Designation: D4502 – 92 (Reapproved 2011)

Standard Test Method for
Heat and Moisture Resistance of Wood-Adhesive Joints[1]

This standard is issued under the fixed designation D4502; 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.

1. Scope

1.1 The purpose of this test method is to estimate the resistance of adhesive-bonded joints to thermal and hydrolytic degradation.

1.2 This test method is primarily for wood-to-wood joints but may be applied to joints of wood to other materials.

1.3 The effects of chemicals such as fire retardants, preservatives, and extractives in the wood upon joint degradation resistance can be estimated.

1.4 This test method does not account for the effects of stress, the other principal degrading factor, nor does it account for cyclic or variable temperature or moisture levels.

1.5 *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:*[2]

D897 Test Method for Tensile Properties of Adhesive Bonds

D905 Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading

D907 Terminology of Adhesives

D2304 Test Method for Thermal Endurance of Rigid Electrical Insulating Materials

D2307 Test Method for Thermal Endurance of Film-Insulated Round Magnet Wire

D2339 Test Method for Strength Properties of Adhesives in Two-Ply Wood Construction in Shear by Tension Loading

2.2 *IEEE Standard:*

IEEE No. 1 General Principles for Temperature Limits in the Rating of Electrical Equipment[3]

3. Terminology

3.1 *Definitions*

3.1.1 For definitions of terms used in this test method, refer to Terminology D907.

3.2 *shear strength, n*—in an adhesive joint, the maximum average stress when a force is applied parallel to the joint.

3.2.1 *Discussion*—In most adhesive test methods, the shear strength is actually the maximum average stress at failure of the specimen, not necessarily the true maximum stress in the material.

4. Summary of Test Method

4.1 The degradation of adhesive joints is a physicochemical process. The speed of degradation is related to the levels of temperature, moisture (and other chemicals), and physical stress to which the joint is exposed. This test method is based on the principles of chemical kinetics and uses the Arrehenius temperature dependence relationship to estimate the long-term effects of heat and moisture at the service temperature.

4.2 Specimens whose unaged properties have been estimated by control tests are subjected to an accelerated thermal or hydrolytic aging environment in groups. Aging is accelerated by using elevated temperature. Periodically, a group of specimens is removed from the aging environment and tested. The estimated property after aging and the time of aging are recorded. After several groups have been tested in this manner, the rate of property loss in the aging environment can be estimated. This basic experiment is repeated at several other elevated temperatures, and the rates of property loss at those temperatures estimated. The rate of property loss relationship to temperature is estimated. This relationship can be extrapolated to lower service temperatures for estimating service life.

4.3 This test method employs a smaller version of the Test Method D905 block shear specimen, but other shear strength or tensile strength specimens may also be used.

[1] This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.70 on Construction Adhesives.

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[2] 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.

[3] Available from Institute of Electrical and Electronics Engineers, Inc. (IEEE), 445 Hoes Ln., P.O. Box 1331, Piscataway, NJ 08854-1331, http://www.ieee.org.

5. Significance and Use

5.1 This test method can serve as a useful tool for durability assessment and service life forecasting.

5.1.1 This test method can be used to measure the effects of heat and moisture and the effect of their interaction on adhesives and bonded joints. Knowledge of these effects is useful to an adhesive formulator or manufacturer. Moist heat aging is particularly useful for determining the effects of acidic adhesive systems on the hydrolysis of wood adherends.

5.1.2 This test method provides a means of comparing the rate of degradation of an unknown adhesive-adherend combination to the rate of degradation of a known combination in thermal or hydrolytic aging environments. Such a comparison can be useful to adhesive manufacturers for introducing a new product to the market and for helping designers selecting adhesives.

5.1.3 This test method does not duplicate any natural service environment, but it does provide a means of estimating the service life of joints in similar environments. Service-life estimates are useful to designers of bonded structures or structures using bonded products.

