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# StandardTest Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials<sup>1</sup>

This standard is issued under the fixed designation D5229/D5229M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

#### INTRODUCTION

Consistent evaluation and comparison of the response of polymer matrix composites to moisture absorption can only be performed when the material has been brought to a uniform through-the-thickness moisture profile. The procedures described in Test Method D570 and Practices D618 do not guarantee moisture equilibrium of the material. A similar, but more rigorous, procedure for conditioning to equilibrium is described by this test method, which can also be used with fluid moisture other than water, and which, additionally, can provide the moisture absorption properties necessary for the analysis of single-phase Fickian moisture diffusion within such materials.

# 1. Scope

1.1 This test method covers a procedure for the determination of moisture absorption or desorption properties in the through-the-thickness direction for single-phase Fickian solid materials in flat or curved panel form. Also covered are procedures for conditioning test coupons prior to use in other test methods; either to an essentially moisture-free state, to equilibrium in a standard laboratory atmosphere environment, or to equilibrium in a non-laboratory environment. Also included are procedures for determining the moisture loss during elevated temperature testing, as well as moisture loss

resulting from thermal exposure after removal from the conditioning environment, such as during strain gage bonding. While intended primarily for laminated polymer matrix composite materials, these procedures are also applicable to other materials that satisfy the assumptions of 1.2.

1.2 The calculation of the through-the-thickness moisture diffusivity constant in Procedure A assumes a single-phase Fickian material with constant moisture absorption properties through the thickness of the specimen. The validity of the equations used in Procedure A for evaluating the moisture diffusivity constant in a material of previously unknown moisture absorption behavior is uncertain prior to the test, as the test results themselves determine if the material follows the single-phase Fickian diffusion model. A reinforced polymer matrix composite material tested below its glass-transition temperature typically meets this requirement, although twophase matrices such as toughened epoxies may require a multi-phase moisture absorption model. While the test procedures themselves may be used for multi-phase materials, the calculations used to determine the moisture diffusivity constant in Procedure A are applicable only to single-phase materials. Other examples of materials and test conditions that may not meet the requirements are discussed in Section 6.

1.3 The evaluation by Procedure A of the moisture equilibrium content material property does not assume, and is therefore not limited to, single-phase Fickian diffusion behavior.

1.4 The procedures used by this test method may be performed, and the resulting data reduced, by suitable automatic equipment.

1.5 This test method is consistent with the recommendations of CMH-17 Rev G (1),<sup>2</sup> which describes the desirable attributes of a conditioning and moisture property determination procedure.

1.6 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

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 $<sup>^{2}</sup>$  The boldface numbers in parentheses refer to the list of references at the end of this standard.

(L) D5229/D5229M – 12

1.6.1 Within the text the inch-pound units are shown in brackets.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

# 2. Referenced Documents

2.1 ASTM Standards:<sup>3</sup>

D570 Test Method for Water Absorption of Plastics

D618 Practice for Conditioning Plastics for Testing

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

**D883** Terminology Relating to Plastics

D2584 Test Method for Ignition Loss of Cured Reinforced Resins

D2734 Test Methods for Void Content of Reinforced Plastics

D3171 Test Methods for Constituent Content of Composite Materials

D3878 Terminology for Composite Materials

2.2 Military Standard:

MIL-B-131 Barrier Materials, Watervaporproof, Greaseproof, Flexible, Heat-Sealable<sup>4</sup>

### 3. Terminology

3.1 Definitions:

3.1.1 Terminology D3878 defines terms relating to highmodulus fibers and their composites. Terminology D883 defines terms relating to plastics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other terminology standards.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental dimensions, shown within square brackets: [M] for mass, [L] for length, [T] for time,  $[\Theta]$  for thermodynamic temperature, and [nd] for non-dimensional quantities. Use of these symbols is restricted to analytical dimensions when used within square brackets, as the symbols may have other definitions when used without the brackets.

