



# Standard Test 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. 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 two-phase 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.

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 high-modulus 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,  $[Θ]$  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:

$$M, \% = \frac{W_i - W_o}{W_o} \times 100 \quad (1)$$

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,  $T_g$  [Θ]*, *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,  $D_z$  [ $L^2T^{-1}$ ]*, *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>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<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  $23 \pm 2^\circ\text{C}$  [ $73.4 \pm 3.6^\circ\text{F}$ ] 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-the-thickness 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;

$M_{ev}$ , 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;

$W_i$ , 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 Mm;
6.1.2 Beyond the initially linear portion, both absorption and desorption curves must be concave to the abscissa axis until Mm 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 in-plane, 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

Table with 2 columns: Specimen Mass, g and Balance Accuracy Requirement, mg. Rows include values for mass ranges like >=5 but <50 and >=50.



**7.2 Oven or Vacuum Drying Chamber**—An air-circulating oven is required that shall be capable of maintaining the required temperatures to within  $\pm 3^{\circ}\text{C}$  [ $\pm 5^{\circ}\text{F}$ ]. 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  $\pm 3^{\circ}\text{C}$  [ $\pm 5^{\circ}\text{F}$ ]. 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 and Calipers**—A micrometer with a 4 to 7 mm [0.16 to 0.28 in.] nominal diameter ball interface shall be used to measure the specimen thickness when at least one surface is irregular (such as the bag-side of a laminate). A micrometer with a 4 to 7 mm [0.16 to 0.28 in.] nominal diameter ball interface or with a flat anvil interface shall be used to measure the specimen thickness when both surfaces are smooth (such as tooled surfaces). A micrometer or caliper, with a flat anvil interface, shall be used to measure the width and length of the specimen. The accuracy of the instruments shall be suitable for reading to within 1% of the sample dimensions. For typical specimen geometries, an instrument with an accuracy of  $\pm 0.0025$  mm [ $\pm 0.0001$  in.] is adequate for the thickness measurement, while an instrument with an accuracy of  $\pm 0.025$  mm [ $\pm 0.001$  in.] is adequate for the width and length measurements.

**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 ( $190^{\circ}\text{C}$  [ $375^{\circ}\text{F}$ ])) 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**.

8.2.3 *Specimen Thickness*—The specimen thickness shall not vary by more than  $\pm 5\%$  over the surface of the specimen and shall comply with and **Eq 2**, as applicable.

**NOTE 7**—No minimum specimen thickness is required. However, the specimen thickness has a profound effect on the total time required to reach equilibrium, as well as on the reference time period of . Also, when designing a coupon, consideration should be given to the macrostructure of the composite material in the through-the-thickness direction. If the moisture absorption coupon is substantially thinner than the material in the end-use form, there should be sufficient number of fibers through the thickness that the absorption properties of the material coupon can be considered equivalent to the absorption properties of the same material in a much thicker form, under the same environmental conditions. A thin (even one-ply) laminate, if reinforced by a significant number of fibers that are small in diameter relative to the thickness, could be acceptable if it met the other specimen geometry requirements of . However, a substantially thicker multi-ply laminate, reinforced by fibers having a diameter on the order of the ply thickness, may not be acceptable. A minimum laminate thickness of 10 times the nominal fiber diameter is recommended.

### 8.2.4 Specimen Size and Shape For Use With Procedure A:

8.2.4.1 *Thin Specimen*—Since the purpose of the "thin" test specimen is only for moisture equilibrium content determination, there is no size requirement other than the minimum mass in accordance with **8.2.2**.

8.2.4.2 *Thick Specimen*—As edge effects must be minimized in materials with anisotropic moisture diffusivity constants in order to accurately determine the through-thickness diffusion constant, the "thick" test specimen shall consist of either (1) or (2) below. The "thick" test specimen for moisture diffusivity constant determination shall be thick enough such that achieving a moisture content of approximately 70 % of the

moisture equilibrium content for the same exposure conditions shall require a minimum of 35 days of exposure (if less time is required then a repeat of the test with a thicker specimen is recommended).(1) A nominally square plate or curved panel with dimensions that satisfy the relation:

$$\frac{w}{h} \geq 100 \quad (2)$$

where:

$w$  = nominal length of one side, mm [in.], and  
 $h$  = nominal thickness, mm [in.].

(2) A  $100 \pm 10$ -mm [ $4.0 \pm 0.5$ -in.] square plate with stainless steel foil bonded to the edges such that moisture absorption through the edges is essentially eliminated. When this specimen is prepared care shall be taken to weigh the coupon both before and after bonding of the foil in order to obtain the mass increase due to the foil and the adhesive. The adhesive used shall be incapable of absorbing sufficient moisture to affect the results.

**NOTE 8**—A typical "thin" specimen for the carbon/epoxy material described in **X2.2** having dimensions of 1 mm [0.040 in.] thick by 100 mm [4 in.] square would have a mass of approximately 18 g. If conditioned at 74°C [170°F] and 90 % relative humidity, use of Eq X1.7 (using an accuracy criteria of 0.02 % and a maximum difference between  $M_m$  and  $M_f$  of 0.02 %) predicts that this specimen would reach equilibrium in approximately 57 days. The minimum reference time period used to test equilibrium is established by as the greater of  $0.04 h^2/D_z$  (equal to 44 h) or 24 h; rounding to a practical even time gives a reference period of 2 days. A typical "thick" specimen for the carbon/epoxy material described in **X2.2** may have dimensions of 3.0 mm [0.120 in.] thick by 100 mm [4 in.] square with sealed edges, satisfying (2). If conditioned at 74°C [170°F] and 90 % relative humidity, use of Eq X1.7 (using the same criteria) predicts that this specimen would reach 70 % of the effective moisture equilibrium in approximately 38 days.

8.2.5 *Specimen Size and Shape For Use With Procedures B-Z*—The specimen size and shape requirement for Procedure A (**8.2.4**) is not a requirement for the conditioning procedures

**TABLE 2 Summary of Specimen Geometry Requirements**

Property Testing Procedure A—Thick Specimen	Conditioning Procedures B-Z
<p><i>Known Reference Time Period:</i></p> <ol style="list-style-type: none"> <li>Determine the maximum thickness from <math>h = \sqrt{D_z t / 0.04}</math>. If a thicker specimen is required then the reference time period must be changed. This equation is valid for the 0.02% moisture change criteria in <b>10.2</b>; if a different value for effective equilibrium moisture change is used, see <b>10.1.8</b> and <b>Appendix X2</b>.</li> <li>Determine the plate size from either <b>Eq 2</b> (non-sealed edges) or (2) (sealed edges).</li> <li>Estimate the specimen mass from <math>Mass = w^2 \times h \times density</math>. The specimen mass shall be <math>\geq 5</math> g. If the estimated mass is too small then the plate size must be increased (or the specimen made thicker and the reference time period correspondingly increased).</li> </ol> <p><i>Known Specimen Thickness:</i></p> <ol style="list-style-type: none"> <li>Determine the reference time period from . If the value so determined is unacceptable then the specimen thickness must be changed.</li> <li>Determine the plate size from either <b>Eq 2</b> (non-sealed edges) or (2) (sealed edges).</li> <li>Estimate the specimen mass from <math>Mass = w^2 \times h \times density</math>. The specimen mass shall be <math>\geq 5</math> g. If the estimated mass is too small then the plate size must be increased (or the specimen made thicker and the reference time period correspondingly increased).</li> </ol>	<p><i>Known Specimen Thickness:</i></p> <ol style="list-style-type: none"> <li>Determine the reference time period from . If this is unacceptable then the specimen thickness must be changed.</li> <li>Estimate the specimen mass from the material density, known thickness, and the configuration. Specimen mass shall be <math>\geq 5</math> g. If the estimated mass is too small, or the specimen has attached tabs or other features that violate the assumptions of this test method, then a traveler coupon must be used.</li> </ol>

(B-Z) of this test method. The coupon size and shape used in Procedures B-Z is normally that required for subsequent material evaluation following conditioning, as long as the coupon meets the mass and thickness requirements of 8.2.2 and 8.2.3. When the coupon is of such type or geometry that the moisture change in the material cannot be properly measured by weighing the specimen itself, *traveler* coupon(s) of the same material and thickness, and of appropriate size (but without tabs, if present) shall be used to determine moisture equilibrium for the specimens being conditioned. Material evaluation tests that require traveler moisture conditioning coupons include mechanical tests using tabbed coupons, specimens containing fasteners, specimens with strain gages, and test methods using coupons that do not meet the minimum mass requirement for this test method, such as commonly used in thermo-mechanical analysis methods.