5.2 Service-life estimates rely on the assumption that the chemical degradation mechanism is the same at the elevated aging temperatures as at the service temperature. However, this may not be true in every case. This possibility, together with the variability in specimen preparation, in the aging exposures, and in the strength measurements, require that caution be used in accepting the estimate of service life.

6. Apparatus

6.1 *Aging Ovens*—Ovens are required that are capable of control within ±2 % of specified exposure temperature throughout the chamber for extended periods of time (±0.5°C control is desirable).[4] The ovens must be capable of operating at temperatures from 60 to 175°C. The oven must have an internal capacity for up to 100 specimens well-spaced and supported on racks to allow free air flow.

6.2 *Environmental Chambers*—Chambers for moist-heat aging must be capable of ±0.5°C temperature and 0.5 % relative humidity control uniformly throughout the chamber. The chamber must be capable of operating at temperatures from 60 to 90°C and relative humidity from 60 to 80 %. The chamber must have the capacity for up to 100 specimens well-spaced and supported on racks to allow free air flow.

6.3 *Moist Aging Jars*—Heat-resistant glass jars are required to expose specimens to constant relative humidity and temperature over saturated salt solutions. Wide-neck canning jars with volumes of 3½ L (1 gal), rubber gaskets, and clamp lids have proven satisfactory at temperatures of 100°C (212°F) and below. The jars must have a platform inside (without legs) to support specimens above the saturated salt solution. A 6-mm (¼-in.) diameter bead of silicone sealant around the inside surface of the jar and about 5 cm (2 in.) above the bottom provides a ledge to support the platform. The platform must be perforated to permit free-flow of water vapor. It may be cut from any material that is resistant to corrosion, heat, and moisture. Perforated high-density hardboard has proven satisfactory. The platform must be cut in half to pass through the neck of the jar. An aging jar with platform is shown in Fig. 1. The jars must be placed in an aging oven, such as described in 5.1, to achieve the required temperature.

6.4 *Water Baths*—Constant-level water baths capable of control to within 0.5°C of the desired temperature are required. The baths must be able to contain 100 specimens.

6.5 *Testing Machine*—The testing machine shall have a capacity of not less than 3000 kg (6210 lbf) in compression. The machine shall be capable of maintaining a uniform rate of loading such that the load may be applied with a continuous motion of the movable head to the maximum load at a rate of 10.0 ± 5 mm/min (0.40 in./min) with a permissible variation of +0.5 %.

6.6 *Shearing Tool*—A shearing tool similar to the tool pictured in Test Method D905 is satisfactory. The tool must have a self-aligning seat to ensure uniform lateral distribution of the load.

7. Materials

7.1 *Adhesive to Be Tested:*

7.2 *Joints*—Wood for wood-to-wood joints or joints of wood to metal or plastic shall be free of defects such as knots, cracks, short-grain and sharp-grain deviations, or any discolorations or soft spots indicative of decay. Generally, a high-density uniform-textured wood is desirable so that the maximum stress will be placed on the adhesive joint during testing. The standard shall be hard maple (*Acer saccharum* or *Acer nigrum*) having a minimum specific gravity of 0.65 (based on oven-dry weight and volume). Other species may be used where evaluation of the adhesive's performance in contact with that species is a specific requirement.

7.3 *Saturated Salt Solutions*—A constant relative humidity at a given temperature can be maintained in sealed aging jars by a saturated aqueous solution in contact with an excess of the solid phase of a specific salt. Tables are available that show relative humidities at given temperatures for many salts.[5] Sodium chloride is recommended. A saturated solution of sodium chloride will produce a relative humidity of 73 to 76 % over the temperature range from 40 to 100°C. This translates to wood moisture content in the approximate range from 9 to 13 %.

8. Test Specimens

8.1 A modified block shear specimen (Fig. 2) is suggested. The specimen is similar to the specimen of Test Method D905, but its smaller size allows more specimens to fit in the aging chambers. Other specimens such as used in Test Method D897 or Test Method D2339 are also satisfactory. If a type from Test

[4] Millett, M. A., Western, L. J., and Booth, J. J., "Accelerated Aging of Cellulosic Materials: Design and Application of a Heating Chamber," TAPPI, Vol 50, No. 11, 1967, pp 74A–80A.

