Designation: D 5229/D 5229M – 92 (Reapproved 2004)

Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[1]

This standard is issued under the fixed designation D 5229/D 5229M; 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 (ε) indicates an editorial change since the last revision or reapproval.

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

INTRODUCTION

Consistent evaluation and comparison of the response of polymer matrix composites to moisture absorption can only be performed when the material has been brought to a uniform through-the-thickness moisture profile. The procedures described in Test Method D 570 and Practices D 618 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 (Procedure A) 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 equilibrium in a non-laboratory environment (Procedure B), to equilibrium in a standard laboratory atmosphere environment (Procedure C), or to an essentially moisture-free state (Procedure D). 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 1.4.

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 MIL-HDBK-17B **(1)**,[2] 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. Within the text the inch-pound units are shown in brackets. 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.

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.*

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[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 570 Test Method for Water Absorption of Plastics
D 618 Practice for Conditioning Plastics for Testing
D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D 883 Terminology Relating to Plastics
D 2584 Test Method for Ignition Loss of Cured Reinforced Resins
D 2734 Test Methods for Void Content of Reinforced Plastics
D 3171 Test Method for Constituent Content of Composite Materials
D 3878 Terminology for Composite Materials

2.2 *Military Standard:*

MIL-B-131 Barrier Materials, Watervaporproof, Greaseproof, Flexible, Heat-Sealable[4]

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other terminology standards.

3.2 *Definitions of Terms Specific to This Standard*—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 nondimensional 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.

3.2.1 *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.*)

3.2.2 *average moisture content, M (%), n*—the average amount of absorbed moisture in a material, taken as the ratio of the mass of the moisture in the material to the mass of the oven-dry material and expressed as a percentage, as follows:

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

where:

W_i = current specimen mass, g, and
W_o = oven-dry specimen mass, g.

(See also *oven-dry*.)

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

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

3.2.4 *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.

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

3.2.5 *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.

3.2.5.1 *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.

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

3.2.7 *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.

3.2.7.1 *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.

3.2.8 *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.*)

3.2.8.1 *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 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.

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

[4] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

3.2.9 *moisture equilibrium content,* $M_{\mathrm{m}}(\%)$, *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.*)

3.2.9.1 *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.

3.2.10 *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.*)

3.2.11 *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.*)

3.2.12 *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.

3.2.13 *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.*)

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

3.2.14 *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.*)

3.2.15 *standard laboratory atmosphere*, *n*—an atmosphere (environment) having a temperature of 23 ± 2°C [73.4 ± 3.6°F] and a relative humidity of 50 ± 10 %.

3.2.16 *standard conditioned specimen*, *n*—the material condition of a test coupon that has reached effective moisture equilibrium at a nominal relative humidity of 50 % in accordance with Procedure C of this test method.

3.2.17 *test temperature*, *n*—the environmental temperature used in Procedures A, B, C, or D.

3.2.17.1 *Discussion*—This is distinguished, for the purposes of this test method, from the environmental temperature used during any subsequent material evaluation testing.

3.2.18 *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_f, the average moisture content at establishment of effective moisture equilibrium; M_i, the average moisture content at a given time; M_{i-1}, the average moisture content at the previous time; 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*—For the purposes of this test method M_m and M_f are assumed to be equivalent.

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 a coupon that is exposed on two sides to a specified environment. There are four test procedures described by this test method. Procedure A covers the determination of the two Fickian moisture diffusion material properties, the moisture diffusivity constant and the moisture equilibrium content. The other three procedures cover material conditioning to a specific moisture environment; Procedure B covers general moisture conditioning of material coupons prior to other types of testing; Procedure C covers the conditioning of material coupons to a nominal relative humidity level of 50 %, prior to other types of testing; and Procedure D covers the conditioning of material coupons to an essentially moisture-free condition.

4.2 In Procedure A, the percent moisture mass gain versus time is monitored for a material specimen that is maintained in a steady-state environment at a known temperature and moisture exposure level until the material reaches effective moisture equilibrium. From this data 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 a specified temperature and moisture exposure level until the material reaches effective moisture equilibrium. However, the periodic monitoring requirement of Procedure A is not necessary, and the data needed to calculate the moisture diffusivity constant is not obtained.