8.2.6 *Preparation*—In specimens cut from plates precautions shall be taken to avoid notches, undercuts, rough or uneven surfaces, or delaminations due to inappropriate machining methods. Final dimensions should be obtained by water lubricated precision milling or grinding, or both, or the use of a wet diamond saw. The procedure used shall be reported.

8.2.7 *Labeling*—Label the specimen coupons so as to be distinct from each other in a manner that will both be unaffected by the test and not influence the test. The coupon must be weighed prior to adding the label (see 10.3.5) when using a marking method that adds mass to the coupon. Examples of various marking methods include coded wire overwrap and edge marking.

NOTE 9—A coded-wire overwrap is a wire, of a material (such as stainless steel) that is substantially unaffected by the pending environmental exposure. The wire is wrapped around the specimen tightly enough to prevent loss of the wire, but loosely enough that it does not interfere with the absorption process. The wire is coded by various means, including but not limited to tags, notches, knots, or color.

## 9. Calibration of Apparatus

9.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

**TABLE 3 Procedure B Sub-Procedure Codes for Common Conditioning Methods**

Procedure Code	Conditioning Method
Procedure BHEP	Non-ambient moisture conditioning in a humidity chamber at a prescribed constant conditioning environment to equilibrium; periodic coupon weighing is required. The default conditioning environment is 70°C [160°F], 85 % RH.
Procedure E (BHVP + BHEP)	Non-ambient moisture conditioning in a humidity chamber for two prescribed constant conditioning environment steps; in step one the conditioning is continued until a specified percentage of equilibrium is reached, then the step conditioning is continued to equilibrium; periodic coupon weighing is required. The default conditioning environment is 70°C [160°F], 95 % RH for step 1 and 70°C [160°F], 85 % RH for step 2.
Procedure BHEE	Non-ambient moisture conditioning in a humidity chamber at a prescribed constant conditioning environment to equilibrium; periodic coupon weighing is not required. The default conditioning environment is 70°C [160°F], 85 % RH.
Procedure BWEP	Non-ambient moisture conditioning in a water immersion tank at a prescribed constant temperature to equilibrium; periodic coupon weighing is required. The default conditioning environment is 70°C [160°F].
Procedure BWFF	Non-ambient moisture conditioning in a water immersion tank at a prescribed constant temperature for a fixed time period. The default conditioning environment is 70°C [160°F].
Procedure BBFF	Non-ambient moisture conditioning in a boiling water immersion tank for a fixed time period.
Procedure BFEP	Non-ambient moisture conditioning in a fluid (other than water) immersion tank at a prescribed constant temperature to equilibrium; periodic coupon weighing is required.
Procedure BFFF	Non-ambient moisture conditioning in a fluid (other than water) immersion tank at a prescribed constant temperature for a fixed time period.
Procedure BGFF	Non-ambient conditioning in a chamber with a specified gas composition environment for a fixed time period.



## 10. Procedures

### 10.1 Parameters To Be Specified Prior To Test:

10.1.1 The procedure to be used (A-Z), the sub-procedure codes Bxxx when using Procedure B (listed in [Table 4](#)), the sequential Procedure B sub-procedure codes when using Procedure E, and whether the specimens shall be dried using Procedure D prior to moisture conditioning. Procedure B sub-procedure codes for some common conditioning methods are listed in [Table 3](#). If Procedure B is specified without a sub-procedure code, then conditioning shall be performed per code BHEE.

NOTE 10—If a drying step is included, whether as an initial step prior to moisture conditioning, or as part of an oven-dry experiment, care should be taken to avoid excessively high drying temperatures and high thermal excursions that may induce thermal cracking, oxidation, or mass loss, or combinations of the three in the material.

10.1.2 The density and fiber volume sampling method (required only for Procedure A), coupon geometry, and test method (see [Note 11](#) for guidance).

NOTE 11—For many polymer matrix composites the volume percent reinforcement can be determined by one of the matrix digestion procedures of Test Method [D3171](#), or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method [D2584](#). Test Methods [D2734](#) describes the limitations and use of the calculations required to approximate the void content in the composite. The void content equations of Test Methods [D2734](#) are applicable to both Test Methods [D2584](#) and [D3171](#). Test Methods [D792](#) can be used to determine specific gravity.

10.1.3 The moisture absorption specimen sampling method, type, and geometry (and travelers, if required), from [8.1](#).

10.1.4 The balance measurement accuracy, 1.0 mg or 0.1 mg, from [7.1](#).

10.1.5 Conditioning chamber test temperature (for both the initial drying step, if required, and for the moisture conditioning step(s)).

NOTE 12—Maximum test temperatures are recommended for all Procedures in [Table 5](#). Keep the test temperature for any material not listed in the table at least 25°C [45°F] below the wet  $T_g$  of the material.

10.1.6 Moisture type (water vapor, liquid water, specified fluid, or specified gas) and moisture exposure level (in terms of relative humidity for water vapor exposure). For gas exposures, the gas components, concentrations, and the pressure level for the conditioning chamber.

NOTE 13—CMH-17 Rev G (1) notes that a worst-case aircraft service water vapor environment is generally considered to be 85 % relative humidity. Two-step accelerated conditioning schemes consisting of 95 to 98 % relative humidity exposure for a period of time followed by additional exposure time at a lower relative humidity level can be used to reduce the conditioning time, as long as equilibrium is reached at the final humidity level. References (7) and (8) discuss methods of accelerated

**TABLE 4 Procedure B Sub-Procedure Codes:  
B(Env)(Time)(Monitoring)**

Environment	Time	Monitoring
H-Humidity	E-To Effective Equilibrium	E-Initial+3 Final Weighings
W-Water	F-Fixed	F-Initial + Final Weighings
B-Boiling water	V-Variable	P-Periodic Weighings
F-Fluid other than water		
G-Gas		

**TABLE 5 Maximum Recommended Test Temperatures**

Polymer Matrix Material	Maximum Recommended Test Temperature, °C [°F]
121°C [250°F] cure epoxies	70 [158]
177°C [350°F] cure epoxies	80 [176]
Other	25 [45] less than wet $T_g$

conditioning. Also note that exposure to liquid immersion is not generally equivalent to exposure to an environment of 100 % relative humidity.

10.1.7 Time interval between weighings. Procedure A requires repeated weighings for the determination of the diffusivity constant, which shall be performed within +/- 1 % of the nominal time interval. Procedure BxxP requires periodic weighings at specified intervals. Procedures BxxE, BxxP, C, D, Y, and Z require a final set of three weighings spaced apart by the reference time period. Procedure BxxF only requires a final weighing.

