cable shall be connected to one of the three electrodes applied to the test specimen. The opposite ends of the cables shall be brought outside the chamber and terminated at a convenient location for connection to the measuring instrument. Shields shall be trimmed back from the ends of the centre conductor insulation and interconnected to the guard post of the measuring instrument. The system of electrical connections to the specimens may benefit from a coaxial cable set-up designed to shield the measurement of volume or surface resistances from electrical interference.

Support the specimen parallel to the air flow through the chamber during conditioning.

Special care shall be taken to ensure that materials used in the fixture are such that resistance readings are that of the material being tested and not the fixture.

- e) A measurement device capable of measuring laminate thickness to the nearest 0,0025 mm.  
f) Material and apparatus for formation of specimen conductors.  
g) Conductive silver paint.  
h) A system for applying the paint to the specimen, such as silk screening.  
i) A mask, fixture, photoprinting system, or equivalent, for applying the applicable electrodes/ test pattern to the specimen (See Table 6).

**Table 6 – Specimen dimension (cm)**

Base thickness	$D_1$ diameter	$D_2$ diameter	$D_3$ diameter	$D_4$ diameter	$D_5$ diameter	A cm <sup>2</sup>	$P/D_4$
Less than 0,50 mm	1,000 ± 0,005	1,020 ± 0,005	1,375 ± 0,005	0,010 ± 0,001	0,177 ± 0,005	5,169	317,4
0,50 mm and greater	2,000 ± 0,015	2,500 ± 0,015	2,000 ± 0,015	0,250 ± 0,005	0,250 ± 0,015	25,652	28,27

For the above:

$$D_0 = (D_1 + D_2)$$

$$A = \frac{\pi D_0^2}{4}$$

$$P = \pi D_0$$

### 10.3.4 Procedure

#### 10.3.4.1 Preparation of specimens

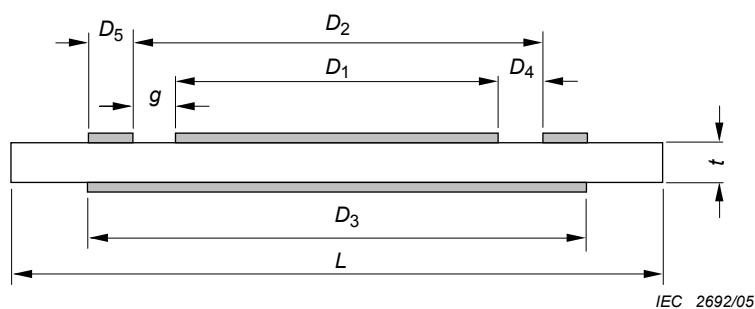
Prepare test patterns with the applicable dimensions shown in Table 6, and in accordance with Figures 22a, 22b and 22c. The specimens shall be generated as follows:

- a) Metal-clad materials having a thickness of less than 0,5 mm shall be photoprinted with a pattern of the outline of the conductors and etched using an appropriate etching solution. The electrodes shall then be completed (filled in) using silver conductive paint.
- b) Metal-clad laminates 0,5 mm or thicker shall be etched with an appropriate etching solution. A pattern of the outline of the conductors may be photoprinted before etching. Test electrodes shall be applied using silver conductive paint and an appropriate test pattern application system.
- c) Solid metal foil electrodes shall not be acceptable in any case, except for the outer electrode for laminates less than 0,5 mm. A small pad of retained cladding may be retained within the electrode borders to facilitate soldering of leads.

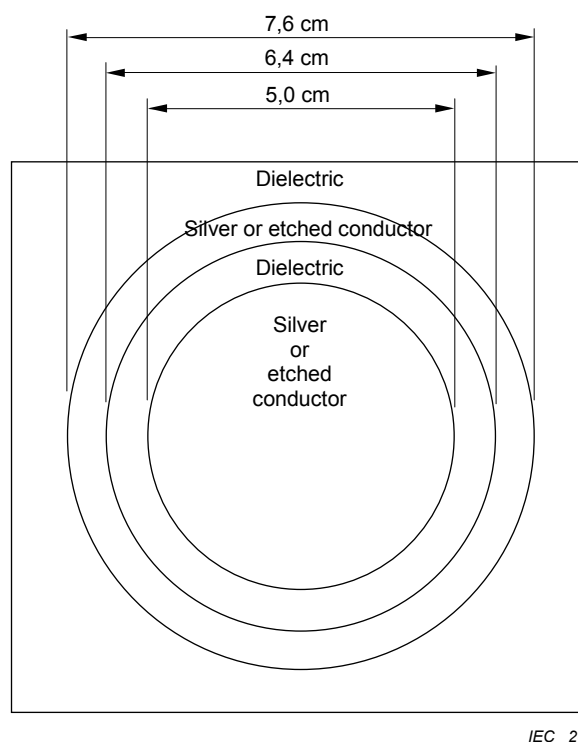


- d) If soldering techniques are used to attach leads, suitable cleaning procedures shall be used to remove flux and other residue.

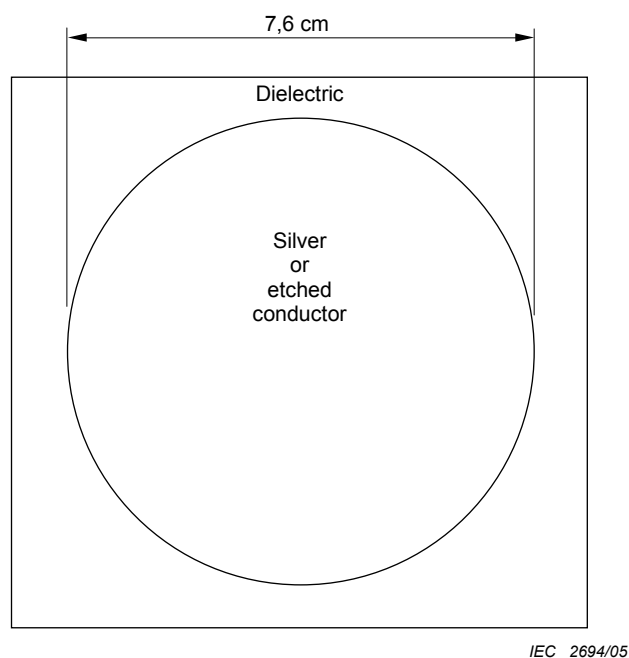
See IEC 60068-2-78 for general description of the damp-heat test.



**Figure 22a – Test pattern dimensions (see Table 6)**



**Figure 22b – Volume and surface resistivity test pattern (side 1)**



**Figure 22c – Volume and surface resistivity test pattern (side 2)**

#### 10.3.4.2 Conditioning

##### a) Humidity conditioning

Specimens shall be subjected to  $(90^{+5}_0)$  % RH and  $(35 \pm 2)^\circ\text{C}$  for a period of  $(96^{+2}_0)$  h prior to electrical measurement.

Specimens of a thickness greater than or equal to 0,5 mm shall be prepared by soldering single stranded (to decrease the opportunity for flux contamination from the wire) insulated wire which is not affected by the test environment to each of the connection points of the test specimens. These wires will be used to connect the test patterns of the test specimens to the power supply and for insulation resistance testing.

Following the tenth cycle, the conditioning chamber shall maintain a temperature of  $(25 \pm 2)^\circ\text{C}$  and  $(90^{+5}_0)$  % RH during the interval that electrical measurements are being made.

##### b) At elevated temperature conditioning

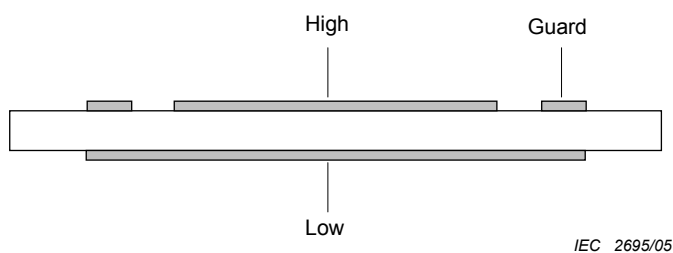
Specimens shall be subjected to the specified temperature (based on material type and specification requirements) for a period of  $(24^{+2}_0)$  h. Unless otherwise specified, the temperature shall be  $(125 \pm 2)^\circ\text{C}$ .

#### 10.3.4.3 Electrical measurements

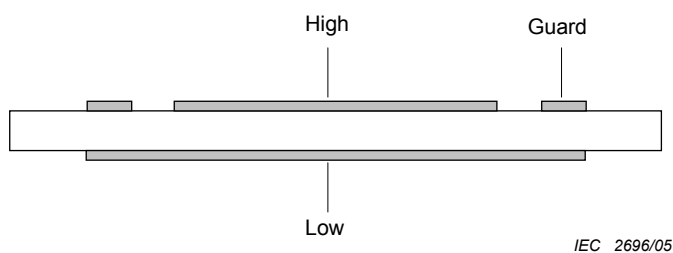
All electrical measurements shall be made inside the applicable conditioning chamber and at the conditions specified in 10.3.4.2.

- a) Electrical measurements taken on specimens conditioned in accordance with 10.3.4.2 a) shall be completed within the tolerance of the conditioning.
- b) Electrical measurements taken on specimens conditioned in accordance with 10.3.4.2 b) shall be made after achieving 1,5 h steady state of  $(25 \pm 2)^\circ\text{C}$  and  $(90^{+5}_0)$  % RH. All measurements shall be completed within 2 h.

- c) Electrical measurements taken on specimens conditioned in accordance with 10.3.4.3 shall be completed within the tolerance of the conditioning.
- d) All electrical measurements shall be made using 500 V direct current. The voltage shall be applied to the specimen for  $(60 + \frac{5}{0})$  s prior to taking the actual reading for stabilization purposes.
- e) Measure the volume resistance by connecting the resistance measuring device to the specimen electrodes through the fixture system as described in 10.3.3 d) in accordance with Figure 22.
- f) Measure the surface resistance by interchanging the test cables connecting the solid back electrode and the outer ring to the instrument for the arrangement shown in Figure 23.



**Figure 22 – Measurement of volume resistance**



**Figure 23 – Measurement of surface resistance**

#### 10.3.4.4 Specimen thickness

Each specimen shall be measured for its thickness without cladding. Specimens for each test condition shall have their thickness readings averaged.

#### 10.3.4.5 Calculations

The surface resistivity shall be calculated as follows:

$$r_1 = \frac{R_1 P}{D_4}$$

where

$r_1$  is the surface resistivity in  $M\Omega$ ;

$R_1$  is the measured surface resistance in  $M\Omega$ ;

$P$  is the effective perimeter of the guarded electrode in cm;

$D_4$  is the width of the test gap in cm.

NOTE The ratio of  $P/D_4$  for the electrode configuration being used may be obtained from the dimension table included in Table 6.

#### 10.3.4.6 Reporting

- The volume resistivity of each specimen and the average shall be reported. Each condition tested shall be reported separately.
- The surface resistivity of each specimen and the average shall be reported. Each condition shall be reported separately. The surface resistance is the direct reading of the megohmmeter scale and should be recorded in megohms.