3.2.2 accuracy criterion, *n*—the maximum amount of change in average moisture content for a test coupon, over the span of the reference time period, which is allowable for the establishment of effective moisture equilibrium. (See also average moisture content, moisture equilibrium, and reference time period.)

3.2.3 average moisture content, M (%), n—the average amount of absorbed moisture in a material, taken as the ratio of

the mass of the moisture in the material to the mass of the oven-dry material and expressed as a percentage, as follows:

$$M, \% = \frac{W_i - W_o}{W_o} \times 100 \tag{1}$$

where:

 $W_i$  = current specimen mass, g, and

 $W_o$  = oven-dry specimen mass, g.

(See also oven-dry.)

3.2.4 *Fickian diffusion*, *n*—a model of material moisture absorption and desorption that follows Fick's second law, as follows in one-dimension:

$$\frac{\partial c}{\partial t} = D_z \frac{\partial^2 c}{\partial z^2}$$

3.2.5 glass transition temperature,  $T_g[\Theta]$ , *n*—the approximate midpoint of the temperature range over which a reversible change takes place between a viscous or rubbery condition and a hard, relatively brittle condition, in an amorphous polymer, or in amorphous regions of a partially crystalline polymer.

3.2.5.1 *Discussion*—The glass transition temperature of many polymer matrix composites is lowered by the presence of absorbed moisture.

**3.2.6** *moisture*, *n*—liquid (water, jet fuel, salt water, or any other liquid) that is either diffused in relatively small quantity and dispersed through a gas as a vapor, condensed on a surface as visible dew, or present in quantity sufficient for immersion of an object.

3.2.6.1 *Discussion*—The dictionary definition of moisture for this test method is extended to include not only the vapor of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

3.2.7 moisture concentration,  $c [ML^{-3}]$ , *n*—the absolute amount of absorbed moisture in a material expressed as the mass of moisture per unit volume.

3.2.8 moisture diffusivity constant,  $D_z [L^2 T^{-1}]$ , *n*—the property of a material that describes the rate at which the material absorbs or desorbs moisture.

3.2.8.1 *Discussion*—In Fickian materials this property is relatively independent of the moisture exposure level (and thus the moisture equilibrium content material property). However, the moisture diffusivity constant is strongly influenced by temperature. Moisture diffusivity can be anisotropic; the subscript z indicates the value in the through-the-thickness direction for anisotropic diffusion behavior.

3.2.9 moisture equilibrium, n—the condition reached by a material when there is essentially no further change in its average moisture content with the surrounding environment. Moisture equilibrium can be either *absolute* or *effective*. Absolute moisture equilibrium requires no measurable change in moisture content, while effective moisture equilibrium allows a specified small change in the average moisture content of a material (the accuracy criterion) over a specified time span (the reference time period). (See also accuracy criterion, average moisture content, and reference time period.)

<sup>&</sup>lt;sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>4</sup> Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.



3.2.9.1 *Discussion*—Effective moisture equilibrium is a satisfactory definition for most engineering applications. Unless otherwise specified, references to moisture equilibrium in this test method mean effective moisture equilibrium, as quantified in section 10.2. Moisture equilibrium can also be either *static*, when there is no moisture transport at all across the surfaces, or *dynamic*, when moisture transport exists, but the net sum for the material is zero. This test method is not capable of discerning between these two types of moisture equilibrium.

3.2.10 moisture equilibrium content,  $M_m$  (%), n—the maximum amount of absorbed moisture that a material can contain at moisture equilibrium for a given moisture exposure level, expressed as a percent of dry material mass. (See also moisture saturation content.)

3.2.10.1 *Discussion*—In polymer matrix composites, this property is relatively independent of temperature (and thus the moisture diffusivity constant material property), but it is a function of the moisture exposure level. For the purposes of this test method  $M_m$  is assumed to be equivalent to the average moisture content at effective moisture equilibrium,  $M_f$ .