[5] Dean, J. A., ed., *Lange's Handbook of Chemistry*, 12th ed., McGraw-Hill Book Co., Inc., 1978.

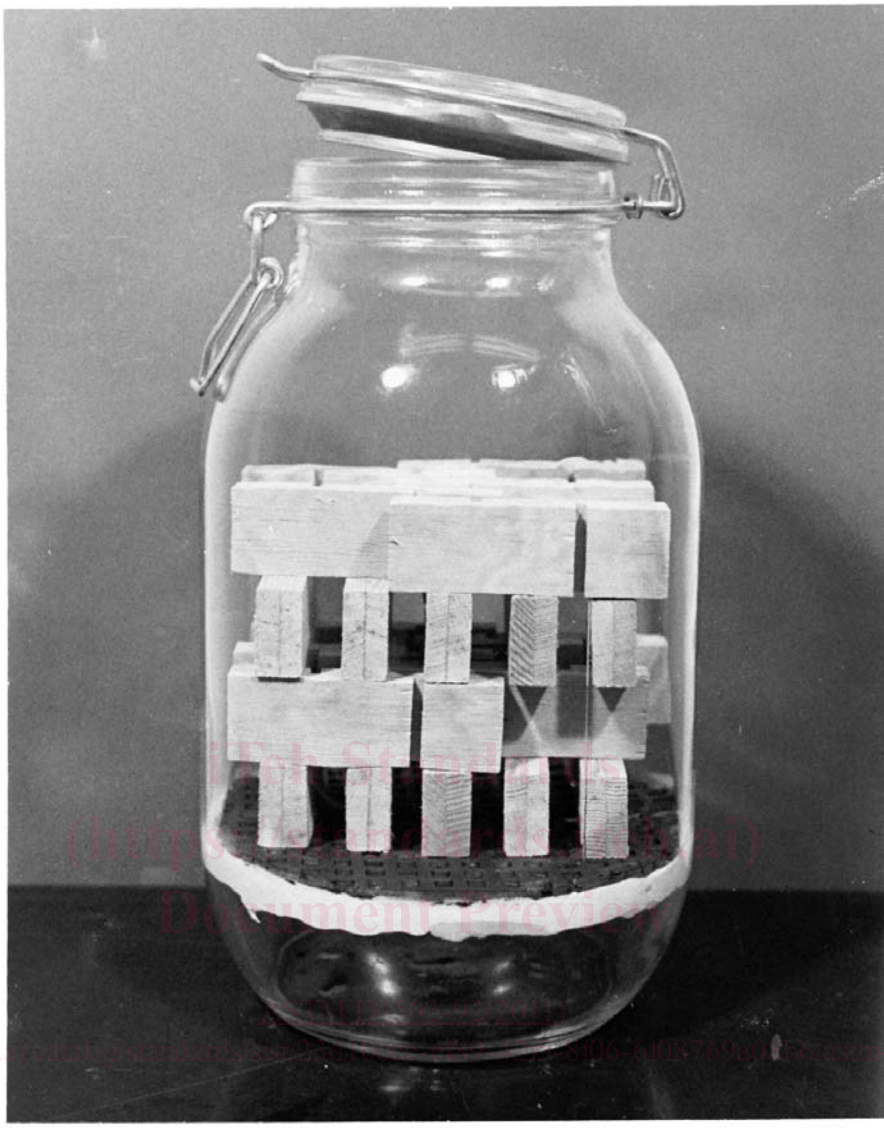

FIG. 1 Moist Aging Jar with a Shelf for Aging Specimens Over a Saturated Salt Solution

Method D2339 is selected, then use 6.5-mm (¼-in.) lumber for each lamina, and increase the specimen length to 130 mm (5.1 in.) while maintaining the 25.4-mm (1-in.) overlap. Other bonded joints or products may also be tested if a suitable specimen can be devised.