10.1.7.1 Determine the reference time period from [Table 6](#). If the moisture diffusivity constant,  $D_z$ , is known, or if a reasonable value for  $D_z$  can be assumed from past experience, previous testing, data from the literature, or similarity to other known materials, then the minimum length of the reference time period shall be established by use of the YES value from [Table 6](#). If  $D_z$  is unknown then the NO value from [Table 6](#) shall be used. If this required reference time period does not match a reasonable human time schedule for weighing, then a more regular time interval may be adopted and the mass change requirement (less than 0.02 % mass change over the reference time period in accordance with [10.2](#)) shall be prorated to the adjusted time interval. For example, if a required reference time period for equilibrium is determined to be 115 000 s (32 h), the coupons may be weighed at either 24-h intervals or 48-h intervals, with the mass change requirement adjusted from 0.020 % to either 0.015 % ( $24/32 \times 0.020$ ) or 0.030 % ( $48/32 \times 0.020$ ), respectively.

10.1.8 The average moisture content change percentage for effective equilibrium if different from the standard value of 0.020 % in [10.2](#). If a different value is specified, then the coefficient in the first row of [Table 2](#) and [Table 6](#) must be recalculated in accordance with [X2.4 – X2.7](#).

10.1.9 For Procedure Y, the environmental conditions for the specimen when out of the conditioning chamber (in the case of strain gage bonding specify the room temperature hold time, elevated temperature cure time, and the strain gage cure temperature). Also specify one of Procedures Bxxx, C, or E as the preconditioning step.

**TABLE 6 Establishing the Reference Time Period**

Is $D_z$ Known?	Reference Time Period
Yes	( $0.04 \cdot h^2/D_z$ ) seconds the greater of { or 86 400 seconds (1 day)
	where: $D_z$ = through-the-thickness moisture diffusivity constant, $\text{mm}^2/\text{s}$ [ $\text{in.}^2/\text{s}$ ] $h$ = thickness, mm [ $\text{in.}$ ]
No	604 000 seconds (7 days)



10.1.10 For Procedure Z, the ramp time, soak time, test duration time, and the test temperature for the elevated temperature mechanical property tests. Also specify one of Procedures Bxxx, C, or E as the preconditioning step.

10.2 *Definition of Effective Moisture Equilibrium*—For the purposes of this test method, if not otherwise specified by the test requestor, a material shall be defined to be in a state of effective moisture equilibrium when the average moisture content of the material changes by less than 0.020 % over each of two consecutive reference time period spans, AND examination of the moisture content versus  $\sqrt{\text{time}}$  plot supports the percent change criteria that effective equilibrium is reached (see Note 25). This criteria for change in moisture content over a reference time period can be expressed in several equivalent ways; four equivalent expressions are shown below:

$$|M_i - M_{i-1}| < 0.020\% \quad (3)$$

or

$$\left| \frac{W_i - W_b}{W_b} - \frac{W_{i-1} - W_b}{W_b} \right| \times 100 < 0.020\% \quad (4)$$

or

$$\left| \frac{W_i - W_b}{W_b} - \frac{W_{i-1} - W_b}{W_b} \right| < 0.00020 \quad (5)$$

or

$$\left| \frac{W_i - W_{i-1}}{W_b} \right| < 0.00020 \quad (6)$$

where:

- $M$  = specimen average moisture content, %,
- $W$  = specimen mass, g, and the subscripts indicate:
- $i$  = value at current time,
- $i - 1$  = value at previous time, and
- $b$  = value at baseline time.

NOTE 14—The above criterion has been found to provide acceptable conditioning for many polymer composite materials which have moisture equilibrium values of at least 0.5 %. For materials with lower moisture equilibrium values, a tighter moisture content change criteria may be necessary, along with more accurate mass measurements, to obtain accurate and repeatable conditioning.

### 10.3 General Instructions:

10.3.1 Report any inadvertent deviations from the procedure.

10.3.2 Shield the balance from air drafts and isolate it from vibrations that could affect its accuracy.

10.3.3 Wear clean, non-linting gloves when handling specimens.

10.3.4 Expose both surfaces of the specimen to the test environment. Specimens shall be individually spaced apart to provide adequate air or vapor circulation in drying or vapor exposure environments, and adequate fluid circulation in an immersion bath.

10.3.5 Remember to subtract the mass of any additional material such as wire overwrap label or edge sealant from the mass of the specimen at each time interval weighing.

### 10.4 Procedure A—Moisture Absorption Property Determination:

#### 10.4.1 Specimen Preconditioning:

10.4.1.1 Perform Procedure D as a preconditioning step on the "thin" coupons to be used to determine the moisture equilibrium content. Drying of the "thick" coupons to be used to determine the moisture diffusivity constant is not required; however, if the specimen is suspected to have significant initial moisture, then drying is recommended.

10.4.1.2 Perform and report representative material density and fiber volume tests.

10.4.1.3 Measure the specimen thickness in a minimum of three locations and report the average measurement with an accuracy of 1 % of the thickness or better. For typical coupon dimensions, a measurement to the nearest 0.01 mm [0.0005 in.] is adequate. Measure the nominal plate side dimensions to the nearest 1.0 mm [0.050 in.] and report these. Report as "dry" dimensions.

#### 10.4.2 Moisture Absorption:

10.4.2.1 Record the oven-dry "thin" specimen mass as the baseline mass,  $W_{bn}$ , for moisture absorption. Record the initial "thick" specimen mass,  $W_{bk}$ . Place the specimen in the conditioning chamber, which has previously reached the specified steady-state environment.

10.4.2.2 At the end of each specified weighing time interval (if not otherwise specified by the test requestor, the weighing time interval shall be 24 h), remove the specimens from the conditioning chamber, and place the specimens in the specimen bag. Close or seal the bag and allow the specimens to come to an acceptable temperature for laboratory handling. Remove each specimen from the bag (resealing the bag if other specimens remain within) and wipe the specimen free of surface moisture with an absorbent lint-free towel. Immediately weigh each specimen to the required precision. Record each measurement as  $W_i$ , along with the corresponding total elapsed time and the time interval since the previous weighing, and return the specimens to either the specimen bag or the conditioning chamber. The specimen shall not be out of the conditioning chamber for more than 30 min/weighing and shall not be out of the specimen bag for more than 5 min/weighing.

NOTE 15—Minimizing the specimen time out of the conditioning chamber, and especially time out of the specimen bag, is critical to getting a smooth and accurate moisture absorption curve. The maximum allowable times of 10.4.2.2 may need to be shortened for some specimen/material combinations, particularly early in the moisture absorption test.

10.4.2.3 Calculate and record the percent mass change at each time interval by Eq 7. For the "thin" specimen, monitor the absorption of moisture until effective moisture equilibrium is reached as determined by 10.2 using the reference time period from 10.1.7.1. Record the last value of percent mass change as the moisture equilibrium content,  $M_m$ . For the "thick" specimen, monitor the absorption of moisture until at least 70 % of the effective moisture equilibrium is reached.

NOTE 16—It is recommended to leave the Procedure A specimens in the conditioning chamber for a significantly longer period of time after reaching the effective moisture equilibrium in order to ensure that the material moisture absorption follows a single phase Fickian behavior and that the effective moisture equilibrium value is sufficiently close to the actual moisture equilibrium value.

10.4.2.4 Measure and report, as post-test observations, the thickness and plate side dimensions again. Report as "conditioned" dimensions.

10.4.3 *Moisture Desorption Following Absorption*—If the material is known to contain, or is suspected to contain, any appreciable amount of moisture-soluble ingredients, the "thin" specimen (following moisture absorption) shall be dried in an oven in accordance with Procedure D to a state of essentially moisture-free equilibrium. This may also be done using either specimen thickness for evaluation of the moisture desorption characteristics of the material.