3.2.11 *moisture exposure level, n*—a measure or description of the severity of a conditioning environment in terms of the amount of liquid or vapor present. (See also *moisture* and *relative vapor level.*)

3.2.12 *moisture saturation content*, *n*—the moisture equilibrium content at the maximum possible moisture exposure level, wherein the material contains the greatest possible amount of absorbed moisture. (See also *moisture equilibrium content*.)

3.2.13 *oven-dry*, *n*—the condition of a material that has been dried in accordance with Procedure D of this test method until moisture equilibrium is achieved.

3.2.14 reference time period, n—the time interval for mass measurement used to define effective moisture equilibrium in a material. (See also accuracy criterion, average moisture content, and moisture equilibrium.)

3.2.14.1 *Discussion*—A small change in the average moisture content (the accuracy criterion) for a material during the reference time period indicates effective moisture equilibrium.

3.2.15 *relative vapor level* (%), n—the ratio of the pressure of a vapor present to the pressure of the saturated vapor, at the same temperature, expressed as a percent. Applicable only to the gaseous form of a fluid. When the vapor is water vapor the term is called *relative humidity*. (See also *moisture exposure level*.)

3.2.16 standard laboratory atmosphere, n—an atmosphere (environment) having a temperature of  $23 \pm 2^{\circ}$ C [73.4  $\pm$  3.6°F] and a relative humidity of 50  $\pm$  10 %.

3.2.17 *standard conditioned specimen, n*—the material condition of a test coupon that has reached effective moisture equilibrium at a nominal relative humidity of 50 % (considered to be a standard laboratory environment) in accordance with Procedure C of this test method.

3.2.18 *test temperature*, *n*—the environmental temperature used in Procedures A-E, Y, Z.

3.2.18.1 Discussion—This is distinguished, for the purposes

of this test method, from the environmental temperature used during any subsequent material evaluation testing.

3.2.19 *traveler coupon*, *n*—a surrogate coupon of the same material and thickness, and of appropriate size (but without tabs) that is used in a conditioning procedure to determine moisture content for specimen configurations (such as a tabbed mechanical coupon, or a coupon that does not meet the minimum mass requirement) that cannot otherwise be properly measured by this test method.

3.3 Symbols:

3.3.1 *c*—moisture concentration.

3.3.2  $D_z$ —moisture diffusivity constant in the through-thethickness direction.

3.3.3 G(T,t)—moisture absorption or desorption function for materials that follow Fickian diffusion.

3.3.4 h—thickness of a material panel or plate in the through-the-thickness direction for double-sided moisture exposure.

3.3.5 M—average moisture content of a material. The following subscripts denote the average moisture content for specific conditions:

 $M_{b}$ , the average moisture content at a baseline time;

 $M_e$ , the average moisture content at establishment of effective moisture equilibrium;

 $M_{ed}$ , the average moisture content at establishment of effective moisture equilibrium as a delta from the average moisture content at a baseline time;

 $M_{et}$ , the average moisture content at establishment of effective moisture equilibrium as a total value from the condition of zero moisture content;

 $M_{f}$ , the final moisture content at the end of conditioning for a fixed time;

 $M_i$ , the average moisture content at a given time;

 $M_{i-1}$ , the average moisture content at the previous time;

 $M_L$ , the moisture loss from the state of effective equilibrium due to subsequent heating; and

 $M_m$ , the moisture equilibrium content that is reached when a uniform through-the-thickness moisture profile occurs for a given temperature and moisture exposure level.

3.3.5.1 *Discussion*—Procedures A-H of this test method condition specimens to  $M_e$ . Except for the use of a thin specimen in Procedure A, conditioning specimens to  $M_m$  is generally not practical. See also the discussion in Appendix X2.

3.3.6 *t*—time.

3.3.7  $t_m$ —the maximum time required for a material to reach moisture equilibrium under specified conditions of temperature and initial moisture content.

3.3.8  $T_{g}$ —glass transition temperature.

3.3.9 w—the width of a nominally square moisture absorption test coupon.