8.2 Condition the wood at 23 ± 2°C (73.4 + 3.6°F) and relative humidity of either 30 or 65 %, or other conditions, depending on the adhesive manufacturer's requirement.

8.3 Prepare modified shear block specimens as described in Test Method D905 with the following exceptions:

8.3.1 Cut rough 25.4-mm (1-in.) lumber into 127 or 63 by 305-mm (5 or 2½ by 12-in.) billets as required by Section 9. Saw each billet in half through the thickness using a bandsaw. Joint the surface of each half that is to be bonded and plane to 8-mm (5⁄16-in.) thickness. (Note 1) Bond the billets as described in Test Method D905.

NOTE 1—If during strength testing specimens fail in compression parallel to the grain at the ends, the laminae thickness should be increased from 8 mm (5⁄16 in.) to 9.5 mm (⅜ in.) or greater, as necessary.

8.3.2 After bonding, trim one edge and one end of each panel. Then cut two rows of five specimens each from the 63 by 305-mm (2½ by 12-in.) panels, as shown in Fig. 3, or four rows of five specimens each from the 127 by 305-mm (5 by 12-in.) panels.

NOTE 2—The adhesive should be thoroughly cured by hot pressing, oven heating, high-frequency heating, or whatever method is appropriate. Undercured adhesives cause unwanted results in the early stages of elevated temperature aging.

8.4 Mark each specimen using a template before cutting to indicate the panel and position in the panel.

9. Sampling

9.1 *Sample Size:*

9.1.1 If using the modified block shear specimen, prepare the following numbers and sizes of panels, depending on the

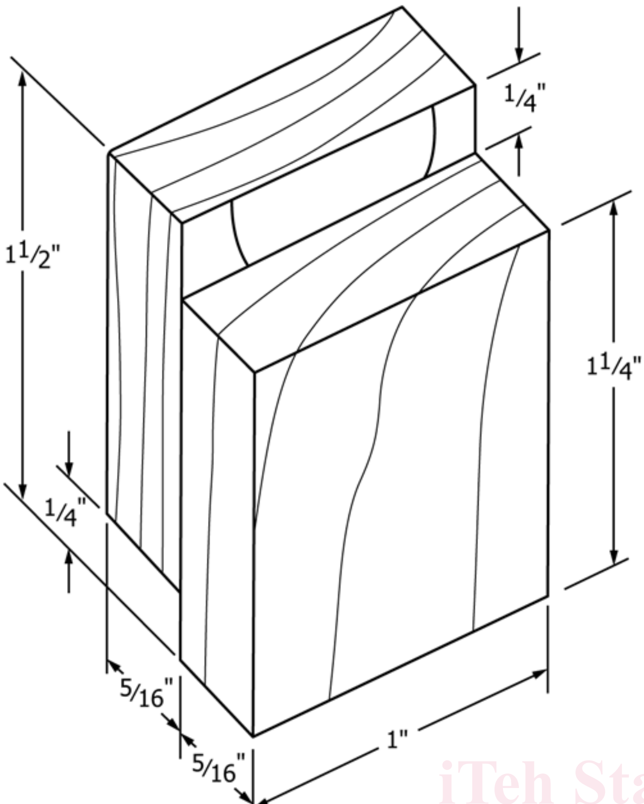

FIG. 2 Modified Block Shear Specimen

type of experiment to be performed (service life, rate comparison, or quality control):

Service life estimation 10 panels,
127 by 305 mm
Rate comparison:
One adhesive/different exposures
(10 panels, 127 by 305 mm)
Two adhesives/same exposure
(10 panels, 63 by 305 mm)
(for each adhesive)
Quality control (10 panels,
63 by 305 mm)

9.1.2 If using some other specimen, prepare 10 panels, each panel large enough to yield the following minimum number of specimens depending on the type of experiments to be performed:

Service life	26
Rate comparison:	
One adhesive/different exposure	22
Two adhesives/same exposure	12
Quality control	3

9.2 *Sampling Method:*

9.2.1 In a given experiment (service life, rate comparison, or quality control) pair the 6.5 by 127 (or 63) by 305-mm billets randomly for bonding into panels.