10.4.3.1 If the post-test oven-dry equilibrium mass is lower than the preconditioned mass (as established in 10.4.1), the difference shall be considered soluble matter lost during the testing process, as calculated by Eq 9. For such materials, the effective moisture equilibrium content shall be calculated by adding the mass of the moisture-soluble matter ( $W_b - W_p$ ) to the mass of the coupon at establishment of effective moisture equilibrium from 10.4.2.3. This corrected moisture equilibrium mass shall be used in Eq 7 to calculate the percent mass change, and the resulting value shall be recorded as the moisture equilibrium content,  $M_e$ .

NOTE 17—If specimen mass loss due to solvent degradation or physical handling is detected, then the calculated moisture diffusion properties may be in error.

10.4.3.2 Measure and report, as post-test observations, the thickness and plate side dimensions again. Report as "postconditioned dry" dimensions.

#### 10.5 Procedure B—Non-Ambient Specimen Conditioning:

10.5.1 *Specimen Preconditioning*—Not required for sub-procedures BHxx, BWxx, BBxx but may be specified. Required for sub-procedures BFxx and BGxx. If specified or required, perform Procedure D as a preconditioning step.

NOTE 18—If this step is skipped then the increase in moisture measured during conditioning will, in general, not be equal to the effective moisture equilibrium content, due to the presence of initial moisture. 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 on additional traveler coupons, or (c) if the material diffusivity constant and effective moisture equilibrium content are known, by using the procedures in Appendix X3.

10.5.2 *Initial Weighing*—Record the initial specimen mass (after any preconditioning) as the baseline mass,  $W_b$ .

10.5.3 *Conditioning*—Place the specimen in one of the following conditioning environments.

10.5.3.1 *Procedure BHxx—Humidity Chamber Conditioning*—Place the specimen in the conditioning chamber, which has previously reached the specified steady-state environment. If the conditioning temperature is not specified, condition at 70°C [160°F]. If the conditioning relative humidity is not specified, condition at 85 % RH.

10.5.3.2 *Procedure BWxx—Water Immersion Conditioning*—Place the specimen in the water bath, which has previously reached the specified steady-state temperature. If the conditioning temperature is not specified, condition at 70°C [160°F].

10.5.3.3 *Procedure BBxx—Boiling Water Immersion Conditioning*—Place the specimen in the water bath, which has previously reached a steady-state boiling temperature. Record the temperature of the water.

10.5.3.4 *Procedure BFxx—Fluid Immersion Conditioning*—Place the specimen in the specified fluid bath, which has previously reached the specified steady-state temperature.

10.5.3.5 *Procedure BGxx—Gas Environment Conditioning*—Place the specimen in the chamber with the specified gas concentration and pressure.

10.5.4 *Weighing*—At the end of each specified weighing time interval, remove the specimens from the conditioning chamber or fluid bath and place the specimens in the specimen bag. Close or seal the bag and allow the specimens to come to an acceptable temperature for laboratory handling. Remove each specimen from the bag (resealing the bag if other specimens remain within) and wipe the specimen free of surface moisture with an absorbent lint-free towel. Immediately weigh each specimen to the required precision. Record each measurement as  $W_i$ , along with the corresponding total elapsed time and the time interval since the previous weighing, and return the specimens to either the specimen bag or the conditioning chamber or fluid bath. The specimen shall not be out of the conditioning chamber or fluid bath for more than 30 min/weighing and shall not be out of the specimen bag for more than 5 min/weighing.

10.5.5 *Mass Change*—Calculate and record the percent mass change at each time interval by Eq 7.

10.5.6 *Conditioning Time*—Condition the specimen for the time period corresponding to one of the following subprocedures:

10.5.6.1 *Procedure BxEx—Effective Equilibrium*—Monitor the absorption of moisture until effective moisture equilibrium is reached as determined by 10.2 using the reference time period from 10.1.7.1. Record the last value of percent mass change as the moisture equilibrium content,  $M_e$ . A minimum of four weighings are necessary to ensure effective moisture equilibrium; the pretest weighing, and three final weighings. However, additional conditioning time and weighings may be required to establish effective moisture equilibrium. Previous experience (such as a curve showing equilibrium time versus thickness), or analytical estimation using techniques such as those described in the Appendixes, together with data obtained from Procedure A, can provide an approximate total elapsed conditioning time and minimize the required number of weighings. Record the last value of percent mass change as the total moisture equilibrium content,  $M_{et}$ , if the specimens were preconditioned by drying, or as the delta moisture equilibrium content,  $M_{ed}$ , if the specimens were not preconditioned.

NOTE 19—While similar to Procedure F of Practices D618, this procedure requires quantified moisture equilibrium instead of the fixed conditioning time period used by Procedure F of Practices D618.

10.5.6.2 *Procedure BxFx—Fixed Time*—Condition for the specified fixed time period. Record the value of percent mass change as the final (non-equilibrium) moisture content,  $M_f$ .

10.5.6.3 *Procedure BxVx—Variable Time*—Monitor the absorption of moisture until reaching a specified moisture content



level. Record the value of percent mass change as the final (non-equilibrium) moisture content,  $M_f$ .

10.5.7 *Conditioning Monitoring*—Monitor the specimen mass change using one of the following sub-procedures:

10.5.7.1 *Procedure BxxE—Initial + Three Final Weighings*—After conditioning for the specific time or the time anticipated where effective equilibrium is reached, perform three weighings of the specimen spaced apart by the reference time period from 10.1.7.1. This sub-procedure is typically used with the Equilibrium conditioning time (BxEx) sub-procedure; if Effective Equilibrium in accordance with 10.2 is reached, then stop the specimen conditioning; if Effective Equilibrium is not reached, then continue conditioning and weighing at the reference time period intervals.

10.5.7.2 *Procedure BxxF—Initial + Final Weighings*—Perform a final specimen weighing after the specified conditioning time. This sub-procedure is typically used with the Fixed conditioning time (BxFx) sub-procedure.

10.5.7.3 *Procedure BxxP—Periodic Weighings*—Perform periodic weighing of the specimen at the specified time interval or reference time period from 10.1.7.1. This sub-procedure is typically used with the Equilibrium or Variable conditioning time (BxEx or BxVx) sub-procedures; continue conditioning and weighing at the specified or reference time period intervals until Effective Equilibrium (BxEx) or the specified moisture content (BxVx) is reached.

10.6 *Procedure C—Standard 50 % Humidity Chamber Specimen Conditioning:*

10.6.1 Initially weigh the specimen to the required precision and record this value as the baseline mass,  $W_b$ , for conditioning. Place the specimen in the conditioning chamber, which has previously reached a steady-state environment at the specified temperature and a relative humidity level of 50 %. If the conditioning temperature is not specified, condition at 70°C [160°F].

10.6.2 At the end of each specified weighing time interval, remove the specimen from the conditioning chamber and place the specimen in the specimen bag to cool to an acceptable temperature for laboratory handling. Remove the specimen from the bag (resealing the bag if other specimens remain within) and wipe the specimen free of surface moisture with an absorbent lint-free towel. Immediately weigh the specimen to the required precision. Record each measurement as  $W_i$ , along with the corresponding total elapsed time and the time interval since the previous weighing, and return the specimen to either the specimen bag or the conditioning chamber. The specimen shall not be out of the conditioning chamber for more than 30 min/weighing and shall not be out of the specimen bag for more than 5 min/weighing.