3.3.10 *W*—the mass of a test coupon. The following subscripts are used to denote the mass of a test coupon for specific conditions:

 $W_b$ , the mass at the baseline time;

⊕ D5229/D5229M – 12

 $W_i$ , the mass at a given time;

and  $W_{i-1}$ , the mass at the previous time.

3.3.11 *z*—the coordinate axis in the through-the-thickness direction for a plate or panel.

#### 4. Summary of Test Method

4.1 This is a gravimetric test method that monitors the change over time to the average moisture content of a material specimen by measuring the total mass change of coupons that are exposed on two sides to a specified environment. There are a number of test procedures described by this test method.

4.1.1 Procedure A covers the determination of the two Fickian moisture diffusion material properties, the moisture diffusivity constant and the moisture equilibrium content. The other procedures cover material conditioning to a specific moisture environment.

4.1.2 Procedure B covers non-ambient moisture conditioning of material coupons at a prescribed constant conditioning environment prior to other types of testing. Sub-procedure codes are used to specify the conditioning environment, conditioning time, and mass change monitoring procedure.

4.1.3 Procedure C covers the ambient conditioning of material coupons to a nominal relative humidity level of 50 %, prior to other types of testing.

4.1.4 Procedure D covers the conditioning (drying) of material coupons to an essentially moisture-free condition.

4.1.5 Procedure E covers conditioning in two or more sequential steps using Procedure Bxxx.

4.1.6 Procedure Y covers the determination of the amount of moisture loss in conditioned material coupons when removed from the conditioning environment (such as due to heating during cure of strain gages).

4.1.7 Procedure Z covers the determination of the amount of moisture loss in conditioned material coupons due to heating to the mechanical test temperature and holding at the test temperature for the duration of the mechanical test.

4.2 In Procedure A, the percent moisture mass gain versus time is monitored for thin material specimen(s) that are maintained in a steady-state environment at a known temperature and moisture exposure level until the material reaches effective moisture equilibrium, and thick material specimen(s) that are maintained in a steady-state environment at a known temperature and moisture exposure level until the material is conditioned past the point of linear moisture gain. From the data obtained from these two specimen thicknesses, the moisture equilibrium content,  $M_m$ , and the one-dimensional moisture absorption rate of the coupon may be determined and the through-the-thickness moisture diffusivity constant,  $D_z$ , calculated.

4.3 In Procedure B, a general test coupon (not necessarily the coupon of Procedure A) is maintained, similarly to Procedure A, in a steady-state environment at specified temperature and environmental exposure levels until the material reaches effective moisture equilibrium.

4.4 In Procedure C, a general test coupon is maintained in a steady-state environment at a specified temperature and a relative humidity of 50 % until the material reaches effective moisture equilibrium.

4.5 In Procedure D, a general test coupon is maintained in an air-circulating oven at a prescribed elevated temperature environment until effective moisture equilibrium is reached.

4.6 In Procedure E, a general test coupon is conditioned in two or more sequential non-ambient steady-state environments.

4.7 In Procedure Y, a general test coupon is first conditioned to equilibrium using Procedure B, then is exposed to room temperature ambient followed by an elevated temperature for defined times, then again conditioned to equilibrium.

4.8 In Procedure Z, a general test coupon is first conditioned to equilibrium using Procedure B, then is exposed to the same testing environment (nominal ramp and soak time and an additional hold time to mimic the test time) as the specimens they represent.

#### 5. Significance and Use

5.1 Procedure A is designed to produce moisture diffusion material property data that may be used as follows:

5.1.1 To determine approximate exposure times for coupon conditioning in Procedures B-E, Y, Z;

5.1.2 As input to moisture prediction analysis computer codes; or

5.1.3 For making qualitative decisions on material selection or performance under environmental exposure to various forms of moisture.

5.2 Procedures B-E are designed to condition test coupons to a specified environmental condition or equilibrium state prior to other material property testing (including but not limited to mechanical testing).

5.3 Procedures Y-Z are designed to determine the loss of moisture content due to removal of a test coupon from the conditioning chamber (such as for strain gage bonding) or due to heating of the test coupon prior to and during mechanical loading.