9.2.2 Distribute the specimens from each panel according to the plan shown in the appropriate table for the experiment.

Service-life estimation	Table 1
Rate comparison	Table 2
Quality control	Table 3

9.2.3 The distribution of specimens for subsequent data analysis is summarized by the block experimental designs shown in Table 4 for each of the experiments.

10. Procedure

10.1 *Initial Strength:*

10.1.1 Condition the control specimens to equilibrium moisture content (EMC) at 23 + 2°C and 50 ± 2 % relative humidity or other conditions as agreed upon by the parties involved. One to four weeks may be required to reach EMC, depending on the beginning moisture content.

10.1.2 Test the specimens (after they reach EMC) in the shear tool with the universal test machine crosshead moving at 10 + 0.05 mm/min (0.400 ± 0.002 in./min). Store the specimens in a plastic bag, or remove them one at a time from the conditioned environment during testing. Record the strength and estimated percentage of wood failure for each specimen.

10.2 *Service Life Estimate:*

10.2.1 Aging temperatures are given in Table 4. For a given temperature/moisture condition, mount five groups (10 specimens per group) on suitable racks for dry aging, place in jars for moist aging, or string each group on stainless steel wire for wet aging.

10.2.2 Estimate five aging intervals that will produce approximately equal strength decrements to a total strength loss of 25 to 30 % from the initial strength for each of the five aging temperatures. Previous aging experience may not be available, especially for new adhesives. If this is the case, use the approximate times given in Table 5.

NOTE 3—Twenty-five percent strength loss is a convenient level. Any amount of loss can be defined as failure as long as it is agreeable to the parties requiring this test and it is defined in the report. Higher percentages of loss require longer exposure times.

10.2.3 Place the five groups (see Note 4) in the aging exposure. At the end of the first aging interval, withdraw the first group of specimens, recondition to EMC, and test as described in 10.1.1 and 10.1.2. Based on this test, project the time to reach 25 % loss. If necessary, adjust the remaining intervals to provide approximately equal strength decrements to the 25 to 30 % strength loss (from the initial value). Repeat this projection and adjustment after each of the first four aging intervals.

NOTE 4—When the aging intervals are shorter than the time necessary to recondition the aged specimens to EMC at 23°C and 50 % relative humidity before testing, then all five groups should not be placed in the aging exposure at once. Instead, place only one or two groups on exposure. In this way specimens will still be available for shorter aging intervals in case the strength degraded too far in the first interval.

10.3 *Degradation Rate Comparison:*

10.3.1 Aging may be dry, moist, or wet depending on the aging conditions of the adhesive to which the test adhesive is to be compared. Select three temperatures from Table 1 for the chosen moisture level. If the adhesive is thought to be very durable, use the three highest temperatures. If the adhesive is thought to be less durable, use the three lower temperatures. For a given temperature, use five groups (20 specimens per group). Prepare for aging as described in 9.2.1.