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.

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, C, or D;

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, C, and D are designed to condition test coupons to a specified moisture equilibrium state prior to other material property testing (including but not limited to mechanical testing).

5.3 A single test 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.

5.4 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 1—For many polymer matrix composites the volume percent reinforcement can be determined by one of the matrix digestion procedures of Test Method D 3171, or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method D 2584. Test Methods D 2734 describes the limitations and use of the calculations required to approximate the void content in the composite. The void content equations of Test Methods D 2734 are applicable to both Test Methods D 2584 and D 3171. Test Methods D 792 can be used to determine specific gravity.

6. Interferences

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

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

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

6.1.3 For the same environmental exposures, absorption curves resulting from different specimen thicknesses of the same material must be essentially superimposable 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, or with a nonuniform void distribution, as discussed by Harper et al **(6)**;

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.

7. Apparatus

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

NOTE 2—These accuracy requirements are derived from the definition of effective moisture equilibrium (Eq 6) and the specimen mass of 8.2.2, as discussed in X2.10.

7.2 *Oven or Vacuum Drying Chamber*—An air-circulating oven is required that shall be capable of maintaining the required uniform temperatures to within ±1°C [±2°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 ±1°C [±2°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 ±3 %, or

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

7.4 *Micrometers*—A 5-mm [0.2-in.] nominal diameter double-ball micrometer, accurate to 0.1 % of the specimen thickness, shall be used to measure the thickness of the specimen. For typical coupon dimensions an accuracy of ±2.5 µm [±0.0001 in.] is adequate. A 5-mm [0.25-in.] nominal diameter flat-flat micrometer or caliper, accurate to within ±25 µm [±0.001 in.], shall be used to measure the length and width of the specimen.

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

7.6 *Specimen Bag*—A sealable, flexible, moistureproof 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*—At least five specimens per test condition should be tested unless valid results can be gained through the use of fewer specimens, as in the case of a designed experiment. The number of specimens tested and the method of sampling shall be determined from the statistical requirements.

TABLE 1 Balance Accuracy

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

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. 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 ±5 % over the surface of the specimen and shall comply with 8.2.2 and Eq 2, as applicable.

NOTE 3—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 10.1.7.1. 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 8.2. 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*—As edge effects must be minimized in materials with anisotropic moisture diffusivity constants, the test specimen for moisture diffusivity constant determination shall consist of either 8.2.4.1 or 8.2.4.2.

TABLE 2 Summary of Specimen Geometry Requirements

Property Testing Procedure A	Conditioning Procedures B, C, or D
Known Reference Time Period:	*Known Specimen Thickness:*
1. Determine the maximum thickness from $h = \sqrt{D_z t/0.02}$. If a thicker specimen is required then the reference time period must be changed.	1. Determine the reference time period from 10.1.7.1. If this is unacceptable then the specimen thickness must be changed.
2. Determine the plate size from either Eq 2 (non-sealed edges) or 8.2.4.2 (sealed edges).	2. Estimate the specimen mass from the material density, known thickness, and the configuration. Specimen mass shall be ≥5 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.
3. Estimate the specimen mass from $Mass = w^2 \times h \times density$.The specimen mass shall be ≥5 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).	
Known Specimen Thickness:	
1. Determine the reference time period from 10.1.7.1. If the value so determined is unacceptable then the specimen thickness must be changed.	
2. Determine the plate size from either Eq 2 (non-sealed edges) or 8.2.4.2 (sealed edges).	
3. Estimate the specimen mass from $Mass = w^2 \times h \times density$.The specimen mass shall be ≥5 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).	