5.4 A single pair of tests on thin and thick specimens using Procedure A provides the moisture diffusivity constant,  $D_z$ , and the moisture equilibrium content,  $M_m$ , at the given moisture exposure level and temperature. Multiple tests at differing temperatures are required to establish the dependence of  $D_z$  on temperature. Multiple tests at differing moisture exposure levels are required to establish the dependence of  $M_m$  on moisture exposure level.

Note 1—For many polymer matrix composites the moisture diffusivity is usually only weakly related to relative humidity and is often assumed to be a function only of temperature, usually following an Arrhenius-type exponential relation with inverse absolute temperature. For many of these materials, moisture equilibrium content is only weakly related to temperature and is usually assumed to be a function only of relative humidity. (1)

5.5 Vapor-exposure testing shall be used to condition the specimen when the in-service environmental condition is a vapor such as humid air. Immersion in a liquid bath should be used to simulate vapor exposure only when *apparent* absorption properties are desired for qualitative purposes. Properties determined in the latter manner shall be reported as *apparent* properties.

Note 2-For many polymer matrix composites the moisture absorption



properties under atmospheric humid conditions are generally not equivalent to exposure either to liquid immersion or to pressurized steam. These latter environments may have different material diffusion characteristics.

#### 6. Interferences

6.1 The calculation of the through-the-thickness moisture diffusivity constant in Procedure A assumes a single-phase Fickian material that possess constant moisture diffusivity properties through the thickness of the specimen. The validity of Procedure A for evaluating the moisture diffusivity constant in a material of previously unknown moisture absorption behavior will be uncertain prior to the test, as the test results themselves determine whether the material follows the single-phase Fickian diffusion model. As discussed by Shirrell (2), previous investigators have shown that for diffusion to be rigorously classified as Fickian it must satisfy the following three conditions:

6.1.1 Both absorption and desorption curves must be essentially linear up to 60 % of  $M_m$ ;

6.1.2 Beyond the initially linear portion, both absorption and desorption curves must be concave to the abscissa axis until  $M_m$  is reached; and

6.1.3 For the same environmental exposures, absorption curves resulting from different specimen thicknesses of the same material must be essentially super-imposable if each curve is plotted in the form of a normalized sorption curve in which the abscissa is  $\sqrt{time/h}$ , instead of  $\sqrt{time}$ .

6.2 These conditions are rigorous, and may not be fully met by many engineering materials. In fact these conditions may be difficult to experimentally verify for certain materials, and for this reason the decision on how rigorously these conditions must be met for a given test material is left to the user. For example, a severe complete absorption/desorption cycle may cause damage to a given material, causing cracking and providing a non-Fickian diffusion path, making evaluation of these conditions impossible.

6.3 However, there are a number of material forms or test conditions that are known to have the potential to violate one or more of the assumptions used by this test method, or to potentially cause non-Fickian material behavior. Many of these issues are discussed by several of the papers in ASTM STP 658 (3) or in the paper by Blikstad et al (4). They include:

6.3.1 Materials with fibers that are distributed in three dimensions, and that affect the moisture diffusion mechanism by means such as wicking along the fiber/matrix interface, such as: materials stitched through-the-thickness, or, some injection-molded materials;

6.3.2 Materials with a significant amount of surface cracking;

6.3.3 Material systems that have been shown to behave in a non-Fickian manner, or that have multi-phase moisture diffusion mechanisms as discussed by Bonniau and Bunsell (5);

6.3.4 Material systems that are known to have a moisture diffusivity constant that is strongly dependent upon moisture concentration;

6.3.5 Material systems that are known to have a moisture diffusivity constant that is significantly time-dependent;

6.3.6 Material systems that are known to have a moisture diffusivity constant that is significantly stress-dependent, and that are used in a laminate containing significant amounts of residual stress;