10.6.3 Calculate and record the percent mass change at each time interval by the use of Eq 7. Monitor the specimen mass until effective moisture equilibrium is reached as determined by 10.2. Record the last value of percent mass change as the moisture equilibrium content,  $M_{ed}$ . Periodic weighing of the specimen is not required. A minimum of four weighings are necessary to ensure effective moisture equilibrium; the pretest weighing, and three final weighings. However, additional

conditioning time and weighings may be required to establish effective moisture equilibrium.

10.6.4 Following establishment of moisture equilibrium, store the material at the standard laboratory atmosphere until required for subsequent material testing.

NOTE 20—While similar to Procedure A of Practices D618, this procedure requires quantified moisture equilibrium instead of the fixed conditioning time period used by Procedure A of Practices D618. This procedure also allows for elevated temperature conditioning to accelerate the process.

10.7 *Procedure D—Oven-Dry Specimen Conditioning:*

10.7.1 Initially weigh the specimen to the required precision and record this value as the baseline mass,  $W_b$ , for oven drying. Heat the oven to the specified drying temperature. Place the specimen in the oven, which is located, for the purpose of humidity control, in a facility that has a standard laboratory atmosphere.

NOTE 21—A vacuum drying chamber or a vacuum oven may be used in place of a standard oven. Such equipment can provide a substantial reduction in the time required to reach equilibrium.

10.7.2 At the end of each specified weighing time interval, remove the specimen from the oven and place the specimen in a desiccator to cool to laboratory temperature. After the specimen has cooled to an acceptable temperature for laboratory handling, remove the specimen from the desiccator and wipe the specimen free of surface moisture with an absorbent lint-free towel. Immediately weigh the specimen to the required precision. Record each measurement as  $W_i$ , along with the corresponding total elapsed time and the time interval since the previous weighing, and return the specimen to either the oven or the desiccator. The specimen shall not be out of the oven for a total of more than 30 min/weighing and shall not be out of the desiccator for a total of more than 5 min/weighing.

10.7.3 Calculate and record the percent mass change at each time interval by the use of Eq 7. Monitor the desorption of moisture until effective moisture equilibrium is reached as determined by 10.2. Calculate and record the original moisture content,  $M_b$ , of the material in accordance with Eq 8.

10.7.4 Following establishment of oven-dry equilibrium, cool the specimen in a desiccator and store in a desiccator until final use.

NOTE 22—While similar to Procedure B of Practices D618, this procedure requires quantified moisture equilibrium instead of the fixed conditioning time period used by Procedure B of Practices D618.

10.8 *Procedure E—Multiple Step Conditioning:*

10.8.1 *Specimen Preconditioning*—Not required but may be specified. If specified, perform Procedure D as a preconditioning step. See Note 18.

10.8.2 *Conditioning*—This procedure can be used to accelerate the specimen conditioning time.

10.8.2.1 *Step 1*—Perform conditioning using procedure BxVP and the first specified environment to the specified moisture content level. If the moisture content level is not specified, condition to 80 % +/- 5 % of the expected effective equilibrium for the final specified environment.

NOTE 23—In some situations the 80 % of equilibrium level may be too high leading to a long conditioning time in the subsequent step. Moisture diffusion and content data for each environment can be used in a moisture

gain simulation to determine the optimum conditioning time for the initial step.

10.8.2.2 *Step 2*—(Optional) Perform conditioning using procedure BxVP and the second specified environment to the second specified moisture content level. Repeat this step for any additional intermediate conditioning environments.

10.8.2.3 *Step N*—Perform conditioning using procedure BxEP using the final specified environment to effective equilibrium.

NOTE 24—While certain two-step, accelerated conditioning cycles can be considered acceptable, such as use of an initial high-humidity step (95+ % RH) to speed up moisture gain, followed by completion to equilibrium at a lower final humidity level (85 % RH), one must be careful not to select an accelerating environment that changes the material, alters the physics of diffusion, or both. Long exposures to high temperatures combined with moisture may alter the chemistry of the material. While an initial high relative humidity step is acceptable, the extreme cases of exposure to pressurized steam or immersion in hot/boiling water are *not* accepted methods of accelerating humidity absorption, as they have been found to produce different results from that of 100 % humidity.

10.9 *Procedure Y—Moisture Loss During Time Out of Conditioning Environment:*

10.9.1 *Specimen Preconditioning*—Perform the specified Procedure B, C or E as a preconditioning step.

10.9.2 *Conditioning*—Remove the "moisture loss" specimen from the conditioning chamber and place in the room temperature ambient environment (do not cover or place in bags). After the specified time at room temperature (such as simulating the time to prepare and strain gage a test specimen), place the specimen into an oven at the specified temperature (such as for strain gage adhesive cure) for the specified time. Remove the specimens from the oven and allow to cool to room temperature. Immediately weigh each specimen to the required precision. Record the measurement as  $W_i$ . Calculate and record the percent mass change (loss),  $M_L$ , relative to the equilibrium moisture content.

10.9.3 *Re-Conditioning*—Perform the specified Procedure B, C, or E as a re-conditioning step to return the "moisture loss" specimen to moisture equilibrium. Record the time required to return to effective equilibrium. This re-conditioning time should be used to re-condition mechanical test specimens which have been removed from the conditioning environment for such purposes as strain gage bonding.

10.10 *Procedure Z—Moisture Loss During Specimen Testing:*

10.10.1 *Specimen Preconditioning*—Perform the specified Procedure B, C, or E as a preconditioning step.

10.10.2 *Conditioning*—Remove the "moisture loss" specimen from the conditioning chamber, and place into the test machine. Set up the moisture loss specimen in the same way as the mechanical test specimens that it represents, including any fixtures if required. Heat the specimen and fixture using the same nominal ramp rate and soak time that will be applied to the test specimens followed by an additional specified hold time, after the soak, to mimic the duration of test loading. Once the ramp, soak, and hold are complete, return the moisture loss specimen to room temperature. Immediately weigh each specimen to the required precision. Record the measurement as  $W_i$ .

Calculate and record the percent mass change (loss),  $M_L$ , relative to the equilibrium moisture content.

11. Calculation

11.1 *Absorption/Desorption Rate*—For all procedures where periodic weighings are performed, plot and examine the percent mass change (as calculated by Eq 7) versus  $\sqrt{\text{time}}$  curve as illustrated by Fig. 1.

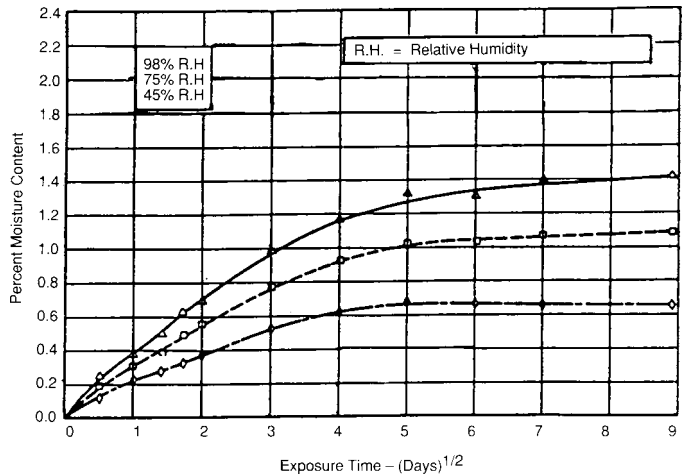


FIG. 1 Moisture Absorption of Carbon/Epoxy (0,45,-45,0)<sub>s</sub> Eight-Ply Laminates at 120°F (7)

NOTE 25—It is important to plot and examine the mass change versus  $\sqrt{\text{time}}$  curve prior to deciding that the specimen has reached effective moisture equilibrium. Often the mass change criteria in 10.2 can indicate reaching effective moisture equilibrium when the plot obviously shows that equilibrium has not been reached. Use of the plot to determine whether or not effective equilibrium has been reached requires engineering judgment and typically some knowledge of the material's moisture gain behavior; test labs should not make decisions about reaching effective moisture equilibrium without concurrence from the test requestor.