8.2.4.1 A nominally square plate or curved panel with dimensions that satisfy the relation:

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

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

8.2.4.2 A 100 ± 10-mm [4.0 ± 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 4—A typical specimen for the carbon/epoxy material described in X2.2 would have dimensions of 1 mm [0.040 in.] thick by 100 mm [4 in.] square and would have a mass of approximately 18 g. If conditioned at 74°C [170°F] and 90 % relative humidity, use of Eq X1.7 predicts that this specimen would reach equilibrium in approximately 22 days. The minimum reference time period used to test equilibrium is established by 10.1.7.1 as the greater of 0.02 h^2/D_z(equal to 22 h) or 24 h. Coincidentally, this specimen satisfies the criteria of both 8.2.4.1 and 8.2.4.2.

8.2.5 *Specimen Size and Shape For Use With Procedures B, C, or D*—The specimen size and shape requirement for Procedure A (8.2.4) is not a requirement for the conditioning procedures (B, C, or D) of this test method. The coupon size and shape used in Procedures B, C, and D 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, a *traveler* coupon 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 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 10.3.7) when using a marking method that adds mass to the coupon. Examples of various marking methods include coded wire overwrap and edge marking.

NOTE 5—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 doesn't 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.

10. Procedures

10.1 *Parameters To Be Specified Prior To Test*:

10.1.1 The procedure to be used (A, B, C or D).

10.1.2 The density and fiber volume sampling method, coupon geometry, and test method (see Note 1 for guidance).

10.1.3 The moisture absorption specimen sampling method, type, and geometry (and travelers, if required), from Section 1.7.

10.1.4 The balance measurement accuracy, 1.0 mg or 0.1 mg, from 7.1.

10.1.5 Conditioning chamber test temperature. Maximum test temperatures are recommended for all Procedures in Table 3. 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 and moisture exposure level.

NOTE 6—MIL-HDBK-17B **(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 in Procedure B, as long as equilibrium is reached at the final humidity level. Refs **(7)** and **(8)** discuss methods of accelerated 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 measured to an accuracy of within 1 % of the nominal time interval. All procedures require a final set of weighings spaced apart by the reference time period.

10.1.7.1 Determine the reference time period from Table 4. 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 4. If D_z is unknown then the NO value from Table 4 shall be used.

10.2 *Definition of Effective Moisture Equilibrium*—A material is in a state of effective moisture equilibrium when the average moisture content of the material changes by less than 0.01 % within the span of the reference time period. This can

TABLE 3 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

TABLE 4 Establishing the Reference Time Period

Is D_z Known?	Reference Time Period
Yes	the greater of $0.02 \cdot h^2/D_z$ s *or* 86 400 s (1 day)
No	604 000s (7 days)

be expressed in several equivalent ways; four equivalent expressions are shown below:

$$|M_i - M_{i-1}| < 0.01\ \% \tag{3}$$

or

$$\left|\frac{W_i - W_b}{W_b} - \frac{W_{i-1} - W_b}{W_b}\right| \times 100 < 0.01\ \% \tag{4}$$

or

$$\left|\frac{W_i - W_b}{W_b} - \frac{W_{i-1} - W_b}{W_b}\right| < 0.0001 \tag{5}$$

or

$$\left|\frac{W_i - W_{i-1}}{W_b}\right| < 0.0001 \tag{6}$$

where:

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

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. Provide adequate vapor circulation in vapor exposure environments.

10.3.5 Following any preconditioning, perform and report representative material density and fiber volume tests.

10.3.6 Following any preconditioning, 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. The nominal plate side dimensions shall also be measured to the nearest 1.0 mm [0.050 in.] and reported.

10.3.6.1 Measure and report, as post-test observations, each of these same dimensions again following completion of the applicable procedure.

10.3.7 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 Pre-Conditioning*—Perform Procedure D as a pre-conditioning step.

10.4.2 *Moisture Absorption*:

10.4.2.1 Record the oven-dry specimen mass as the baseline mass, W_b, for moisture absorption. 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 time interval, remove the specimen from the conditioning chamber and place the specimen in the specimen bag. Close or seal the bag and allow the specimen to come to laboratory temperature. 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.

NOTE 7—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. Monitor the absorption of moisture 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_m.