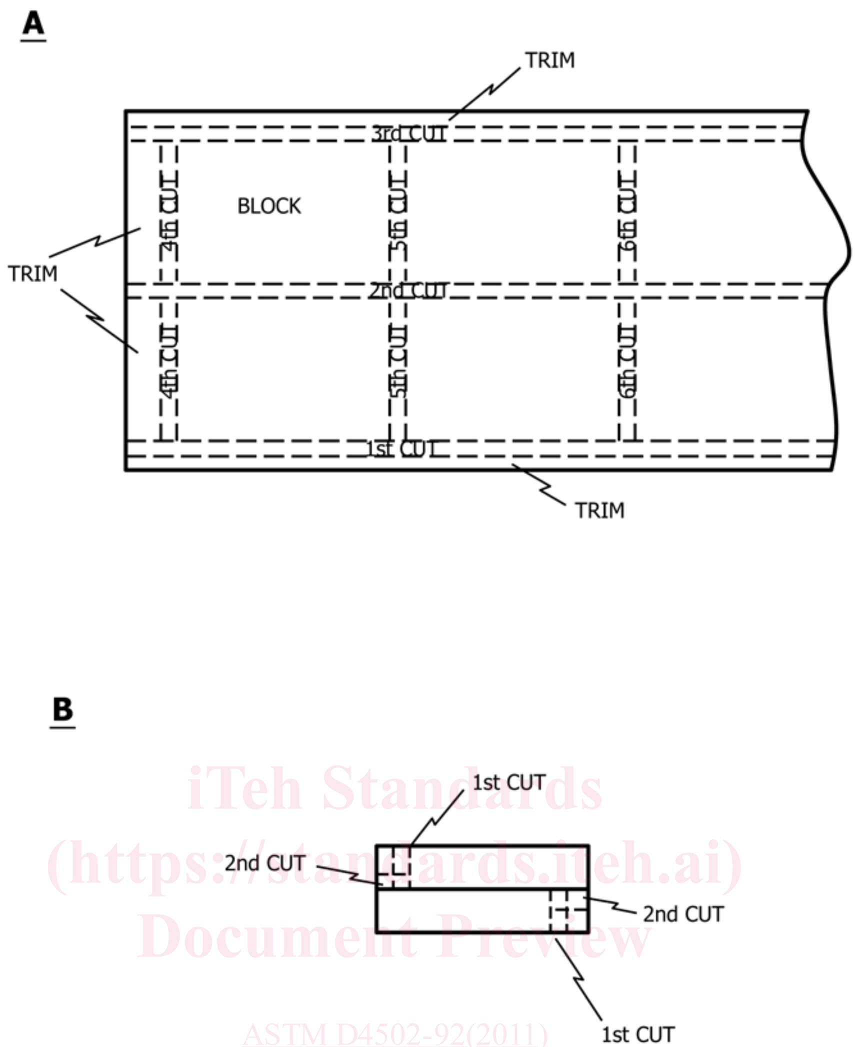

FIG. 3 (*A*) Top View of One End of a Panel Showing Trim and Individual Blocks for Specimens, and (*B*) Side View Showing Two Cuts on Each End of a Block to Form the Offset

10.3.2 Estimate five aging intervals to 25 to 30 % strength loss as described in 10.2.2, 10.2.3, and Note 4.

10.3.3 After aging, recondition the specimens to EMC and test as described in 10.1.1 and 10.1.2.

10.4 *Quality Control:*

10.4.1 Normally the quality control test will be applied to adhesives previously evaluated by the service life or degradation rate procedures. Select one temperature for dry and one temperature for wet aging from Table 4 that should cause a 25 to 30 % strength loss in less than 48 h, based on the previous aging experience. If previous experience is not available, select a temperature/time from Table 5 as a starting point.

10.4.2 Age the group of specimens for a time that is the same as one of the times used in the previous evaluation and that should cause about 25 to 30 % strength loss. After aging, recondition to EMC and test in accordance with 10.1.1 and 10.1.2.

11. Calculations

11.1 *Service Life Estimate:*

11.1.1 Make a visual estimate of the expected service life as follows:

11.1.1.1 Calculate the average values of the residual shear strength from the 10 specimens at each aging interval, for each temperature.

11.1.1.2 Prepare rate curves for each temperature by plotting the log of the average residual strength at a given aging interval as a function of the aging time.

11.1.1.3 Determine the estimate of the initial strength (*y* axis intercept) and the aging time at which each rate curve intersects the 75 % residual strength line (Fig. 4).

11.1.1.4 For the wet and dry aging conditions, plot the aging times to 75 % residual strength as a function of temperature in the Arrhenius convention of log time versus reciprocal temperature (Fig. 5). Visually fit a straight line through the five temperature data points for the wet or dry condition.

11.1.1.5 Project this line to the temperature at which the expected service life is to be estimated but not more than 50°C lower than the lowest accelerated aging temperature.