6.3.7 Material systems containing an abnormal amount of voids (porosity), or with a non-uniform void distribution, as discussed by Harper et al (6); Moisture (in liquid or gaseous form) can fill the voids resulting in an incorrect measurement of the amount of moisture absorbed into the material, and can result in removal of specimens from the conditioning environment at a premature time;

6.3.8 Test apparatus that produces a significant temperature gradient in the specimen, either through-the-thickness or inplane, as the material moisture diffusion constant is generally a strong function of temperature; or

6.3.9 Testing above the glass transition temperature of any constituent or phase-component within the composite material, which generally results in non-Fickian or multiphase moisture diffusion behavior, or both;

6.3.10 Materials that lose mass during moisture conditioning (e.g., materials that have water or fluid soluble components);

6.3.11 Materials that advance their cure state during exposure to water or water vapor (e.g., some phenolic resins).

6.4 For materials with a very high moisture diffusion rate, a low moisture equilibrium content, or both, the time scales required for periodic weighings may be very short (on the order of minutes or hours) and extreme care must be taken with the mass measurements and with the calibration and control of the environmental chamber, in order to avoid large measurement errors. The most convenient solution for reducing the test sensitivity of these materials is to simply increase the thickness of the test specimen.

6.5 The definition of effective equilibrium allows for a small continual increase in moisture absorption at test termination. Consequently the moisture content of the conditioned coupons may not be truly representative of the effects of long term exposure. Examples of materials that may exhibit significant continual moisture uptake after effective equilibrium include those with excessive porosity, concentrated resin pockets, and wrinkled fibers. If prolonged exposure is a concern, conditioning to a real world exposure lifetime is recommended, or at least moisture uptake should be interrogated in a timeframe significantly after reaching effective equilibrium, as defined in this standard.

# 7. Apparatus

7.1 *Balance*—An analytical balance is required that shall be capable of the appropriate accuracy shown in Table 1.

Note 3—These accuracy requirements are derived from the definition of effective moisture equilibrium (Eq 6) and the specimen mass of , as discussed in X2.10.

**TABLE 1 Balance Accuracy** 

Specimen Mass, g	Balance Accuracy Requirement, mg
≥5 but <50	0.1
≥50	1.0

7.2 Oven or Vacuum Drying Chamber—An air-circulating oven is required that shall be capable of maintaining the required temperatures to within  $\pm 3^{\circ}$ C [ $\pm 5^{\circ}$ F]. A vacuum drying chamber or a vacuum oven may also be used.

7.3 Conditioning Chamber—A conditioning chamber is required that shall be capable of maintaining the required temperature to within  $\pm 3^{\circ}$ C [ $\pm 5^{\circ}$ F]. The chamber shall be monitored either on an automated continuous basis or on a manual basis at regular intervals. The chamber shall consist of either of the following:

7.3.1 For Absorption by Vapor Exposure—A temperature and vapor-level controlled vapor exposure chamber that is capable of maintaining the required relative vapor level to within  $\pm 3$  %, or

# 7.3.2 *For Absorption by Liquid Immersion*—A temperature-controlled liquid bath.

Note 4—While many newer models have solid-state controls, a great many environmental chambers control the chamber humidity via monitoring of "dry-bulb" (actual) and "wet-bulb" (moisture depressed) temperatures, which are converted to equivalent relative humidity via a table or algorithm supplied by the manufacturer. The ability of these chambers to control relative humidity is dependent on the accuracy of the thermometer readings. Particularly important in these chambers is regular cleaning of the water reservoir, replacement of the wick, and maintenance of a proper contact between the wick and the wet-bulb thermometer. Chambers that control the dry-bulb temperature and the *differential* between the dry-bulb and wet-bulb temperatures generally have improved control of chamber relative humidity over those that control the dry-bulb and wet-bulb temperatures.

NOTE 5—Since loss of the water supply to a humidity chamber results in the conditioning environment changing to a drying condition with often a significant delay to the desired moisture conditioning, the use of a real time power and water supply monitoring and alarm system for the chamber is recommended. The alarm system should be able to contact off-site lab personnel in the event of an anomaly. A further recommendation is to use a system to shut off the chamber heat when a water supply or humidity fault is detected.