#### 10.3.5 Report

The report shall include:

- the test number and revision;
- the identification and description of the material tested;
- the surface resistance while in the humidity chamber (optional);
- the surface resistance after recovery;
- the date of the test;
- any deviation from the test method;
- the name of the person conducting the test.

### 10.4 Test 2E04: Volume resistivity and surface resistivity

#### 10.4.1 Object

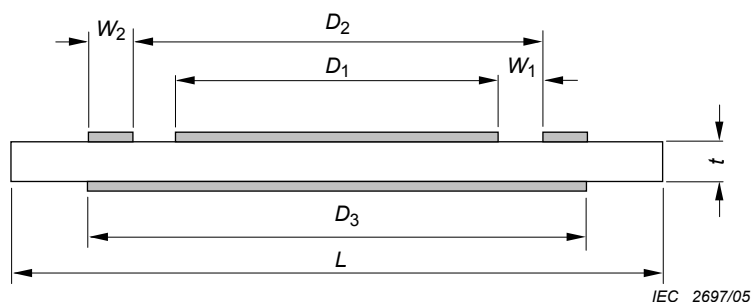
These test methods are designed to determine both volume and surface resistivity of base materials under standard atmospheric conditions for measurements and tests, after damp heat and recovery and at elevated temperature. These methods are not applicable to unclad laminates with a base thickness of 0,50 mm or thinner.

#### 10.4.2 Test specimen

- Foil clad laminates with a base thickness of 0,50 mm or less shall utilize a ring and disk pattern in each of three specimens 50 mm × 50 mm.
- Laminates with a base thickness of 0,50 mm or greater shall utilize three specimens 100 mm × 100 mm. A set of three specimens shall be prepared separately for each test condition of high humidity and elevated temperature.

##### 10.4.2.1 Electrodes

Test patterns shown in Figure 24 with dimensions shown in Table 7 shall be generated by either photo-etching, or by brushing/spraying with conductive silver paint using a suitable dimensioned mask.



IEC 2697/05

Figure 24 – Electrode dimensions for volume resistivity and surface resistivity test

Cleaning of specimens after etching is mandatory.

**Table 7 – Test pattern dimensions**

Thickness of base material	$D_1$ Diameter mm	$D_2$ Diameter mm	$D_3$ Diameter mm	$W_1$ Gap mm	$W_2$ Ring width mm	$P/W_1$
Less than 0,50 mm	24,50 ± 0,3	25,91 ± 0,13	34,93 ± 0,13	0,25 ± 0,03	0,460 ± 0,13	317,4
0,50 mm and thicker	50,80 ± 0,38	63,50 ± 0,38	75,20 ± 0,38	6,36 ± 0,13	6,36 ± 0,38	28,27
<p>NOTE <math>P</math> is the effective perimeter of the guarded electrode in millimetres.</p> $P = \pi \times \frac{D_1 + D_2}{2}$ <p>where  <math>\pi</math> is the circular constant rounded to 3,1416.</p>						

#### 10.4.2.2 Thickness

The thickness  $t$  of the specimen shall be determined by measuring with a micrometer in at least three places outside the test electrodes area to an accuracy of  $\pm 0,0025$  mm. The average of these measurements shall be rounded off to the nearest 0,0025 mm for laminate less than 0,50 mm, and to the nearest 0,025 mm for laminate 0,50 mm and thicker. This average shall be used for the calculation of volume resistivity.

#### 10.4.3 Test apparatus and materials

##### 10.4.3.1 Conditioning of chambers

The following test chambers shall be used:

- a test chamber capable of maintaining a combination of  $(40 \pm 1) ^\circ\text{C}$  and  $(93^{+2}_{-3})$  % relative humidity;
- a test chamber capable of maintaining a temperature of  $80 ^\circ\text{C}$  to  $220 ^\circ\text{C}$  within a tolerance of  $\pm 2 ^\circ\text{C}$ .

All test chambers shall be fitted with a coaxial cable system designed to shield the measurement of volume or surface resistivities from stray interferences. When measuring at damp heat while in humidity chamber or at elevated temperature, three separate cables shall be provided to each specimen being conditioned. The opposite ends of the cables shall be brought outside the chamber and terminated at a convenient location for connection to the measuring instrument.

When conditioning specimens, support specimens parallel to the air flow through the chamber during the conditioning period

##### 10.4.3.2 Micrometer

A hand micrometer, capable of resolving to 0,0025 mm or better shall be used.

##### 10.4.3.3 Resistance measuring device

A device with an accuracy of  $\pm 5$  % throughout the test range shall be used. The equipment shall have the capability of applying 500 V d.c. to the test specimen and be capable of accommodating the shielded cable system described in 10.4.3.1.

#### 10.4.3.4 Other equipment

The following other materials shall be used:

- a) conductive silver paint having a volume resistivity of  $10^{-10} \text{ M}\Omega \times \text{m}$  or less when cured;
- b) hand brushes for painting or air brush for spraying on electrodes;
- c) suitable spray masks for producing the back electrode on single-sided clad laminates, ( $D_3$  in Figure 24).

#### 10.4.4 Procedure

##### 10.4.4.1 Conditioning

###### 10.4.4.1.1 Preconditioning

Specimens shall be preconditioned for  $(96^{+2}_0) \text{ h}$  at  $(20^{+5}_0) ^\circ\text{C}$  and  $(65 \pm 5) \%$  relative humidity. This preconditioning may be expressed as "C-96/20/65".

###### 10.4.4.1.2 Damp heat

Specimens shall be preconditioned for  $(96^{+2}_0) \text{ h}$  at  $(40 \pm 1) ^\circ\text{C}$  and  $(93^{+2}_{-3}) \%$  relative humidity. This conditioning may be expressed as "C-96/40/93".

###### 10.4.4.1.3 Recovery

Specimens shall be preconditioned for  $(90 \pm 15) \text{ min}$  at  $(23 \pm 5) ^\circ\text{C}$  and  $(75 \pm 2) \%$  relative humidity for recovery. This conditioning may be expressed as "C-1,5/23/75".

###### 10.4.4.1.4 Elevated temperature

Specimens shall be conditioned at an elevated temperature i.e.  $(100 \pm 2) ^\circ\text{C}$ ,  $(125 \pm 2) ^\circ\text{C}$ ,  $(170 \pm 2) ^\circ\text{C}$  or  $(200 \pm 2) ^\circ\text{C}$  specified in the relevant specification for 1 h. This conditioning may be expressed as "E-1/100, E-1/125, E-1/170 or E-1/200", respectively.

##### 10.4.4.2 Test method 1: Measuring after damp heat and recovery

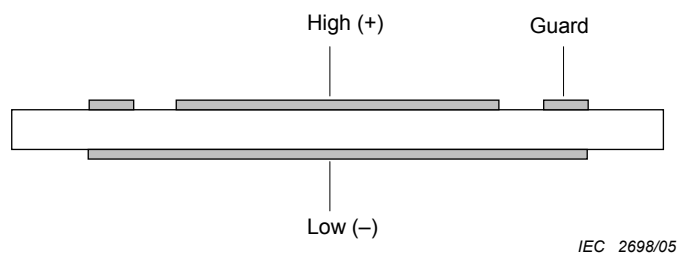
Measurements of volume resistivity and/or surface resistivity shall be made at room temperature after damp heat and recovery, as specified in 10.4.4.1.2 and 10.4.4.1.3, respectively.

These measurements shall commence within the time tolerance of 0 h to 2 h and completed within 3 h.

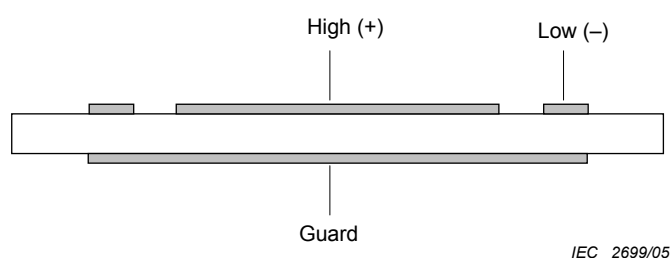
Determination of volume resistivity or surface resistivity shall be made when 500 V d.c. has been applied to the specimens for  $(60^{+5}_0) \text{ s}$ .

Volume resistivity shall be measured by connecting the resistance measuring device to the specimen electrodes through the shielded cable system described in 10.4.3.1. Connect as shown in Figure 25.

The surface resistance of the same specimens shall be measured by interchanging the test cables connecting the solid back electrode and the outer ring to the instrument from the arrangement shown in Figure 25 to the arrangement shown in Figure 26.



**Figure 25 – Electrode connections for measuring volume resistance**



**Figure 26 – Electrode connections for measuring surface resistance**

#### 10.4.4.3 Test method 2: Measuring after damp heat while in the humidity chamber

Measurements of volume resistivity and/or surface resistivity shall be made with specimens inside the humidity chamber as specified in 10.4.4.1.2. The subsequent test procedure and its notes are just the same as for 10.4.4.2.

#### 10.4.4.4 Test method 3: Measuring at elevated temperature

Measurements of volume resistivity and/or surface resistivity shall be made with specimens inside the conditioning chamber as specified in 10.4.4.1.4. The subsequent test procedure and its notes are just the same as for 10.4.4.2.

#### 10.4.4.5 Calculations

Calculation of volume resistivity:

$$\rho_v = \frac{\pi (D_1 + D_2)^2}{16 t} \times R_v$$

where

- $\rho_v$  is the volume resistivity in megohm metres ( $M\Omega \times cm$ );
- $D_1$  is the inner circle diameter of surface electrode in centimetres (cm);
- $D_2$  is the inside diameter of outside ring electrode in centimetres (cm);
- $t$  is the average thickness of specimen in centimetres (cm) excluding metal foil and/or conductive silver paint layer thickness (cm);
- $R_v$  is the measured volume resistance in megohms ( $M\Omega$ );
- $\pi$  is the circular constant rounded to 3,1416.

Calculation of surface resistance:

$$\rho_s = \frac{P}{W_1} \times R_s$$

where

$\rho_s$  is the surface resistivity in megohms (M $\Omega$ );

$R_s$  is the measured surface resistance in megohms (M $\Omega$ );

$W_1$  is the width of the test gap in centimetres (cm);

$P$  is the effective perimeter of the guarded electrode in centimetres (cm).

$$P = \frac{\pi (D_1 + D_2)}{2}$$

where

$D_1$  is the inner circle diameter of surface electrode in centimetres (cm);

$D_2$  is the inside diameter of outside ring electrode in centimetres (cm);

$P$  is the effective perimeter of the guarded electrode in centimetres (cm);

$\pi$  is the circular constant, rounded to 3,1416.

#### 10.4.5 Report

The report shall include:

- a) the test method number and revision index;
- b) the date of the analysis;
- c) the identification of materials tested;
- d) condition utilized;
- e) average and minimum of volume resistivity measured;
- f) average and minimum of surface resistivity measured;
- g) any deviation from this test method.

#### 10.4.6 Additional information

None.