TABLE 1 Specimen Distribution for Service Life Estimation Experiment at a Single Moisture Level Using Ten 127 by 305-mm (5 by 12-in.) Bonded Panels Yielding 2 Specimens Each

Test group		Panel										Total
Temperature	Aging interval	1	2	3	4	5	6	7	8	9	10	
Control		1	1	1	1	1	1	1	1	1	1	10
1	1	1	1	1	1	1	1	1	1	1	1	10
1	3	1	1	1	1	1	1	1	1	1	1	10
1	4	1	1	1	1	1	1	1	1	1	1	10
1	5	1	1	1	1	1	1	1	1	1	1	10
2	1	1	1	1	1	1	1	1	1	1	1	10
2	2	1	1	1	1	1	1	1	1	1	1	10
2	3	1	1	1	1	1	1	1	1	1	1	10
2	4	1	1	1	1	1	1	1	1	1	1	10
2	5	1	1	1	1	1	1	1	1	1	1	10
3	1	1	1	1	1	1	1	1	1	1	1	10
3	2	1	1	1	1	1	1	1	1	1	1	10
3	3	1	1	1	1	1	1	1	1	1	1	10
3	4	1	1	1	1	1	1	1	1	1	1	10
3	5	1	1	1	1	1	1	1	1	1	1	10
4	1	1	1	1	1	1	1	1	1	1	1	10
4	2	1	1	1	1	1	1	1	1	1	1	10
4	3	1	1	1	1	1	1	1	1	1	1	10
4	4	1	1	1	1	1	1	1	1	1	1	10
4	5	1	1	1	1	1	1	1	1	1	1	10
5	1	1	1	1	1	1	1	1	1	1	1	10
5	2	1	1	1	1	1	1	1	1	1	1	10
5	3	1	1	1	1	1	1	1	1	1	1	10
5	4	1	1	1	1	1	1	1	1	1	1	10
5	5	1	1	1	1	1	1	1	1	1	1	10
Total		26	26	26	26	26	26	26	26	26	26	260
Leftover		2	2	2	2	2	2	2	2	2	2	20

TABLE 2 Specimen Distribution for the Experiment to Compare Degradation Rates of a Single Adhesive[A] in Two Exposures Using 127 by 305-mm (5 by 12-in.) Bonded Panels Yielding 28 Specimens Each

Test Group	Aging Interval	Panel										Total
		1	2	3	4	5	6	7	8	9	10	
						Exposure I						
Control		2	2	2	2	2	2	2	2	2	2	20
	1	2	2	2	2	2	2	2	2	2	2	20
	2	2	2	2	2	2	2	2	2	2	2	20
	3	2	2	2	2	2	2	2	2	2	2	20
	4	2	2	2	2	2	2	2	2	2	2	20
	5	2	2	2	2	2	2	2	2	2	2	20
						Exposure II						
Control		2	2	2	2	2	2	2	2	2	2	20
	1	2	2	2	2	2	2	2	2	2	2	20
	2	2	2	2	2	2	2	2	2	2	2	20
	3	2	2	2	2	2	2	2	2	2	2	20
	4	2	2	2	2	2	2	2	2	2	2	20
	5	2	2	2	2	2	2	2	2	2	2	20
Total		24	24	24	24	24	24	24	24	24	24	240
Leftover		4	4	4	4	4	4	4	4	4	4	40

[A] To compare two different adhesives in a single exposure prepare ten 63 by 305-mm (2½ by 12-in.) panels (yielding 14 specimens each) with each adhesive. If comparing two adhesives, a group of control specimens is also required for the second adhesive.

11.1.2 Make a statistical estimate of the service life in a given moisture condition. Detailed procedures are given in Annex A2 or Annex A3 (Version I or II).

11.1.2.1 First, for data at the moisture condition, fit the strength-aging time data obtained at each aging temperature to the following equation:

$$\log_{10} y = \log_{10} a + kt \tag{1}$$

where:

y = strength,
t = aging time,
a = estimated initial strength, and
k = degradation rate constant.

11.1.2.2 Next, use the fitted strength versus aging time equations to determine the time required for the adhesive