10.4.3 *Moisture Desorption Following Absorption*—If the material is known to contain, or suspected to contain, any appreciable amount of moisture-soluble ingredients, the 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 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, 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.

NOTE 8—If specimen mass loss due to solvent degradation or physical handling is detected, then the calculated moisture diffusion properties may be in error.

10.5 *Procedure B—Wet Specimen Conditioning*:

10.5.1 *Specimen Preconditioning*—Perform Procedure D as a preconditioning step if the moisture of interest is not water or humid air.

NOTE 9—This step is not required if the moisture being used is water or humid air. However, 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.

10.5.2 *Conditioning*—Perform the absorption steps of 10.4.2 to uniform equilibrium. However, for Procedure B, periodic weighing of the specimen is not required. A minimum of three weighings are necessary to ensure effective moisture equilibrium; the pre-test weighing, and two 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.

NOTE 10—While similar to Procedure F of Practices D 618, this procedure requires quantified moisture equilibrium instead of the fixed conditioning time period used by Procedure F of Practices D 618.

10.6 *Procedure C—Conditioning for Standard Conditioned Specimen*:

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 %.

10.6.2 At the end of each time interval, remove the specimen from the conditioning chamber and place the specimen in the specimen bag to cool to laboratory temperature. 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. Periodic weighing of the specimen is not required. A minimum of three weighings are necessary to ensure effective moisture equilibrium; the pre-test weighing, and two 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 11—While similar to Procedure A of Practices D 618, this procedure requires quantified moisture equilibrium instead of the fixed conditioning time period used by Procedure A of Practices D 618. This procedure also allows for elevated temperature conditioning to accelerate the process.

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

10.7.1 Select an oven-drying temperature from Table 3. Initially weigh the specimen to the required precision and record this value as the baseline mass, W_b, for oven-drying. 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 12—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 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 laboratory temperature, 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 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 13—While similar to Procedure B of Practices D 618, this procedure requires quantified moisture equilibrium instead of the fixed conditioning time period used by Procedure B of Practices D 618.

11. Calculation

11.1 *Absorption/Desorption Rate*—For Procedure A, plot the percent mass change (as calculated by Eq 7) versus $\sqrt{time}$ as illustrated by Fig. 1.

11.2 *Absorption/Desorption Percent Mass Change*—Calculate the percent change in mass for moisture absorption or desorption as follows:

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

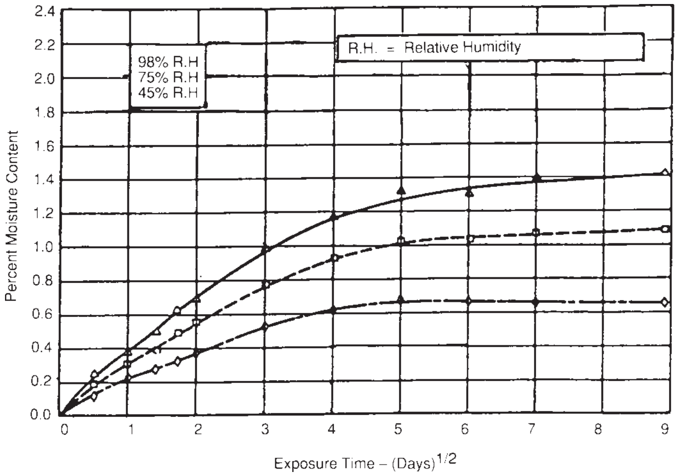

FIG. 1 Moisture Absorption of Carbon/Epoxy $(0,45,-45,0)_s$ Eight-Ply Laminates at 120°F (7)

where:
W_i = current specimen mass, g, and
W_b = baseline specimen mass, g.