#### 10.5 Test 2E05: Permittivity and dielectric dissipation (under consideration)

#### 10.6 Test 2E06: Volume and surface resistivity, 3 electrodes (under consideration)

#### 10.7 Test 2E07: Surface and volume resistivity, elevated temperature (under consideration)

#### 10.8 Test 2E08: Surface corrosion

##### 10.8.1 Object

To determine for base materials any tendency for development of electrolytic corrosion products which occur when an etched conductive pattern is subjected to a polarizing voltage and high humidity.



### 10.8.2 Test specimens

Four specimens, 100 mm × 100 mm, shall be taken from the panel or sheet at least 25 mm from the edge. The specimens shall be printed, on one side, with the ring and disk pattern of Figure 27, or with the comb pattern in Figure 28, by any appropriate method of commercial practice.

### 10.8.3 Test apparatus and materials

- a test chamber as specified in IEC 61189-3 test method 3N06;
- a power supply capable of producing a standing bias potential of  $(100 \pm 5)$  V d.c.;
- a limiting resistor, 100 k $\Omega$ , to limit the current to 1 mA;
- wires, polytetrafluoroethylene-insulated, to connect the specimens to the power supply;
- a fixture with spring loaded contacts, which may be used instead of wire terminations.

### 10.8.4 Procedure

- The specimens shall be connected as shown in Figure 27 or Figure 28. The specimens shall be placed in the chamber in a vertical position, and the termination wires of the specimens connected to the power supply.
- Environmental conditions shall be in accordance with IEC 61189-3 test method 3N06. The duration shall be 21 days.
- The specimens shall be polarized with 100 V d.c. and the current limited to 1 mA.
- After environmental conditioning, the specimens shall be removed from the chamber and examined for corrosion.
- Visual examination may be conducted at a magnification up to 3 $\times$ .

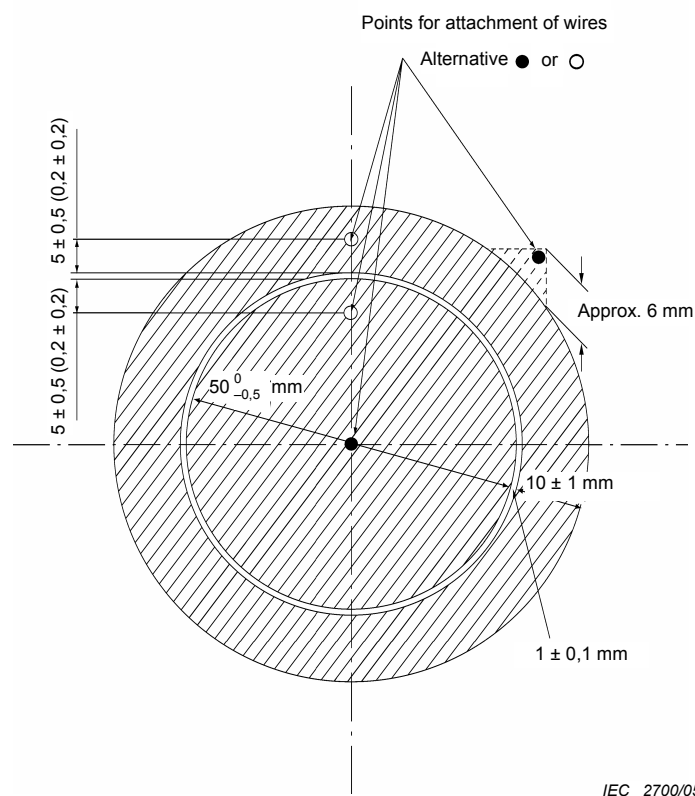
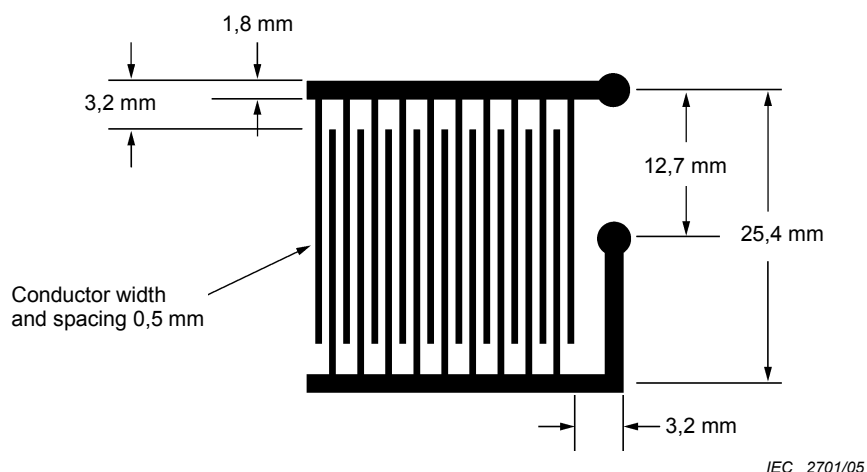


Figure 27 – Ring and disk pattern



**Figure 28 – Comb pattern**

### 10.8.5 Report

The report shall include:

- the test method number and revision;
- the type of pattern used;
- the date of test;
- identification and description of the test material;
- whether corrosion products have been observed or not;
- any deviation from this test method;
- the name of the person conducting the test.

### 10.8.6 Additional information

During this test, handling of the specimen shall be kept to a minimum to avoid contamination. The specimen shall be held by the edges, and rubber or polyethylene gloves shall be worn. When connecting the wires, care shall be taken to prevent contamination of the surface of the material by solder flux. The use of solvents to remove contaminants may result in spreading the contaminants over the whole surface.

## 10.9 Test 2E09: Comparative tracking index (CTI)

### 10.9.1 Object

This method of test indicates the relative resistance of a laminate base material to tracking for voltages up to 600 V when the surface is exposed under electric stress to water with the addition of contaminants.

NOTE The test results as such cannot be used directly for the evaluation of safe creepage distances when designing electrical apparatus.

### 10.9.2 Test specimens

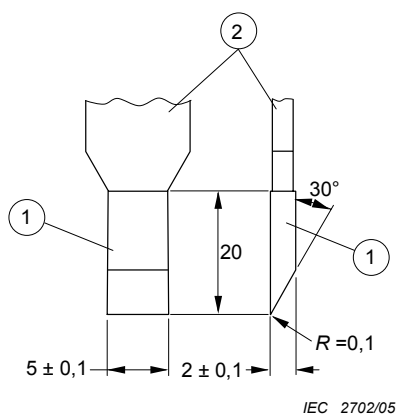
- Specimens shall be cut not less than 25 mm from the edge of the sheet. The thickness of the specimen shall be 1,6 mm and over. Specimens shall be removed from the sheet by sawing, routing, shearing or equivalent method. Copper clad specimens shall have copper removed by etching. The size of each test specimen shall be 20 mm × 20 mm. Five specimens shall be utilized.

- b) The surface of the test specimens shall be clean and free of dust, dirt, fingerprints or other contaminants which may influence the test results. The surface of the test specimen shall be cleaned by gauze with ethanol. Specimens shall be preconditioned at  $(20 \pm 2) ^\circ\text{C}$  and  $(60\sim 70) \% \text{ RH}$  for  $(96 \pm 4) \text{ h}$ .

### 10.9.3 Test apparatus and materials

#### 10.9.3.1 Electrodes

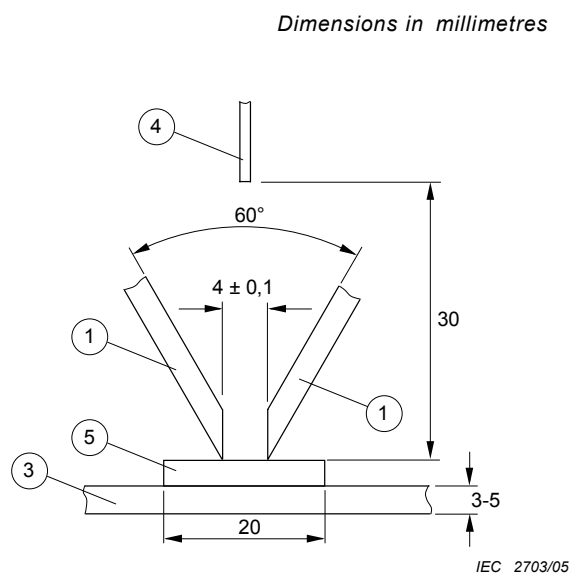
- a) The two platinum electrodes shall have a rectangular cross-section of  $5 \text{ mm} \times 2 \text{ mm}$ , with one end chisel-edged with an angle of  $30^\circ$  against the longitudinal axis (Figure 29). The chisel-edge shall be slightly rounded.
- b) The electrodes shall be symmetrically arranged on a vertical plane, the total angle between them being  $60^\circ$ , and with opposing electrode faces vertical and at a distance of  $(4,0 \pm 0,1) \text{ mm}$  apart on a flat horizontal surface of the specimen (Figure 30). The force exerted by each electrode on the surface shall be  $(1 \pm 0,05) \text{ N}$ . An arrangement for applying the electrodes to the specimen is shown in Figure 31.



#### Components

- 1 platinum electrode  
2 brass extension

**Figure 29 – Electrode**



- 3 support (glass)  
4 tip of dropping device  
5 specimen

**Figure 30 – Electrode arrangement**

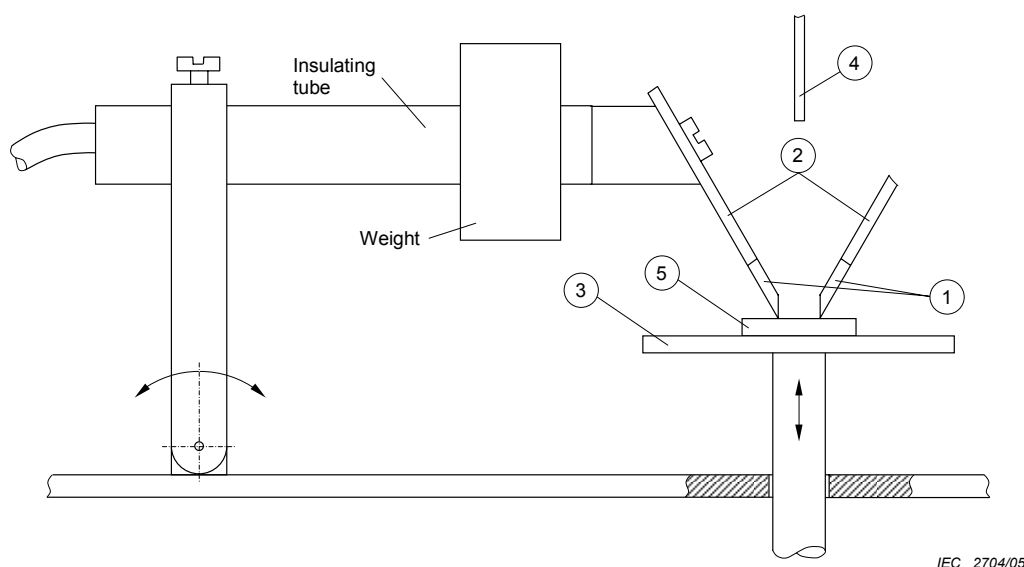


Figure 31 – Example of test apparatus

#### 10.9.3.2 Test circuit

- The electrodes shall be supplied with a substantially sinusoidal voltage, variable between 100 V and 600 V at a frequency of 48 Hz to 60 Hz. The power of the source should not be less than 0,5 kVA. The basic circuit is shown Figure 32.
- The variable resistor shall be capable of adjusting the current between the short-circuited electrodes to  $(1,0 \pm 0,1)$  A and the voltage indicated by the voltmeter shall fall by more than 10 % when this current flows.
- An over-current relay in the test circuit shall trip when 0,5 A or more has persisted for 2 s.