7.4 *Micrometers*—A 5 mm [0.2 in.] nominal diameter double-ball micrometer, accurate to 0.1 % of the specimen thickness, shall be used to measure the thickness of the specimen. For typical coupon dimensions an accuracy of  $\pm 2.5$  µm [ $\pm 0.0001$  in.] is adequate. A 5 mm [0.2 in.] nominal diameter flat-flat micrometer or caliper, accurate to within  $\pm 25$  µm [ $\pm 0.001$  in.], shall be used to measure the length and width of the specimen.

7.5 *Desiccator*—A clean, dry desiccator in which specimens being oven-dried shall be brought to laboratory temperature following removal of the specimens from the oven.

7.6 Specimen Bag—A sealable, flexible, moisture-proof bag (or other suitable sealable container) made of material suitable for exposure to specimens that have been removed from the conditioning chamber for cooling prior to weighing. Bags that meet the requirements of MIL-B-131 have been found satisfactory for use in standard applications.

7.7 *Absorbent Cloth*—Clean, non-linting absorbent cloth for use in wiping exuded or condensed moisture from test specimens.

7.8 *Gloves*—Clean, non-linting gloves for use when handling specimens.

# 8. Test Specimen

# 8.1 Sampling of Test Specimens:

8.1.1 *Procedure A*—test a minimum of one "thin" coupon and one "thick" coupon. Testing of three (3) replicates of each coupon thickness is recommended when sufficient material is available.

8.1.2 Procedures B-E—when a group of test specimens of the same material and thickness for subsequent material evaluation are to be conditioned at the same time, and periodic weighing of the specimens is required by the procedure, a minimum of three (3) specimens or three traveler coupons from the group shall be weighed. See for a discussion of when traveler coupons should be used. If the group of test specimens are not "dried" prior to moisture conditioning, the initial moisture content,  $M_b$ , of the material prior to conditioning may be determined by either (a) drying the traveler coupons using Procedure D after moisture conditioning has been completed, (b) drying using Procedure D an additional three traveler coupons, or (c) if the material diffusivity constant and effective moisture equilibrium content are known, by using the procedures in Appendix X3.

Note 6—In some cases the "dry" weight prior to moisture conditioning to equilibrium may not be equal to the "dry" weight when subsequently dried after moisture conditioning (see Section 6) in which case (a) and (b) above may not be equivalent. If this situation is suspected and determination of an accurate initial moisture content is required, then tests using both approaches are recommended.

8.1.3 *Procedure Y*—In cases where specimens are required to be removed from the conditioning environment prior to testing (such as the case where elevated temperature testing requires the use of high temperature cures (190° C [375°F]) for the strain gage adhesive and the strain gages are bonded after moisture conditioning), the loss of moisture during the time out of the conditioning environment should be quantified by a minimum of three (3) "moisture loss" specimens that represent each material and laminate thickness. Mechanical test specimens should be re-conditioned for the re-conditioning time duration determined by the "moisture loss" specimens.

8.1.4 *Procedure Z*—The moisture loss during testing at elevated temperatures should be quantified by a minimum of three (3) "moisture loss" specimens that represent each: material, laminate thickness, hot/wet specimen geometry (width and length), and test temperature. Separate moisture loss specimens shall be used for fixtured and non-fixtured specimens with the same dimensions. Moisture loss specimens are not required to be tabbed or notched, even if the specimens they represent are tabbed or notched. When simulating tabbed specimens, the moisture loss specimens may be gripped using un-bonded, simulated tabs to prevent specimen damage and mimic heat transfer through tabs into grips.

# 8.2 Test Specimen Geometry:

8.2.1 *Summary*—The following requirements are summarized in Table 2 for ease of reference.

8.2.2 Specimen Mass Requirement—Specimens (including travelers) shall have a mass of at least 5.0 g (see X2.10). Specimen mass affects the balance accuracy requirement established in 7.1.