11.2 *Absorption/Desorption Percent Mass Change*—Calculate the percent change in mass for moisture absorption or desorption to three (3) decimal places as follows:

$$\Delta M = \text{Mass change, \%} = \left| \frac{W_i - W_b}{W_b} \right| \times 100 \quad (7)$$

where:

- $W_i$  = current specimen mass, g, and
- $W_b$  = baseline specimen mass, g.

11.3 *Initial (Baseline) Moisture Content*—Calculate the average moisture present in the material prior to conditioning to three (3) decimal places as follows:

$$M_b = \text{Initial Moisture Content, \%} = \frac{W_{ar} - W_{od}}{W_{od}} \times 100 \quad (8)$$

where:

- $W_{ar}$  = as-received specimen mass, g, and
- $W_{od}$  = oven-dry specimen mass, g.

11.4 *Percent Mass Lost*—Calculate the percent mass lost to three (3) decimal places, if determined, as follows:

$$\text{Mass lost, \%} = \frac{W_{ab} - W_p}{W_{ab}} \times 100 \quad (9)$$

where:

$W_{ab}$  = absorption baseline specimen mass, g, and  
 $W_p$  = post-desorption oven-dry specimen mass, g.

11.5 *Moisture Equilibrium Content*—For Procedure A, the last value of percent mass change from the "thin" specimen is the moisture equilibrium content,  $M_m$ .

11.6 *Diffusivity*—For Procedure A, calculate the diffusivity,  $D_z$ , as follows using the data from the "thick" specimen:

$$D_z = \pi \left( \frac{h}{4M_m} \right)^2 \left( \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2 \quad (10)$$

where:

$h$  = average specimen thickness in accordance with 10.3.6, mm [in.],  
 $M_m$  = effective moisture equilibrium content, %, and  
 $M_2 - M_1$  = slope of moisture absorption plot in the initial  
 $\sqrt{t_2} - \sqrt{t_1}$  = linear portion of the curve,  $\sqrt{\text{seconds}}^{-1}$ .

NOTE 26—Time units must be converted from those time units used during testing to those time units desired for the moisture diffusivity constant (for example, days to seconds).

NOTE 27—If there is no discernible initial linear portion of the moisture absorption versus  $\sqrt{\text{time}}$  plot, then the assumption of single-phase Fickian moisture absorption (and the calculated moisture diffusivity constant) may be invalid.

## 12. Report

12.1 Report the following information, or references pointing to documentation containing this information, to the maximum extent applicable:

12.1.1 The data of issue of this test method and the procedure and sub-procedure used.

12.1.2 The date(s) and location(s) of the test.

12.1.3 The name(s) of the test operator(s).

12.1.4 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.

12.1.5 Identification of the material tested, including: type, source, manufacturer's lot or batch number, previous history, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, polymer matrix type, precursor polymer content, and prepreg volatiles content.

12.1.6 Description of the fabrication method used in preparing the material including: cure cycle, consolidation method, and a description of the equipment used.

12.1.7 Ply orientation stacking sequence of the laminate.

12.1.8 For Procedure A, density, volume percent reinforcement, void content, and average ply thickness of the material.

12.1.9 Results of nondestructive evaluation tests.

12.1.10 Method of preparing the test specimen.

12.1.11 Calibration dates and methods for all measurement and test equipment.

12.1.12 Results of preconditioning.

12.1.13 For Procedure A, dimensions of the specimen following preconditioning prior to moisture conditioning and at the completion of the test procedure.

12.1.14 Type of test chamber used, the moisture used for the test, the nominal and actual test temperatures, moisture exposure levels, and the measurement time interval.

12.1.15 Relative humidity and temperature of the testing laboratory.

12.1.16 Specimen mass at each time interval.

12.1.17 Plots of percent mass change versus  $\sqrt{\text{time}}$ , calculated by Eq 7, with mass change reported to three (3) decimal places.

12.1.18 Material effective moisture equilibrium content, or final moisture content, or moisture loss (in mass percent) depending on the procedure used, with moisture content or loss reported to three (3) decimal places.

12.1.19 Moisture diffusivity constant, as calculated by Eq 10.

12.1.20 Percent of soluble matter lost calculated by Eq 9.

12.1.21 Any unusual test observations such as warping, cracking, or change of appearance, or combination thereof, of the test specimen.

12.1.22 Average, standard deviation, and coefficient of variation of the moisture properties for sample populations of three or more (having the same nominal reinforcement content and subjected to the same conditioning environment).

12.1.23 Moisture desorption data shall be reported in the same manner as the absorption data.

12.2 For material evaluation test specimens which are or are not conditioned in accordance with one of the procedures of this test method, the pretest specimen conditioning shall be reported as one of the following standard descriptions:

12.2.1 *As Fabricated and Desiccated*—A condition in which a specimen (including the material it is fabricated from) is stored in a desiccated container following material fabrication and between test specimen preparation steps, and for which the moisture content is assumed to be low, but has not been explicitly measured.

12.2.2 *Unknown Moisture Content*—If a controlled moisture content is not established during pretest conditioning, the pretest moisture content shall be reported as "unknown." This includes "as fabricated" or "ambient" condition specimens that have not been stored in a controlled low-humidity environment prior to test. This also includes fixed-time, non-equilibrium exposures in which the specimen weight change is not measured (the pretest conditioning environment and time of exposure shall be reported).

12.2.3 *Non-Equilibrium Moisture Content*—A condition of non-equilibrium moisture conditioning resulting in a specific reported moisture content, and established by a documented procedure that is reported in sufficient detail to allow duplication of the conditioning result by an independent party.

12.2.4 *Non-Equilibrium Moisture Profile*—A condition of non-equilibrium moisture conditioning resulting in a specific reported moisture content and a through-thickness moisture

profile determined analytically, and established by a documented procedure that is reported in sufficient detail to allow duplication of the conditioning result by an independent party.

12.2.5 *Effective Moisture Equilibrium*—Moisture content equilibrium of the specimen as established by this test method; the pretest moisture content shall be reported as moisture content at a stated pretest conditioning relative humidity (or immersion) and temperature.

12.2.6 The moisture content reported under 12.2.3 – 12.2.5 above shall be reported as the “total” moisture content,  $M_{et}$ , if the specimen is dried prior to conditioning, or shall be reported as “delta” moisture content,  $M_{ed}$ , if the initial moisture content prior to pretest conditioning is not determined. In the latter case, an analytically determined “initial” moisture content, in accordance with Appendix X3, may also be reported, along

with the “total” moisture content which shall be the sum of the “initial” and “delta” moisture contents.

### 13. Precision and Bias

13.1 *Precision*—The data required for the development of a precision statement are not available for this test method. Committee D30 is currently planning a round-robin test series for this test method in order to determine precision.

13.2 *Bias*—Bias cannot be determined for this test method as no acceptable reference standard exists.