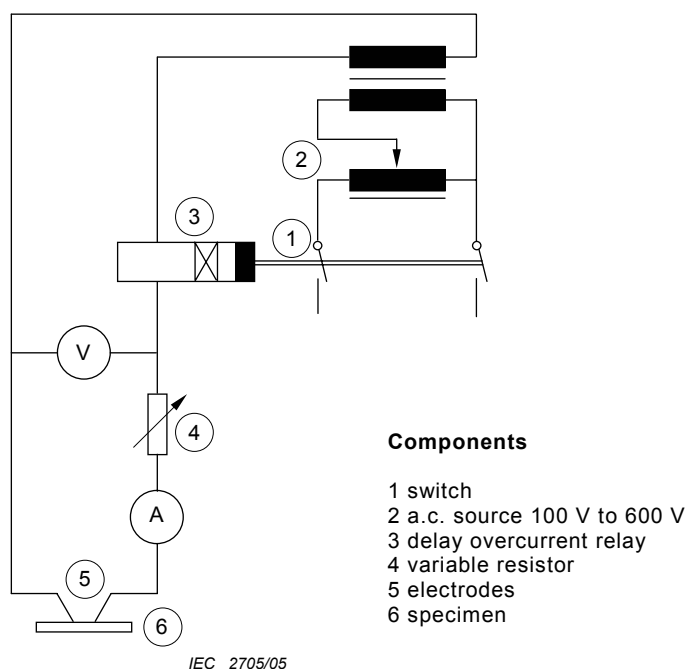


Figure 32 – Example of test circuit

### 10.9.3.3 Dropping device

The surface between the electrodes shall be wetted with drops of the test solution at intervals of  $(30 \pm 2)$  s. The drops shall fall centrally between the electrodes from a height of 30 mm. The size of the drops shall be  $(20^{+3}_0)$  mm<sup>3</sup>. Prior to each test, the needle or other outlet for the drops shall be cleaned and sufficient drops let out to ensure that the correct concentration of the test solution is used

NOTE 1 When the test solution is left in the needle between tests, evaporation will increase its concentration. Letting out about 5 to 20 drops depending upon the delay between tests, will normally remove any liquid with too high a concentration.

NOTE 2 To determine the drop size check that 1 cm<sup>3</sup> of liquid is delivered by not less than 45 and not more than 50 drops. The drop size shall be checked periodically.

NOTE 3 A hypodermic needle, having an outer diameter of 0,9 mm to 1,1 mm with the tip cut off square, is suitable for a dropping device.

### 10.9.3.4 Test solutions

- a) Solution:  $(0,1 \pm 0,002)$  % by mass ammonium chloride (NH<sub>4</sub>Cl) in deionized water.
- b) The resistivity at  $(23 \pm 1)$  °C is  $(395 \pm 5)$  Ω cm.

### 10.9.4 Procedure

- a) The test shall be carried out with the specimen screened from draughts and at an ambient temperature of  $(20 \pm 5)$  °C. Contamination of the electrodes may have an influence on the test results. The electrodes shall be cleaned before every test.
- b) The specimen to be tested shall be placed, with the test surface horizontal, on a glass support so that the chisel edges of both electrodes are pressed against the specimen with the specified force.
- c) The distance between the electrodes shall be checked and they shall make good contact with the specimen. If the electrode edges have become eroded, the edges shall be reshaped. The voltage is set at some suitable value, divisible by 50, and the circuit resistance is adjusted so that the short-circuit current is within the given tolerance. The drops of electrolyte are then allowed to fall on the test surface until failure by tracking occurs or 50 drops fall.
- d) A failure has occurred if a current of 0,5 A or more flows for at least 2 s in a conducting path between the electrodes on the surface of the specimen, thus operating the overcurrent relay; or if the specimen burns without releasing the overcurrent relay.

NOTE 1 If several tests are carried out on the same test specimen, care must be taken that the testing points are sufficiently far from each other so that splashes from the testing point will not contaminate the other areas to be tested.

NOTE 2 Since the test may produce poisonous or noxious gases, it is advisable to provide means for their safe removal or containment.

### 10.9.5 Determination of the CTI

Set the voltage at a selected level and carry out the test for 50 drops or until previous failure occurs. Repeat the test on other test sites at lower or higher voltages until the maximum voltage is established at which no failure occurs at 50 drops in tests on five sites. The numerical value of this voltage is the CTI (e.g. CTI 400).

NOTE 1 If the behaviour of the material is unknown, the initial test voltage should be in the middle of the test range, e.g. 300 V. This voltage should be increased if a specimen withstands 50 drops or decreased if it fails before 50 drops. The voltage change should be 50 V or a multiple of 50 V. The process is continued until the highest voltage at which five specimens can withstand 50 drops is found.

NOTE 2 Warning: At higher voltages and more than 50 drops, a failure (indicated by the trip of the overcurrent relay) may occur by accumulation of solution and contaminants in grooves and holes on the surface. In this case, new tests are necessary or this should be reported.

### 10.9.6 Report

The report shall include:

- a) the test method number and revision;
- b) identification of material tested;
- c) thickness of test specimen;
- d) ambient temperature and humidity;
- e) the size of the drops.
- f) interval of the drops;
- g) comparative tracking index
- h) any deviation from this test method;
- i) the name of the person conducting the test.

### 10.10 Test 2E10: Permittivity and dissipation factor (under consideration)

### 10.11 Test 2E11: Electric strength (under consideration)

### 10.12 Test 2E12: Resistance of foil (under consideration)

### 10.13 Test 2E13: Corrosion at edge (under consideration)

### 10.14 Test 2E14: Arc resistance

#### 10.14.1 Object

This method describes a technique for evaluating the ability of a material to resist tracking when subjected to a high-voltage low-current arc just above the surface of the insulating material. It can be used to assess materials of various thicknesses by stacking materials.

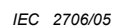
#### 10.14.2 Test specimens

- a) Three test specimens  $(50 \pm 5)$  mm  $\times$   $(50 \pm 5)$  mm  $\times$  thickness with the copper cladding totally removed by any commercially accepted method, and the surface thoroughly cleaned prior to conditioning or testing.
- b) Material under 1,6 mm in thickness shall be built up to provide a specimen at least 1,6 mm in thickness, but not exceeding 3,2 mm. For very thin laminates a 1,5 mm piece of the same type of material may be used under the actual specimen subjected to the arc, permitting a reduction of the material thickness required without significantly affecting the test results.

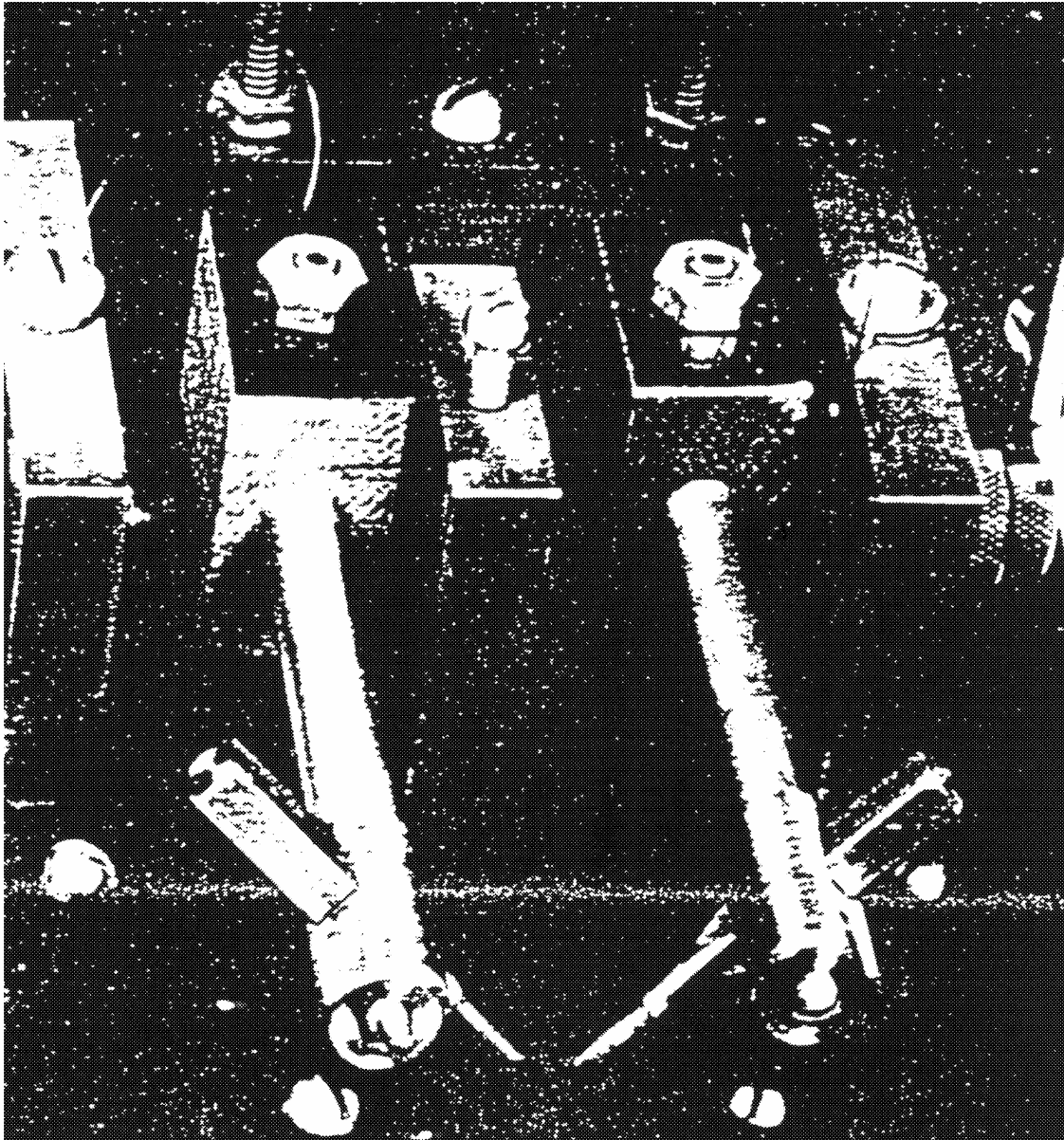
#### 10.14.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) an arc tester that conforms to electrical circuit as shown in Figure 33;
- b) a specimen holder;
- c) a tungsten steel electrodes assembly shown in Figure 34;
- d) a propan 2-ol (isopropyl alcohol);
- e) a mildly abrasive cloth;
- f) gauge blocks for checking 6,3 mm and 6,4 mm electrode spacings;
- g) a water bath capable of maintaining at  $(50 \pm 5)$  °C.