11.3 *As-Received Moisture Content*—Calculate the average moisture present in the material prior to conditioning as follows:

$$\text{As}-\text{Received Moisture Content, }\% = \frac{\mathrm{W_{ar}} - \mathrm{W_{od}}}{\mathrm{W_{od}}} \times 100 \qquad (8)$$

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

11.4 *Percent Soluble Matter Lost*—Calculate the percent soluble matter lost, if determined, as follows:

$$\text{Soluble matter lost, }\% = \frac{\mathrm{W_{ab}} - \mathrm{W_p}}{\mathrm{W_{ab}}} \times 100 \qquad (9)$$

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

11.5 *Diffusivity*—For Procedure A, calculate the diffusivity, D_z, as follows:

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

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

NOTE 14—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 15—If there is no discernible initial linear portion of the moisture absorption versus $\sqrt{time}$ 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 used (A, B, C, or D, or combination thereof).

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 preply 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 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 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 $\sqrt{time}$, calculated by Eq 7.

12.1.18 Material effective moisture equilibrium content (in mass percent).

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.

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 D-30 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:

$$\frac{\partial c}{\partial t}=D_z\frac{\partial^2 c}{\partial z^2} \tag{X1.1}$$

where:

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

X1.1.2 The boundary and initial conditions are:

$$c=c_i \qquad 0< z < h \qquad t\leq 0 \tag{X1.2}$$

$$c=c_a \qquad x=0; \qquad x=h \qquad t>0 \tag{X1.3}$$

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:

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

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 (nondimensional) and can be given as follows:

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

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

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

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):

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

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:

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

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 $D_z = 2.6 \times 10^{-7}$ mm^2/s [4.0×10^{-10} in.^{2}/s] and the 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 weighings is on the order of 0.01 % (based upon the balance accuracy and the minimum specimen mass), 0.01 % is used as the accuracy criterion in the definition of effective moisture equilibrium.

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_f, and the average moisture content at the previous weighing is M_{f-1}. M_f. 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_f can be limited to any reasonable value. In this test method the difference is ≤0.02 %, or, $M_m - M_f \leq 0.02$ %.

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:

$$M_f = M_{f-1} + G(T,t)(M_m - M_{f-1}) \tag{X2.1}$$

or

$$G(T,t) = \frac{M_f - M_{f-1}}{M_m - M_{f-1}} \tag{X2.2}$$

or

$$G(T,t) = \frac{0.01}{0.03} \tag{X2.3}$$

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)* = 0.3333 into Eq X1.6 yields a value of the parameter $D_z t/h^2$ that satisfies these requirements. This value, of $D_z t/h^2$. 0.02119, produces a satisfactory effective equilibrium state (within 0.02 % of the true equilibrium moisture content) for the accuracy criterion of 0.01 %. 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_f is within 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 0.02119 was rounded for convenience to 0.02 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_f and M_m:

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

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_f 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:

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

where D_a = apparent through-the-thickness diffusivity constant, mm^2/s [in.^{2}/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 ±0.1 mg (±0.0001 g), the equilibrium accuracy criterion of 0.01 % (0.0001), and the desire to keep the balance about five times more accurate than the required resolution. Therefore:

$$\text{Minimum specimen mass} = \frac{5 \times \text{balance accuracy}}{\text{accuracy criterion}} \tag{X2.6}$$

$$= \frac{5 \times 0.0001 \text{ g}}{0.0001}$$

$$= 5 \text{ g}$$

TABLE X2.1 Predicted M_f Values for Various Moisture Absorption Parameters

Thickness, mm	Time, h	D_t/h^2	G	Accuracy Criterion, %		
				0.05 %	0.01 %	0.005 %
					Predicted M_f	
0.25	24	0.359	0.966 246	1.498	1.500	1.500
1.0	24	0.022 4	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	0.005 6	0.139 085	1.191	1.438	1.469
2.5	24	0.003 6	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	0.044 9	0.509 524	1.452	1.490	1.495
1.5	48	0.020 0	0.321 431	1.394	1.479	1.489
2.0	48	0.011 2	0.222 649	1.325	1.465	1.483
2.5	48	0.007 2	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	0.069 9	0.629 261	1.471	1.494	1.497
2.0	168	0.039 3	0.475 070	1.445	1.489	1.494
2.5	168	0.025 1	0.369 448	1.415	1.483	1.491
25.0	168	0.000 25	0.032 258	0.000	0.000	0.000

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

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