### 14. Keywords

14.1 conditioning; moisture absorption; moisture desorption; moisture diffusivity; moisture equilibrium; polymer matrix composite materials

## APPENDIXES

### (Nonmandatory Information)

#### X1. PREDICTION OF MOISTURE CONTENT

X1.1 The prediction of moisture content in advanced composite materials is discussed in detail by Springer (9). The following equations (excerpted from the reference with minor notational changes) have several restrictions, but are useful for simple predictions of material behavior such as the design of material conditioning experiments. The restrictions and assumptions include:

X1.1.1 Must be a single-phase Fickian material, a material that behaves according to Fick’s law:

$$\frac{\partial c}{\partial t} = D_z \frac{\partial^2 c}{\partial z^2} \quad (\text{X1.1})$$

where:

- $c$  = specimen moisture concentration,  $\text{g}/\text{mm}^3$  [ $\text{lbm}/\text{in.}^3$ ],
- $t$  = time, s,
- $\partial c/\partial t$  = time rate of change of moisture concentration,  $\text{g}/\text{mm}^3/\text{s}$  [ $\text{lbm}/\text{in.}^3/\text{s}$ ],
- $D_z$  = Fickian material diffusion constant through-the-thickness of the material,  $\text{mm}^2/\text{s}$  [ $\text{in.}^2/\text{s}$ ], and
- $z$  = through-the-thickness direction, mm [in.].

X1.1.2 The boundary and initial conditions are:

$$c = c_i \quad 0 < z < h \quad t \leq 0 \quad (\text{X1.2})$$

$$c = c_a \quad x = 0; \quad x = h \quad t > 0 \quad (\text{X1.3})$$

X1.1.3 Temperature and moisture diffusion are uncoupled (there is generally a six-order-of-magnitude difference between the two differing diffusion speeds, which makes this a reasonable assumption);

X1.1.4 No variation in moisture diffusivity properties through the thickness of the material; and

X1.1.5 Limited to one-dimensional, steady-state problems.

X1.2 Given the above assumptions, the moisture content in the material for a given moisture exposure level is given as follows:

$$M(T, t) = M_b + G(T, t)(M_m - M_b) \quad (\text{X1.4})$$

where:

- $T$  = temperature, °C [°F],
- $M(T, t)$  = moisture content of material as a function of time and temperature, % of oven-dry mass,
- $M_b$  = baseline moisture content of material, % of oven-dry mass,
- $M_m$  = moisture equilibrium content of material, % of oven-dry mass,
- $h$  = thickness of material for double-sided exposure, mm [in.], and
- $G(T, t)$  = moisture absorption function (non-dimensional) and can be given as follows:

$$G(T, t) = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{\exp\left[-(2j+1)^2 \pi^2 \left(\frac{D_z(T)t}{h^2}\right)\right]}{(2j+1)^2} \quad (\text{X1.5})$$

X1.3 The function of Eq X1.5 may be approximated by:

$$G(T, t) = 1 - \exp\left[-7.3 \left(\frac{D_z(T)t}{h^2}\right)^{0.75}\right] \quad (\text{X1.6})$$

X1.4 The moisture equilibrium content,  $M_m$ , of a material is a function only of the ambient moisture exposure level (such as relative humidity). However, the speed at which this moisture equilibrium content is attained is a function of the ambient temperature, due to the temperature dependence of the moisture diffusivity constant. The time necessary to reach a given moisture content for fixed temperature and moisture exposure

level is (from Eq X1.6):

$$t = \frac{h^2}{D_z} \left[ \frac{-1}{7.3} \ln \left( 1 - \frac{M(t) - M_b}{M_m - M_b} \right) \right]^{4/3} \quad (\text{X1.7})$$

X1.5 Thus, the time necessary for a completely oven-dry specimen to reach 99.9 % of material moisture equilibrium at

a given temperature, regardless of the ambient moisture exposure level, is approximately:

$$t_{max}(T) = \frac{0.93h^2}{D_z(T)} \quad (\text{X1.8})$$

## X2. RATIONALE FOR DETERMINATION OF TEST METHOD PARAMETERS

X2.1 The principal test parameters for evaluation of the material moisture properties are tightly coupled. The values of these parameters selected by this test method are believed to produce acceptable results within the current range of applicable engineering materials. The rationale for their selection is described as follows by an illustration of the check of critical test parameters, using a common polymer matrix composite material. The parameters include the following:

### X2.1.1 Material:

X2.1.1.1 Speed of absorption (diffusivity), and

X2.1.1.2 Moisture equilibrium content.

### X2.1.2 Specimen Size:

X2.1.2.1 Aspect ratio,

X2.1.2.2 Thickness, and

X2.1.2.3 Mass.

### X2.1.3 Definition of Effective Equilibrium:

X2.1.3.1 Reference time period, and

X2.1.3.2 Accuracy criterion.

### X2.1.4 Equipment:

X2.1.4.1 Accuracy of balance.

### X2.1.5 Procedure:

X2.1.5.1 Weighing precision.

X2.2 A common material used in the aerospace industry uses oriented carbon filaments reinforcing an epoxy matrix. Springer (9) reports the diffusivity of a carbon/epoxy at 74°C [170°F] as  $D_z = 2.6 \times 10^{-7} \text{ mm}^2/\text{s}$  [ $4.0 \times 10^{-10} \text{ in.}^2/\text{s}$ ] and the

**TABLE X2.2 Specimen Aspect Ratio Versus Error**

w/h	$D_z/D_a$	Error, %
10	0.694	44.1
25	0.857	16.7
40	0.907	10.3
100	0.961	4.1
200	0.980	2.0
1000	0.998	0.2

moisture equilibrium content at 90 % relative humidity as 1.5 %. This material is used in the following examples to illustrate both the use of the moisture prediction equations and the rationale for the selection of test parameters required by this test method.

X2.3 A typical specimen thickness might be as great as 2.5 mm [0.1 in.], although a thinner specimen of approximately 1.0 mm [0.040 in.] will come to moisture equilibrium in a much shorter time. A much thicker specimen could take prohibitively long to reach moisture equilibrium.

X2.4 Effective moisture equilibrium is defined by specifying both an accuracy criterion and a reference time period, and it approaches absolute moisture equilibrium as the accuracy criterion is refined or the reference time period is extended. This test method fixes the accuracy criterion and allows the user to select a reference time period that varies depending upon the material and the specimen thickness being evaluated. As the detectable weight change between two consecutive

**TABLE X2.1 Predicted  $M_t$  Values for Various Moisture Absorption Parameters**

Thickness, mm	Time, h	$D_z t/h^2$	G	Accuracy Criterion, %		
				0.05 %	0.01 %	0.005 %
				Predicted $M_t$		
0.25	24	0.359	0.966 246	1.498	1.500	1.500
1.0	24	0.0224	0.345 303	1.405	1.481	1.491
1.5	24	0.010	0.205 919	1.307	1.461	1.481
2.0	24	0.0056	0.139 085	1.191	1.438	1.469
2.5	24	0.0036	0.101 617	1.058	1.412	1.456
25.0	24	0.000 036	0.032 258	0.000	0.000	0.000
0.25	48	0.719	0.996 651	1.500	1.500	1.500
1.0	48	0.0449	0.509 524	1.452	1.490	1.495
1.5	48	0.0200	0.321 431	1.394	1.479	1.489
2.0	48	0.0112	0.222 649	1.325	1.465	1.483
2.5	48	0.0072	0.164 913	1.247	1.449	1.475
25.0	48	0.000 072	0.032 258	0.000	0.000	0.000
0.25	168	2.51	0.999 999	1.500	1.500	1.500
1.0	168	0.157	0.838 442	1.490	1.498	1.499
1.5	168	0.0699	0.629 261	1.471	1.494	1.497
2.0	168	0.0393	0.475 070	1.445	1.489	1.494
2.5	168	0.0251	0.369 448	1.415	1.483	1.491
25.0	168	0.00025	0.032 258	0.000	0.000	0.000



weighings is on the order of 0.02 % (based upon the balance accuracy and the minimum specimen mass), **0.02 % is used as the accuracy criterion in the definition of effective moisture equilibrium in 10.2.**