**Figure 33 – Arc-resistance test circuit**



IEC 351/97

**Figure 34 – Tungsten steel rod electrode assembly**

#### **10.14.4 Procedure**

- a) Specimens shall be placed in a deionized water bath maintained at  $(50 \pm 2) ^\circ\text{C}$  for  $(48^{+2}_0)$  h.
- b) The specimens shall be removed from this heated water bath and place them in deionized water at room temperature for 30 min minimum, 4 h maximum, to allow the specimens to reach ambient temperature without loss of moisture.
- c) The electrode assembly shall be cleaned using alcohol and an abrasive cloth with light enough abrasive as not to harm the electrodes, if necessary, and adjust the electrode gap to  $(6,35 \pm 0,05)$  mm when the electrodes are resting on a specimen.
- d) A specimen shall be removed from the deionized water and dried with a lint-free paper towel.
- e) A specimen shall be placed in the holder and under the electrodes. Raise the fixture until the electrodes rest on the surface of the specimen.



- f) The door to the test chamber shall be closed and the arc resistance unit turned on. The voltage shall be 12 500 V a.c.
- g) The timer shall be reset and the automatic test begun. The timing of the arc is shown in Table 8.
- h) The arc must be observed carefully. When the arc disappears and tracking occurs, the operator must remove the applied voltage and record the elapsed time to the nearest second.
- i) The specimen shall be removed. The electrodes shall be removed and cleaned by wiping them with a tissue and a solvent, such as isopropyl alcohol (propane 2-ol).
- j) The electrode shall be replaced, the electrode gap rechecked to assure that it is still within tolerance, and the test shall be repeated without a sample for 60 s at 10 mA.
- k) The above steps (a - j) shall be repeated for the remaining specimens.
- l) The arc resistance values of the specimens tested shall be averaged and rounded off to the nearest second.

**Table 8 – Arc resistance**

Time s	On/off time s	Amperage mA
0-60	0,25/1,75	10
60-120	0,25/0,75	10
120-180	0,25/0,25	10
> 180	Continuous	10

#### 10.14.5 Report

The report shall include:

- a) test number and revision index;
- b) the date of the test;
- c) identification of materials tested;
- d) average value of the arc resistance in seconds;
- e) ambient temperature and the relative humidity at the time of the test;
- f) specimen preparation (e.g. individual, number of layers built up, or with a spacer);
- g) any deviation from this test method.

#### 10.14.6 Additional information

The results of arc tests may be significantly affected by contamination of the electrodes. Any irregularity of the arc in the early portion of the test may be an indication of contamination. If this occurs, the test should be stopped, the electrodes cleaned and a fresh specimen should be tested (or the test may be run in a different area of the same specimen).

In the earlier steps, an interrupted arc is used to obtain a less severe condition than the continuous arc. A current of less than 10 mA produces an unsteady (flaring) arc.

**10.15 Test 2E15: Dielectric break-down** (under consideration)

**10.16 Test 2E16: Contact resistance of printed circuit keypad cont** (under consideration)

**10.17 Test 2E17: Insulation resistance of printed board materials**

**10.17.1 Object**

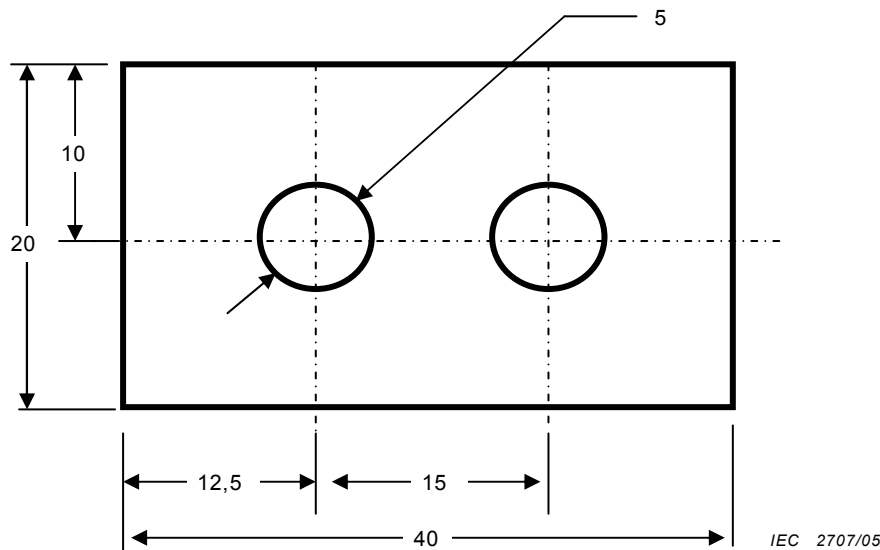
This test method covers the procedure for the determination of insulation resistance which includes the volume resistance and surface resistance of metal-clad base materials.

NOTE Test 2E04 of this standard should be referred to.

**10.17.2 Test specimens**

Test specimens shall be cut not less than 25 mm from the edge of the sheet. Test specimens shall be prepared from a sample of the metal clad base materials under test. The metal shall be completely removed by either etching or mechanical peeling. Three test specimen strips shall measure  $(40 \pm 2)$  mm long, and  $(20 \pm 1)$  mm wide. The edges shall be smoothed. Two holes with a diameter of 5 mm shall be drilled into the specimens (see Figure 35). All three specimens shall be subjected to the preconditioning steps including boiling.

*Dimensions in millimetres*



**Figure 35 – Test specimen for insulation resistance**

**10.17.3 Test apparatus and materials**

The following test apparatus and materials shall be used:

- High voltage breakdown tester (generally 50 kV minimum) with a current rating of 5 A over the entire voltage range and a motorized control capable of voltage increase of 500 V/s.
- Oil tank filled with insulating oil, capable of exceeding the requirements of the specification.
- Tapered pin electrode fixture utilizing two appropriate pins.  
NOTE Spherical ends on the pins are permitted and recommended to reduce likelihood of breakdown in the oil.
- High voltage test leads (leads rated in excess of machine capacity are recommended).
- Constant temperature water bath, capable of maintaining the adjusted temperature within  $\pm 2$  °C, filled with distilled water.

- f) Beaker or pan filled with ambient temperature water.
- g) Racks for supporting specimens in 50 °C water with all specimen surfaces exposed.
- h) Timer 0–60 s.
- i) Lint-free paper towels.

#### 10.17.4 Procedure

##### 10.17.4.1 Preconditioning

Precondition specimens for  $(96^{+2}_0)$  h at  $(20^{+5}_0)$  °C and  $(65 \pm 5)$  % RH. This preconditioning may be expressed as “C-96/20/65”

##### 10.17.4.2 Test method 1: Measurement at standard atmospheric conditioning

Measurements of insulation resistance shall be made at a temperature of  $(25 \pm 10)$  °C for  $(30 \pm 5)$  min after precondition (see 10.17.4.1). Each taper pin of 5 mm diameter shall be pushed into each hole of the specimen. Insulation resistance ( $\Omega$ ) shall be measured at the measuring voltage of 500 V d.c., using the device and the insulation resistance measuring equipment under the condition specified at  $(25 \pm 10)$ °C and  $(65 \pm 5)$  % RH after applying the test voltage to the specimen for 1 min.

##### 10.17.4.3 Test method 2: Measurement after boiling conditioning

- a) The preconditioned test specimens shall be boiled in distilled water for  $(120 \pm 10)$  min.
- b) After boiling, the test specimen shall be cooled for  $(30 \pm 5)$  min in clean water maintained at a temperature of  $(25 \pm 10)$  °C. The test specimen shall then be removed from the water and wiped with a clean dry gauze or similar material. The test specimen shall be measured after conditioning of  $(2,0 \pm 0,1)$  min under the same atmospheric conditions as given in 10.17.4.2.

#### 10.17.5 Report

The report shall include:

- a) the test method and revision;
- b) the date of the test;
- c) the description and identification of materials;
- d) the test condition description;
- e) average and minimum of insulation resistance;
- f) any deviation from this test method;
- g) the name of the person conducting the test.

#### 10.18 Test 2E18: Fungus resistance of printed board materials

##### 10.18.1 Object

The fungus resistance test is used to determine the resistance of materials to fungi and to determine if such material is adversely affected by fungi under conditions favourable for their development, namely high humidity, warm atmosphere and presence of inorganic salts.

##### 10.18.2 Test specimens

Specimens shall be a minimum size of 50 mm × 50 mm with copper foil (if applicable) removed by etching using standard commercial practices.

### 10.18.3 Apparatus and reagents

- a) Test chamber – The chamber shall be capable of maintaining  $(30 \pm 1) ^\circ\text{C}$  and  $(95 \pm 2) \%$  RH and an ultra violet (360 nm) source for subsequent decontamination. Provisions shall be made to prevent condensation from dripping on the test specimen. There shall be free circulation of air around the test specimen and the contact area of fixtures supporting the test specimen shall be kept to a minimum.
- b) Sterilizer
- c) Centrifuge
- d) pH meter
- e) Colony counter
- f) Incubator
- g) Dishwasher
- h) Petri dishes
- i) Filter paper
- j) Test media solutions
- k) Micro-organisms
- l) Atomizer,  $(15\,000 \pm 3\,000)$  spores
- m) Sodium dioctyl sulfosuccinate, or sodium lauryl sulphate
- n) Yeast
- o) Glycerol
- p) Benzalkonium choride

### 10.18.4 Procedure

#### 10.18.4.1 Preparation of test media

- a) Mineral-salts solution  
Prepare the solution to contain the following:  
Potassium dihydrogen orthophosphate ( $\text{KH}_2\text{PO}_4$ ) 0,7 g  
Potassium monohydrogen orthophosphate ( $\text{K}_2\text{HPO}_4$ ) 0,7 g  
Magnesium sulfate heptahydrate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) 0,7 g  
Ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) 1,0 g  
Sodium chloride ( $\text{NaCl}$ ) 0,005 g  
Ferrous sulfate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) 0,002 g  
Zinc sulfate heptahydrate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) 0,002 g  
Manganous sulfate monohydrate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ ) 0,001 g  
Benzalkonium chloride  
Distilled water 1 000 ml
- b) Sterilize the mineral salts solution by autoclaving at  $121 ^\circ\text{C}$  for 20 min. Adjust the pH of the solution by the addition of 0,01 normal solution of NaOH so that after sterilization the pH is between 6,0 and 6,5. Prepare sufficient salts solution for the required tests.

#### 10.18.4.2 Purity of reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise specified, it is intended that all reagents shall conform to the specification of the appropriate National Body that defines chemical reagents.

#### 10.18.4.3 Purity of water

Unless otherwise specified, references to water shall be understood to mean distilled water or water of equal purity.

#### 10.18.4.4 Preparation of mixed spore suspension

- a) The following test fungi shall be used:

NOTE The fungi are specified with numbers by ATCC. See item n) Sources for micro-organisms.

