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Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials¹

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

INTRODUCTION

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

1. Scope

~~1.1 This test method covers a procedure (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~~

1.1 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 4.6.

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

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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1.5 This test method is consistent with the recommendations of MIL-HDBK-17/BCMH-17 Rev G (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.

1.6.1 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:³

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-Sealable⁴

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:

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

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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:

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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.

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

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, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS, 19111-5098, <http://dodssp.daps.dla.mil>.

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.*)

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, B, C, or D. —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;
 - $M_{e\Delta}$, the average moisture content at establishment of effective moisture equilibrium as a delta from the average moisture content at a baseline time;
 - M_{eT} , the average moisture content at establishment of effective moisture equilibrium as a total value from the condition of zero moisture content;
 - M_f , the average moisture content at establishment of effective moisture equilibrium; *the final moisture content at the end of conditioning for a fixed time;*
 - M_t , the average moisture content at a given time;
 - M_{t-1} , the average moisture content at the previous time; and
 - 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*—~~For the purposes of this test method—Procedures A-H of this test method condition specimens to M_m and e . Except for the use of a thin specimen in Procedure A, conditioning specimens to M_f are assumed to be equivalent. 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_t , the mass at a given time;
 - and W_{t-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,~~
- 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 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.

4.3 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, C, or D; 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, 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):

5.2.1 A single test using Procedure A provides the moisture diffusivity constant,

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.

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

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 1—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. 2—For many polymer matrix composites the moisture absorption properties under atmospheric humid conditions are generally not equivalent to exposure either to liquid immersion or to pressurized steam. These latter environments may have different material diffusion characteristics.

6. Interferences

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

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

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

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

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

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

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

6.3.2 Materials with a significant amount of surface cracking;

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

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

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

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

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

6.3.8 Test apparatus that produces a significant temperature gradient in the specimen, either through-the-thickness or 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.

6.3.9 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 2—These 3—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.

TABLE 1 Balance Accuracy

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

7.2 *Oven or Vacuum Drying Chamber*—An air-circulating oven is required that shall be capable of maintaining the required uniform temperatures to within $\pm 1^{\circ}\text{C}$ [$\pm 2^{\circ}\text{F}$], $\pm 3^{\circ}\text{C}$ [$\pm 5^{\circ}\text{F}$]. 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 $\pm 1^{\circ}\text{C}$ [$\pm 2^{\circ}\text{F}$], $\pm 3^{\circ}\text{C}$ [$\pm 5^{\circ}\text{F}$]. 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*—A 5-mm [0.2-in.] 5 mm [0.2 in.] nominal diameter double-ball micrometer, accurate to 0.1 % of the specimen thickness, shall be used to measure the thickness of the specimen. For typical coupon dimensions an accuracy of $\pm 2.5\ \mu\text{m}$ [$\pm 0.0001\ \text{in.}$] is adequate. A 5-mm [0.25-in.] 5 mm [0.2 in.] nominal diameter flat-flat micrometer or caliper, accurate to within $\pm 25\ \mu\text{m}$ [$\pm 0.001\ \text{in.}$], shall be used to measure the length and width of the specimen.

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

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

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

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

8. Test Specimen

8.1 *Sampling of Test Specimens* —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.:

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 8.2.5 for a discussion of when traveler coupons should be used. If the group of test specimens are not “dried” prior to moisture conditioning, the initial moisture content, M_b , of the material prior to conditioning may be determined by either (a) drying the traveler coupons using Procedure D after moisture conditioning has been completed, (b) drying using Procedure D an additional three traveler coupons, or (c) if the material diffusivity constant and effective moisture equilibrium content are known, by using the procedures in Appendix X3.

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

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

8.1.4 *Procedure Z*—The moisture loss during testing at elevated temperatures should be quantified by a minimum of three (3) “moisture loss” specimens that represent each: material, laminate thickness, hot/wet specimen geometry (width and length), and



test temperature. Separate moisture loss specimens shall be used for fixtured and non-fixtured specimens with the same dimensions. Moisture loss specimens are not required to be tabbed or notched, even if the specimens they represent are tabbed or notched. When simulating tabbed specimens, the moisture loss specimens may be gripped using un-bonded, simulated tabs to prevent specimen damage and mimic heat transfer through tabs into grips.

8.2 Test Specimen Geometry:

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

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

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 8.2.2 and Eq 2, as applicable.

NOTE 3—No 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 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.

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

8.2.4.1 *Thin Specimen*—Since the purpose of the "thin" test specimen is only for moisture equilibrium content determination,

TABLE 2 Summary of Specimen Geometry Requirements

Property Testing Procedure A—Thick Specimen	Conditioning Procedures B, C,
<p><i>Known Reference Time Period:</i></p> <ol style="list-style-type: none"> Determine the maximum thickness from $h = \sqrt{D_r t / 0.02}$. If a thicker specimen is required then the reference time period must be changed. Determine the plate size from either Eq 2 (non-sealed edges) or 8.2.4.2 (sealed edges). 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). <p>1. Determine the maximum thickness from $h = \sqrt{D_r t / 0.04}$. 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 10.2; if a different value for effective equilibrium moisture change is used, see 10.1.8 and Appendix X2.</p> <ol style="list-style-type: none"> Determine the plate size from either Eq 2 (non-sealed edges) or 8.2.4.2 (2) (sealed edges). 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). <p><i>Known Specimen Thickness:</i></p> <ol style="list-style-type: none"> Determine the reference time period from 10.1.7.1. If the value so determined is unacceptable then the specimen thickness must be changed. Determine the plate size from either Eq 2 (non-sealed edges) or 8.2.4.2 (2) (sealed edges). 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). 	<p><i>Known Specimen Thickness:</i></p> <ol style="list-style-type: none"> Determine the reference time period from 10.1.7.1. If this is unacceptable then the specimen thickness must be changed. 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.

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:

$$\geq 100 \text{ D5229_D5229M-12_5}$$

where:

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

h = nominal thickness, mm [in.].

~~8.2.4.2A 100 ± 10-mm [4.0 ± 0.5-in.]~~ (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 8—A typical "thin" specimen for the carbon/epoxy material described in X2.2 would have having 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 (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 2257 days. The minimum reference time period used to test equilibrium is established by 10.1.7.1 as the greater of $0.020.04 \text{ h}^2/D_z$ (equal to 22 h) or 24 h. Coincidentally, this specimen satisfies the criteria of both 8.2.4.1 and 8.2.4.2-D, (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 8.2.4.2 (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, C, or D~~ 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 (~~B, C, or D~~) (B-Z) of this test method. The coupon size and shape used in Procedures ~~B, C, and D~~ 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, a *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 ~~10.3.7~~ 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 5—A 9—A~~ 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 ~~doesn't~~ 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, 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

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 Method 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.

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		