X2.5 The coupon mass for the final two weighings of a moisture absorption experiment can be predicted using Eqs X1.4 and X1.6. The average moisture content at the final weighing is  $M_e$ , and the average moisture content at the previous weighing is  $M_{e-1}$ .  $M_e$  in general, differs from  $M_m$ , the absolute moisture equilibrium content, because the material is still absorbing moisture at the conclusion of the test. **By providing a suitable reference time period the difference between  $M_m$  and  $M_e$  (absolute tolerance) can be limited to any reasonable value. In this test method the difference is  $\leq 0.02$  %, or,  $M_m - M_e \leq 0.02$  %.**

X2.6 Redefining Eq X1.4 in terms of the final two weighings and  $M_m$  allows the calculation of a value of  $G(T,t)$  that satisfies the requirement for bounded error while using a given accuracy criterion:

$$M_e = M_{e-1} + G(T,t)(M_m - M_{e-1}) \quad (\text{X2.1})$$

or

$$\begin{aligned} G(T,t) &= \frac{M_e - M_{e-1}}{M_m - M_{e-1}} \\ &= \frac{M_e - M_{e-1}}{(M_m - M_e) + (M_e - M_{e-1})} \\ &= \frac{\text{accuracy}}{\text{absolute\_tolerance} + \text{accuracy}} \end{aligned} \quad (\text{X2.2})$$

or

$$G(T,t) = \frac{0.02}{0.02 + 0.02} = \frac{0.02}{0.04} = 0.50 \quad (\text{X2.3})$$

X2.7  $G(T,t)$  is the multiplier of the difference between the moisture content at the beginning of a time span and the moisture equilibrium content. Inserting  $G(T,t) = 0.50$  into Eq X1.6 yields a value of the parameter  $D_z t/h^2$  that satisfies these requirements. This value, of  $D_z t/h^2$ , 0.0433, produces a satisfactory effective equilibrium state (within the absolute tolerance, 0.02 % of the true equilibrium moisture content) for the accuracy criterion of 0.02 %. From  $D_z t/h^2$ , and knowing the moisture diffusivity constant and coupon thickness, a reference time period can be determined that ensures that  $M_e$  is within the absolute tolerance, 0.02 % of  $M_m$ . The parameter  $D_z t/h^2$  can also be used to guide the selection of specimen thickness for a specific, or more desirable, equilibrium reference time period. (Since this is an approximation, the coefficient 0.0433 was

rounded for convenience to 0.04 in 10.1.7.1 of the test method.)

X2.8 Insertion of the accuracy criterion (in percent) into Eq X1.4 results in the following equation relating  $M_e$  and  $M_m$ :

$$M_e = M_m + (\text{accuracy criterion}) \times \left(1 - \frac{1}{G}\right) \quad (\text{X2.4})$$

This equation was used to predict the effect of specimen thickness, reference time period, and accuracy criterion on a test series. The results, shown in Table X2.1, predict values of  $M_e$  for carbon/epoxy material at 74°C [170°F] and 90 % relative humidity for various specimen thicknesses, reference time periods, and accuracy criteria.

X2.9 Specimen size for moisture diffusivity property measurement on coupons without sealed edges is established from a 100:1 aspect ratio requirement for a square specimen. Rationale for the 100:1 aspect ratio comes from Springer's derivation (from 1-D theory) of correction factors accounting for moisture ingress through the edges of a rectangular specimen. While the correction factor is extremely complicated for laminated anisotropic materials, the point is illustrated by evaluating the width-to-thickness aspect ratio for a square specimen versus edge effect error for a nominally isotropic material. For this case the correction factor is:

$$D_z = D_a \times \frac{1}{\left(1 + \frac{2h}{w}\right)^2} \quad (\text{X2.5})$$

where  $D_a$  = apparent through-the-thickness diffusivity constant,  $\text{mm}^2/\text{s}$  [ $\text{in.}^2/\text{s}$ ], as measured by this test method. The resulting predicted percent error is tabulated versus specimen aspect ratio in Table X2.2. This table indicates that determination of diffusivity, within acceptable engineering accuracy on specimens not using sealed edges, requires an aspect ratio on the order of 100; thus the requirement.

X2.10 The minimum specimen mass requirement was established from the balance accuracy of  $\pm 0.1$  mg ( $\pm 0.0001$  g), the equilibrium accuracy criterion of 0.02 % (0.0002), and the desire to keep the balance about ten times more accurate than the required resolution. Therefore:

$$\text{Minimum specimen mass} = \frac{10 \times \text{balance accuracy}}{\text{accuracy criterion}} \quad (\text{X2.6})$$

$$= 5 \text{ g}$$

$$= \frac{10 \times 0.0001 \text{ g}}{0.0002}$$

### X3. PREDICTION OF BASELINE MOISTURE CONTENT

X3.1 The relative moisture content as a function of time from a given exposure condition can be estimated using the following equation (combining Eq X1.4 and Eq X1.6):

$$(M(T,t) - M_b) = (M_m - M_b) \left\{ 1 - \exp \left[ -7.3 \left( \frac{D_z(T)t}{h^2} \right)^{0.75} \right] \right\} \quad (X3.1)$$

X3.2 With an assumption of a baseline moisture content,  $M_b$ , and known values for the specimen thickness,  $h$ , the diffusion constant,  $D_z$ , and the moisture equilibrium value,  $M_m$ , a curve of moisture content versus time can be plotted. Using the constants listed in X2.2, a series of curves with different baseline moisture content values are shown in Fig. X3.1 for a

laminates thickness of 3.76 mm [0.148 in]. Some dummy test data for a specimen which was not preconditioned to the dry state is plotted in the Figure; interpolating between the curves, the baseline moisture content for this specimen can be estimated as 0.3 %.

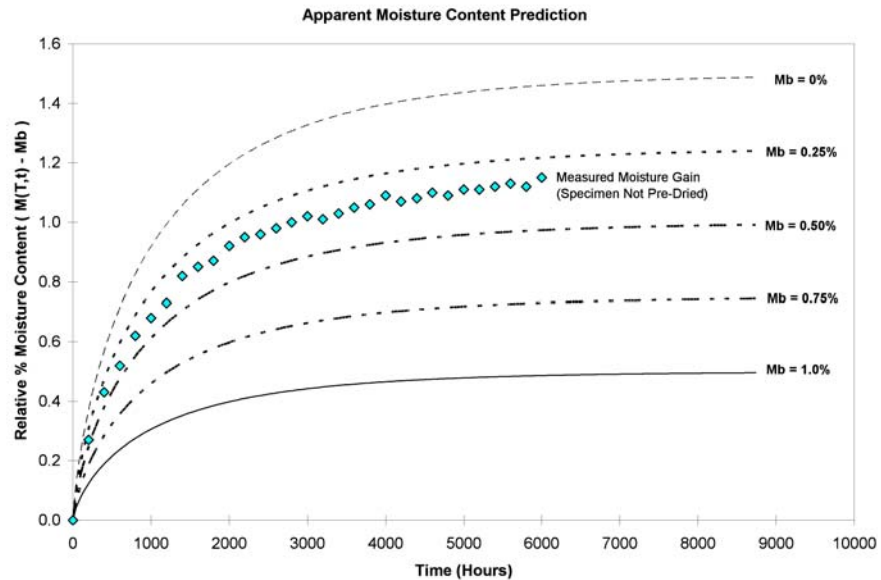


FIG. X3.1 Moisture Content Prediction for Various Levels of Baseline Moisture Content.

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