*Aspergillus niger*

*Chaetomium globosum*

*Gliocladium virans*

*Aureobasidium pullulans*

*Penicillium funiculosum*

- b) Maintain cultures of these fungi separately on an appropriate medium such as potato dextrose agar. However, the culture of *Chaetomium globosum* shall be cultured on strips of filter paper on the surface of mineral salts agar. (Mineral salts agar is identical to mineral salts solution, but contains in addition 15,0 g of agar per litre.)
- c) The stock cultures may be kept for not more than 4 months at  $(6 \pm 4) ^\circ\text{C}$  at which time sub-cultures shall be made and new stocks shall be selected from the sub-cultures.
- d) If genetic or physiological changes occur, obtain new cultures as specified above. Sub-cultures used for preparing new stock cultures or the spore suspension shall be incubated at  $(30 \pm 1) ^\circ\text{C}$  for 9 to 12 days or longer.
- e) Prepare a spore suspension of each of the five fungi by pouring into one subculture of each fungus, a 10-ml portion of a sterile solution containing 0,05 g per litre of a non-toxic wetting agent such as sodium dioctyl sulfosuccinate or sodium lauryl sulfate.
- f) Use a sterile platinum or nichrome inoculating wire to scrape gently the surface growth from the culture of the test organism.
- g) Pour the spore charge into a sterile 125 ml, glass-stoppered Erlenmeyer flask containing 45 ml of sterile water and 50 to 75 solid glass beads, 5 mm in diameter.
- h) Shake the flask vigorously to liberate the spores from the fruiting bodies and to break the spore clumps.
- i) Filter the dispersed fungal spore suspension, through a 6 mm layer of glass wool contained in a glass funnel, into a sterile flask.
- j) This process should remove large mycelial fragments and clumps of agar which could interfere with the spraying process.
- k) Centrifuge the filtered spore suspension aseptically and discard the supernatant liquid.
- l) Resuspend the residue in 50 ml of sterile water and centrifuge. Wash the spores obtained from each of the fungi in this manner three times.
- m) Dilute the final washed residue with sterile mineral salts solution in such a manner that the resultant spore suspension shall contain  $(1\,000\,000 \pm 200\,000)$  spores per ml as determined with a counting chamber.
- n) Repeat this operation for each organism used in the test and blend equal volumes of the resultant spore suspension to obtain the final mixed spore suspension. The spore suspension may be prepared fresh each day or may be held at  $(6 \pm 4) ^\circ\text{C}$  for not more than 7 days.

NOTE Sources for micro-organisms:

The fungi numbers specified by ATCC.

*Aspergillus niger* 9642

*Chaetomium globosum* 6205

*Gliocladium virans* 9645

*Aureobasidium pullulans* 9348

*Penicillium funiculosum* 9644

American Type Culture Collection  
12301 Parklawn Drive  
Rockville, MD 20852 USA  
(301) 881-2600 TELEX: 908768 ATCCROVE

Secondary Sources for Micro-organisms  
Pioneering Research Division  
U.S. Army Natick Laboratories  
Natick, MA 01760

North University St.  
Peoria, IL 61604  
Contact: Dr. Stephen Peterson  
309-685-4011

#### **10.18.4.5 Viability control by means of inoculation**

With each daily group of tests, place each of 3 pieces of sterilized filter paper, 2,5 cm<sup>2</sup>, on hardened mineral salts agar in separate Petri dishes. Inoculate these with the spore suspension by spraying the suspension from a sterilized atomizer until initiation of droplet coalescence. Incubate these at (30 ± 1) °C at a RH not less than 85 % and examine them after 7 days of incubation. There shall be copious growth on all three of the filter paper control specimens. Absence of such growth requires repetition of the test.

#### **10.18.4.6 Control specimens**

- a) In addition to the viability of inoculum control, known susceptible substrates shall be inoculated along with the test specimen to insure that proper conditions are present in the incubation chamber to promote fungus growth.
- b) The control specimens shall consist of cotton duck 8,25 oz. strips that are 5 cm in length, that have been dipped into a solution containing 10 % glycerol, 0.1 % potassium dihydrogen orthophosphate (KH<sub>2</sub>PO<sub>4</sub>), 0,1 % ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>), 0,025 % magnesium sulfate (MgSO<sub>4</sub> 7H<sub>2</sub>O), and 0,05 % yeast extract (pH 5,3), and from which the excess liquid has been removed.
- c) The strips should be hung to air dry before being inoculated and placed into the chamber.

#### **10.18.4.7 Inoculation of test specimens and control specimens**

- a) Mount the test specimens and control specimens on suitable fixtures or suspend from hangers. No cleaning of the test specimens shall be permitted for 72 h prior to the beginning of the fungus test. Equipment handling prior to and during the fungus test shall be accomplished without contamination of the equipment.
- b) The test and control specimens shall be inserted into the test chamber and preconditioned at a temperature of (30 ± 1) °C and a RH of (95 ± 2) % for at least 4 h.
- c) Inoculate the test and control specimens with the mixed fungus spore suspension (10.18.4.4) by spraying it on and into the test and control specimens (if not hermetically sealed) in the form of a fine mist from a previously sterilized atomizer or nebulizer. In spraying the test and control specimens, care should be taken to spray all surfaces which are exposed during use or maintenance. If the surfaces are non-wetting, spray until initiation of droplet coalescence. Incubation is to be started immediately following the inoculation.

#### 10.18.4.8 Test incubation of test specimens

- a) Incubate test specimens under cyclic temperature and humidity conditions to include 20 h of RH at  $(95 \pm 5) \%$  at an air temperature of  $(30 \pm 1) ^\circ\text{C}$  followed by 4 h of 100 % RH at  $(25 \pm 1) ^\circ\text{C}$ .
- b) After 7 days, inspect the growth on the control specimens to be assured that the environmental conditions are suitable for growth. If inspection reveals that the environmental conditions are unsuitable for growth, the entire test shall be repeated.
- c) If the control specimens show satisfactory fungus growth, continue the test for a period of 28 days from the time of inoculation, or as specified.

#### 10.18.5 Evaluation

- a) Report those specimens which were found to act as nutrient to fungus growth.
- b) Corrosion should be noted separately from the fungus test results.
- c) After evaluation, the materials and the test chamber shall be decontaminated by exposure on all sides to ultraviolet rays (360 nm) for a minimum of 2 h, or sprayed with a solution of 1:750 benzalkonium chloride solution (one part benzalkonium chloride to 750 parts distilled water).

#### 10.18.6 Safety

Observe all appropriate precautions on material safety data sheets (MSDS) for chemicals involved in this test method.

### 11 N: Environmental test methods

#### 11.1 Test 2N01: Pressure cooker test (under consideration)

#### 11.2 Test 2N02: Water absorption

##### 11.2.1 Object

This test method is designed for use in determining the amount of absorbed water in a specimen which has been immersed in water under specified conditions.

##### 11.2.2 Test specimens

Dimensions: The specimen shall be  $(50 \pm 1) \text{ mm} \times (50 \pm 1) \text{ mm} \times$  its thickness.

Edge finish: The edges of the specimens shall be milled or sanded smooth with 400 grit sandpaper.

Number of specimens: Three specimens shall be used for this test.

Removal of metal-cladding; The metal-cladding shall be completely removed by etching or other suitable method which does not affect the surface of laminates.

##### 11.2.3 Test apparatus and materials

The following test apparatus and materials shall be used:

- a) A balance, with an accuracy of 0,001 g.
- b) An oven, capable of being controlled at  $(50 \pm 2) ^\circ\text{C}$ .

- c) A container, containing distilled water, or water of equivalent purity, capable of being controlled at  $(23 \pm 0,5) ^\circ\text{C}$ .
- d) A desiccator, containing a sufficient amount of dry silica gel or calcium chloride.
- e) Slide calipers, with an accuracy of 0,1 mm or equivalent.

#### 11.2.4 Procedure

Dry three specimens for  $(24 \pm 1)$  h in an oven controlled at  $(50 \pm 2) ^\circ\text{C}$ , allow to cool to ambient temperature in the desiccator, and weigh each specimen to the nearest 0,001 g ( $M_1$ ).

Place the specimens in a container containing distilled water, maintained at  $(23 \pm 0,5) ^\circ\text{C}$ .

After immersion for  $(24 \pm 1)$  h, take out the specimens from the water and remove all surface water with a clean, dry cloth or with filter paper. Reweigh the specimens to the nearest 0,001 g within 1 min of taking them out of the water ( $M_2$ ).

For each specimen, calculate the water absorption rate to the nearest 0,01 % or the water absorption weight to the nearest 0,001 g as follows:

$$(1) \text{ Water absorption rate} = \frac{M_2 - M_1}{M_1} \times 100 \text{ (\%)}$$

$$(2) \text{ Water absorption weight} = M_2 - M_1 \text{ (g)}$$

#### 11.2.5 Report

The report shall include:

- a) the test method number and revision;
- b) date of test;
- c) the identification of the material tested;
- d) calculation method used for result as either water absorption rate (%) or water absorption weight (g);
- e) the average of the three measurements;
- f) any deviation from this test method.

#### 11.2.6 Additional information

Take care that the specimens do not come into contact with each other and/or with the walls of the container during immersion in the water.

## 12 X: Miscellaneous test methods

### 12.1 Test 2X02: Dimensional stability of thin laminates

#### 12.1.1 Object

This test method covers the procedure for the determination of dimensional stability of copper clad thin laminates for use in the fabrication of multilayer printed boards.

It provides information on all the linear dimensional changes occurring during multilayer printed board fabrication up to the lamination step.



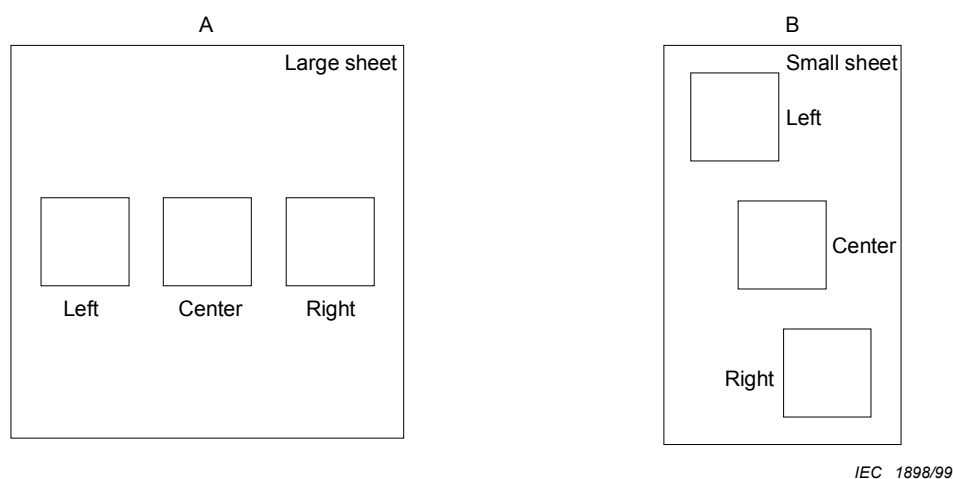
### 12.1.2 Test specimens

Specimens shall be cut not less than 25 mm from the edge of the sheet.

The specimens shall be prepared from a sample of the material under test by cutting them from different locations across the sample sheet as shown in Figure 36. One side of the specimen shall be cut parallel to the machine direction (warp direction) and it shall be identified.

Specimen size shall be agreed upon between purchaser and supplier, but shall be a minimum of 300 mm in both length and width.

Three specimens shall be tested.



**Figure 36 – Location of specimens on original sheet for dimensional stability test**

### 12.1.3 Test apparatus and materials

The following test apparatus and materials shall be used:

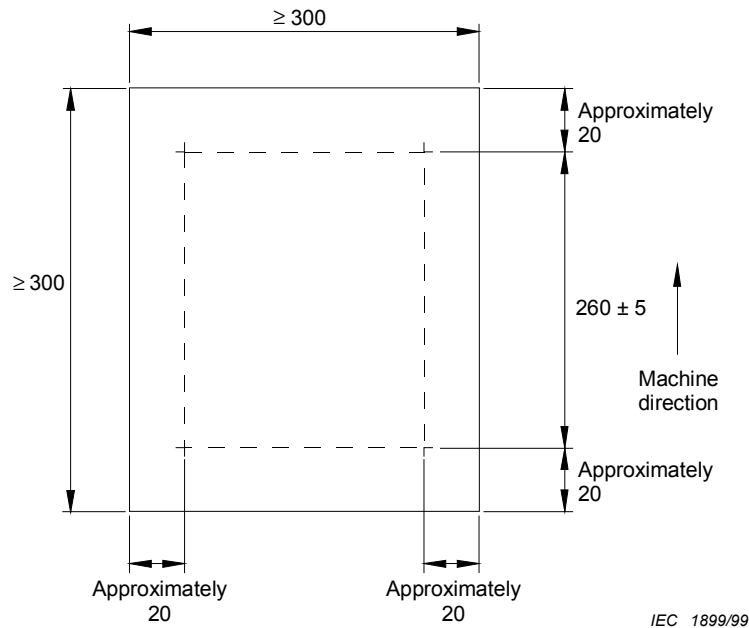
- Means for preparing suitable markings (drilling equipment, marking ink).
- Optical or mechanical measuring device with a measurement uncertainty not greater than 13  $\mu\text{m}$ .
- Oven of circulating type capable of maintaining  $(150 \pm 2) ^\circ\text{C}$ .

### 12.1.4 Procedure

The procedure includes the marking of the specimens, stabilization and conditioning steps before the measurement, measurements at the beginning and the end of the multilayer board fabrication process and the fabrication process itself.

Preparation of suitable markings (fiducials, drilled holes) in each corner of the specimen, of a minimum of approximately 20 mm from the edges, so that they form a square of the side length  $(260 \pm 5)$  mm. These markings shall be numbered 1-2-3-4 (see Figure 37).

*Dimensions in millimetres*



**Figure 37 – Location of marks on specimen for dimensional stability**

Stabilization of the specimens for  $(180^{+10}_0)$  min at  $(150 \pm 5) ^\circ\text{C}$  in a suitable oven and consequently conditioning for at least 18 h as specified in 5.2 of IEC 60068-1:  $(23 \pm 1) ^\circ\text{C}$  and  $(50 \pm 2) \% \text{RH}$ . For normal quality conformance testing, 3 h of conditioning before measuring is sufficient.

Initial measurement of the distances between the marks shown in Figure 37: 1 to 2, 2 to 3, 3 to 4, 4 to 1.

Stabilization of the specimens for  $180^{+10}_0$  min at  $(150 \pm 5) ^\circ\text{C}$  in a suitable oven and cooling down to room temperature.

Masking the measuring marks with suitable squares of side length not exceeding 13 mm capable of withstanding the following process steps.

Complete etching of the remaining area, stripping, oxidation and drying according to the relevant fabrication process.

Lamination of the specimens using two prepregs and one copper foil or single-sided copper-clad laminate on each side to form a four-layer multilayer printed board. One press package shall contain a stack of at least three multilayer boards, constructed with steel separator plates, release films and kraft paper.

The type of prepregs (for example glass fabric style 2112 or 7628), the kind of outer copper layer (foil or laminate) and the press cycle parameters shall be agreed upon between purchaser and supplier.

Removal of separator plates, release films and kraft paper. Trimming to original specimen size and complete etching of the copper surface.

Conditioning for  $180^{+10}_0$  min at  $(150 \pm 5) ^\circ\text{C}$  in a suitable oven and consequently conditioning for at least 18 h as specified in 5.2 of IEC 60068-1:  $(23 \pm 1) ^\circ\text{C}$  and  $(50 \pm 2) \% \text{RH}$ . For normal quality conformance testing, 3 h of conditioning before measurement is sufficient.

Final measurement of the distances between the marks shown in Figure 37: 1 to 2, 2 to 3, 3 to 4, 4 to 1.

#### 12.1.5 Report

The report shall include:

- a) the test number and revision;
- b) the date of the test;
- c) the identification of the material tested;
- d) the size of the specimens (warp direction);
- e) the process parameters (including lamination cycle);
- f) the multilayer board construction;
- g) the type, method, and accuracy of measurement;
- h) the average of the dimensional change in relation to the initial dimension for each direction of the sample sheet (expressed in mm/mm followed by the word "shrinking" or "expansion");
- i) any deviation from this test method.

#### 12.1.6 Additional information

If this test is required by the material specification, the process parameters and the requirements shall be specified.

## Annex A (informative)

### Worked examples

The following are worked examples of the uncertainty estimate for various printed board related tests:

#### Example 1:

Method: Repeated measurement of a 200 N (nominal mass) deadweight with a tensometer.  
(Comparable with PTH pull-out test)

<u>Raw data:</u>	198,50	198,50	198,75	198,75	198,75
	198,00	198,50	198,50	198,50	198,75
	198,20	198,50	198,50	198,50	198,75
	198,20	198,50	198,50	198,50	198,50
	198,00	198,50	198,75	198,50	198,75

Table “t” value:	t
Sample size (n):	25
Graduation of dial:	1,0 N
Mean:	198,50 N
Standard deviation ( $\sigma_{n-1}$ ):	0,215 N

$$U_r = \pm t (\sigma_{n-1}) / \sqrt{n} = \pm 0,08858 \text{ N}$$

$$U_s = \pm 1 \% \text{ (as shown on calibration certificate)} = \pm 1,985 \text{ N}$$

$$U_i = \pm 20 \% \text{ of graduation} = \pm 0,2 \text{ N}$$

$$U_t = \pm \sqrt{(U_s^2 + U_r^2)} + U_i = \pm 2,187 \text{ N} = \pm 1,1 \%$$

Thus, uncertainty of measurement is estimated as  $\pm 1,1 \%$ .

#### Example 2:

Method: Repeated measurement of the level of ionic contamination in a test solution, as represented by the introduction of a metered quantity of the calibration solution. Measured in a commercial ionic contamination test instrument.

<u>Raw data:</u>	1,00	1,03	1,11	1,03	1,05
	1,10	0,92	1,06	0,91	0,92
	(measurement unit $\mu\text{g}/\text{cm}^2$ EQ. NaCl)				

Table “t” value:	t
Sample size (n):	10
Mean:	1,013 $\mu\text{g}$
Standard deviation ( $\sigma_{n-1}$ ):	0,074 $\mu\text{g}$
Graduation of dial:	not applicable

$$U_r = \pm t (\sigma_{n-1}) / \sqrt{n} = \pm 0,0533 \text{ mg}/\text{cm}^2$$

$$U_s = \pm 5 \% \text{ (calibration of Gilman pipette/test solution)} = \pm 0,05 \text{ mg}$$

$$U_t = \pm \sqrt{(U_s^2 + U_r^2)} = \pm 0,0729 \text{ mg} = \pm 7,29 \%$$

Thus, uncertainty of measurement is estimated as  $\pm 7,29 \%$ .

**Example 3:**

Method: Repeated measurement of a surface insulation resistance (SIR) test specimen (nominal value 100 MΩ) using a megohmmeter.

Raw data:	68,00	102,50	105,00	108,75
	81,25	102,50	107,50	113,75
	90,00	102,50	107,50	

Table “t” value:	t
Sample size (n):	11
Mean:	99,02 MΩ
Standard deviation ( $\sigma_{n-1}$ ):	13,73 MΩ
Graduation of dial:	1,25 MΩ

$$U_r = \pm t (\sigma_{n-1}) / \sqrt{n} = \pm 9,2223 \text{ M}\Omega$$

$$U_s = \pm 0,5 \% \text{ (as shown on calibration certificate)}$$

$$= \pm 0,5 \% \text{ of } 99,02 = \pm 0,4951 \text{ M}\Omega$$

$$U_i = \pm 20 \% \text{ of graduation (1,25 M}\Omega) = \pm 0,25 \text{ M}\Omega$$

$$U_t = \pm \sqrt{(U_s^2 + U_r^2)} + U_i = \pm 9,456 \text{ M}\Omega = \pm 9,5 \%$$

## Annex B (informative)

### Conversion table

Publication number <sup>1)</sup>	Test method number <sup>2)</sup>	Description	Current publication number	Test method number
IEC 61189-1	1P01	Preconditioning, standard atmospheric conditioning	IEC 60326-2/ 9.1.1	18a
	1P02	Preconditioning, 125 °C	IEC 60326-2/ 9.1.2	18b
	1P03	Accelerated ageing, steam/oxygen	IEC 60326-2/9.4	20a
IEC 61189-2	2C01	Resistance to sodium hydroxide		
	2C02	Gel time of prepregnation		
	2C03	Resin content of prepregnation by treated weight		
	2C04	Volatile content of prepregnation		
	2C05	Blistering after thermal shock	IEC 60249/3.7	
	2C06	Flammability, vertical	IEC 60249/4.3.4	
	2C07	Flammability, horizontal	IEC 60249/4.3.3	
	2C08	Flammability, flex material	IEC 60249/4.3.5	
	2C09	Melting viscosity of prepregnation materials		
	2C10	Resin content of prepregnation by sublimation		
	2D01	Thickness	IEC 60249/3.14	
	2E01	Surface tracking, moisture condition	IEC 60112	
	2E02	Electrical strength at pwr	IEC 60243	
	2E03	Surface resistance, damp heat, steady state	IEC 60249/2.2	
	2E04	Volume resistivity, damp heat, steady state	IEC 60249/2.3	
	2E05	Permittivity and dielectric dissipation	IEC 60250	
	2E06	Volume and surface resistivity, 3 electrodes	IEC 60093	
	2E07	Surface and volume resistivity, elevated temperature	IEC 60249/2.9	
	2E08	Surface corrosion	IEC 60249/2.4	
	2E09	Comparative tracking index	IEC 60249/2.6	
<p><b>NOTES</b></p> <p>1) IEC 61189: Test methods for electrical materials, interconnection structures and assemblies  Part 1: General test methods and methodology  Part 2: Test methods for materials for interconnection structures  Part 3: Test methods for interconnection structures (printed boards)</p> <p>2) C = chemical  D = dimensional  E = electrical  M = mechanical  N = environmental  P = preparation/conditioning  V = visual  X = miscellaneous</p>				

Publication number	Test method number	Description	Current publication number	Test method number
IEC 61189-2	2E10	Permittivity and dissipation factor	IEC 60249/2.7	
	2E11	Electric strength	IEC 60249/2.8	
	2E12	Resistance of foil	IEC 60249/2.1	
	2E13	Corrosion at edge	IEC 60249/2.5	
	2E14	Arc resistance		
	2E15	Dielectric break-down		
	2E16	Contact resistance of printed circuit keypad cont.		
	2M01	Test method for Bow and twist	IEC 60249/3.1	
	2M02	Bow/twist after etching and heating	IEC 60249/3.2	
	2M03	Cure factor of base materials by DSC/TMA	IEC 60249/3.3	
	2M04	Twist after heating	IEC 60249/3.4	
	2M05	Pull-off strength	IEC 60249/3.5	
	2M06	Peel strength after exposure to solvent vapour	IEC 60249/3.6.4	
	2M07	Peel strength/solvent dip	IEC 60249/3.6.6	
	2M08	Flexural strength	ISO 178	
	2M09	Resin flow of prepregation		
	2M10	Delta glass transition (DSC)		
	2M11	Glass transition temperature (TMA)		
	2M12	Surface waviness		
	2M13	Peel strength/after condition	IEC 60249/3.6.1	
	2M14	Peel strength/thermal shock	IEC 60249/3.6.2	
	2M15	Peel strength/dry heat	IEC 60249/3.6.3	
	2M16	Peel strength/simulated plating	IEC 60249/3.6.5	
	2M17	Peel strength/elevated temperature	IEC 60249/3.6.7	
	2M18	Surface quality	IEC 60249/3.9	
	2M19	Punching	IEC 60249/3.8	
	2M20	Flexural strength	IEC 60249/4.1	
	2M21	Flexural fatigue for flexible laminates	IEC 60249/3.12	
	2M22	Weight of foil after lamination (etching)	IEC 60249/3.13	
	2M23	Squareness	IEC 60249/3.15	
	2M24	Coefficient of thermal expansion	IEC 60249/4.5	
	2M25	Time to delamination		
	2M26	Scaled flow of prepregation		

Publication number	Test method number	Description	Current publication number	Test method number
IEC 61189-2	2M27	Resin flow properties, coverlay and bonding films		
	2N01	Pressure cooker test	IEC 60249/4.2	
	2N02	Water absorption	IEC 60249/4.4	
	2P01	Dry heat	IEC 60068-2-2/Ba	
	2P02	Solder float stress	IEC 60068-2-20/T	
	2X02	Dimensional stability, thin laminates	IEC 60249/3.11	
IEC 61189-3	3C01	Flammability, rigid printed board metal removal	IEC 60326-2/8.4.1	16a
	3C02	Flammability, rigid printed board glow-wire test	IEC 60326-2/8.4.2	16b
	3C03	Flammability, rigid printed board needle flame test	IEC 60326-2/8.4.3	16c
	3C04	Solvent and flux resistance	IEC 60326-2/8.5	17a
	3C05	Electrolytic corrosion, rigid and thin film	IEC 60426/all	
	3C06	Flammability, glow-wire test, rigid printed boards	IEC 60695-2-1	
	3C07	Flammability, needle flame, rigid printed boards	IEC 60695-2-2	
	3C08	Vertical burning	ISO R 1326	
	3C09	Water absorption	ISO Std 62	
	3C10	Surface organic contaminants (in-house)		
	3C11	Resistivity of solvent extract (ionic contaminants)		
	3C12	Surface organic contaminants (infrared)		
	3D01	Optical method	IEC 60326-2/5.2.2	2a
	3D02	Conductor width and spacing		
	3D03	Automated optical inspection		
	3D04	Dimensional examination, general	IEC 60326-2/5.2	2
	3E01	Circuit isolation	IEC 60326-2/6.2.1	4a
	3E02	Circuit continuity	IEC 60326-2/6.2.2	4b
	3E03	Insulation resistance, surface layers	IEC 60326-2/6.4.1	6a
	3E04	Insulation resistance, internal layers	IEC 60326-2/6.4.2	6b
	3E05	Insulation resistance, between layers	IEC 60326-2/6.4.3	6c
	3E06	Frequency drift	IEC 60326-2/6.6	8a
	3E07	Circuit impedance	IEC 60326-2/6.7	9a
	3E08	Plated through hole resistance change, thermo cycle	IEC 60326-2/6.1.3	3c
	3E09	Voltage proof, surface layers	IEC 60326-2/6.5.1	7a
	3E10	Voltage proof, between layers	IEC 60326-2/6.5.2	7b



Publication number	Test method number	Description	Current publication number	Test method number
IEC 61189-3	3E11	Interconnection resistance, multilayer printed boards		
	3E12	Resistance of conductors	IEC 60326-2/6.1.1	3a
	3E13	Resistance of interconnections	IEC 60326-2/6.1.2	3b
	3E14	Current proof, plated through hole	IEC 60326-2/6.3.1	5a
	3E15	Current proof, conductors	IEC 60326-2/6.3.2	5b
	3E16	Plated through hole resistance change, thermal shock	IEC 60326-2/6.1.3	3c
	3E17	Production determination of characteristic impedance by time domain reflectometry		
	3M01	Peel strength, standard atmospheric	IEC 60326-2/7.1.1	10a
	3M02	Peel strength, elevated temperature	IEC 60326-2/7.1.2	10b
	3M03	Pull-out strength, landless plated through hole	IEC 60326-2/7.2.2	11b
	3M04	Flatness	IEC 60326-2/7.3	12a
	3M05	Peel strength, flexible printed board, standard atmospheric conditions	IEC 60326-2/7.1.3	10c
	3M06	Flexural fatigue, flexible printed board	IEC 60326-2/7.4	21a
	3M07	Pull-off strength, lands with plain holes	IEC 60326-2/7.2.1	11a
	3M08	Resistance of abrasion of printed board organic surface coatings		
	3M09	Degree of cure of printed board organic surface coatings		
	3N01	Thermal shock, immersion, oil bath	IEC 60326-2/9.2.1	19a
	3N02	Thermal shock, float, solder, 280 °C	IEC 60326-2/9.2.3	19c
	3N03	Thermal shock, hand soldering	IEC 60326-2/9.2.4	19d
	3N04	Thermal shock, dip soldering	IEC 60326-2/9.2.5	19e
	3N05	Thermal shock, floating, solder bath 288 °C	IEC 60326-2/9.2.6	19f
	3N06	Damp heat, steady state	IEC 60068-2-3/Ca	
	3N07	Temperature cycling	IEC 60068-2-30/Da	
	3N08	Thermal shock, immersion fluidized sand	IEC 60326-2/9.2.2	19b
	3N12	Moisture and insulation resistance for printed boards		
	3V01	3 X magnification	IEC 60326-2/5.1.1	1a
	3V02	10 X magnification	IEC 60326-2/5.1.2	1b
	3V03	250 X magnification	IEC 60326-2/5.1.3	1c
	3V04	General visual	IEC 60326-2/5.1	1

Publication number	Test method number	Description	Current publication number	Test method number
IEC 61189-3	3X01	Plating adhesion, tape method	IEC 60326-2/8.1.1	13a
	3X02	Plating adhesion, burnish method	IEC 60326-2/8.1.2	13b
	3X03	Porosity, gas exposure	IEC 60326-2/8.1.3	13c
	3X04	Porosity, electrographic, gold on copper	IEC 60326-2/8.1.4	13d
	3X05	Porosity, electrographic, gold on nickel	IEC 60326-2/8.1.5	13e
	3X06	Plating thickness	IEC 60326-2/8.1.6	13f
	3X07	Solderability, edge dip test	IEC 60326-2/8.2	14a
	3X08	Delamination, thermal shock	IEC 60326-2/8.3.1	15a
	3X09	Microsection	IEC 60326-2/8.3.2	15b
	3X10	Solderability, rotary dip test	IEC 60326-2/8.2	14a
	3X11	Assessment of multilayer printed board for inner layers		
	3X12	Adhesion of organic surface finish to printed board		

## Annex C (informative)

### Laboratory pro forma (form)

Date:...../...../.....

Specimen description: .....

.....

Specimen thickness:.....mm

#### As received

Repeat if  $t_1, t_2 > 10$  s  
or if  $t = 50$  s to 55 s

		1	2	3	4	5		1	2	3	4	5
1 <sup>st</sup> Burn time; $t_1$	s											
2 <sup>nd</sup> Burn time; $t_2$	s											
2 <sup>nd</sup> Burn time and glow	s											
Burns up to clamp?	Y/N											
Drips?	Y/N											

Total burn time ( $t$ ): .....s Repeated total burn time:.....s

#### After 24 h at 125 °C

Repeat if  $t_1, t_2 > 10$  s  
or if  $t = 50$  s to 55 s

		1	2	3	4	5		1	2	3	4	5
1 <sup>st</sup> Burn time; $t_1$	s											
2 <sup>nd</sup> Burn time; $t_2$	s											
2 <sup>nd</sup> Burn time and glow	s											
Burns up to clamp?	Y/N											
Drips?	Y/N											

Total burn time ( $t$ ): .....s

Repeat total burn time: .....s

**Annex D**  
(informative)

**Laboratory pro forma**

**Laboratory pro forma (form)**

**Date:**...../...../.....

**Specimen description:** .....

.....

**Specimen thickness:**.....mm

**As received**

		1	2	3	4
Burn time	s				
Burns past line?	Y/N				
Drips?	Y/N				
Drips burn	Y/N				

Average burn time (t): .....s

..... Signed:

\_\_\_\_\_



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**International Electrotechnical Commission**

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or

Fax to: **IEC/CSC** at +41 22 919 03 00

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.....

**Q2** Please tell us in what capacity(ies) you bought the standard (tick all that apply). I am the/a:

- purchasing agent ☐  
 librarian ☐  
 researcher ☐  
 design engineer ☐  
 safety engineer ☐  
 testing engineer ☐  
 marketing specialist ☐  
 other.....

**Q3** I work for/in/as a:  
(tick all that apply)

- manufacturing ☐  
 consultant ☐  
 government ☐  
 test/certification facility ☐  
 public utility ☐  
 education ☐  
 military ☐  
 other.....

**Q4** This standard will be used for:  
(tick all that apply)

- general reference ☐  
 product research ☐  
 product design/development ☐  
 specifications ☐  
 tenders ☐  
 quality assessment ☐  
 certification ☐  
 technical documentation ☐  
 thesis ☐  
 manufacturing ☐  
 other.....

**Q5** This standard meets my needs:  
(tick one)

- not at all ☐  
 nearly ☐  
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 exactly ☐

**Q6** If you ticked NOT AT ALL in Question 5 the reason is: (tick all that apply)

- standard is out of date ☐  
 standard is incomplete ☐  
 standard is too academic ☐  
 standard is too superficial ☐  
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 I made the wrong choice ☐  
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- (1) unacceptable,  
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 (5) exceptional,  
 (6) not applicable

- timeliness.....  
 quality of writing.....  
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 logic of arrangement of contents .....  
 tables, charts, graphs, figures.....  
 other .....

**Q8** I read/use the: (tick one)

- French text only ☐  
 English text only ☐  
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**Q9** Please share any comment on any aspect of the IEC that you would like us to know:

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