Designation: D 3632 – 98 (Reapproved 2004)

Standard Test Method for Accelerated Aging of Adhesive Joints by the Oxygen-Pressure Method[1]

This standard is issued under the fixed designation D 3632; 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 This test method describes how to estimate the relative resistance to deterioration of adhesive films and adhesive-bonded joints placed in a high-pressure oxygen environment. The instructions include both wood-to-wood and wood-to-metal joints as well as free film of adhesive. The effects of chemicals such as fire retardants, preservatives, or wood extractives, can be evaluated by using materials containing these chemicals for adherends.

1.2 This test method is primarily intended for elastomer-based construction adhesives, but is also applicable to other types of adhesives that may be susceptible to oxygen degradation. This accelerated test does not correlate exactly with the natural aging of the adhesive because of the varied conditions of natural aging and the absence of factors such as moisture and stress. The results of this accelerated test are only comparative and must be evaluated against the performance of bonded joints whose natural and accelerated aging characteristics are known.

1.3 *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]

D 143 Test Methods for Small Clear Specimens of Timber

D 454 Test Method for Rubber Deterioration by Heat and Air Pressure

D 572 Test Method for Rubber—Deterioration by Heat and Oxygen

D 573 Test Method for Rubber—Deterioration in an Air Oven

D 907 Terminology of Adhesives

D 2339 Test Method for Strength Properties of Adhesives in Two-Ply Wood Construction in Shear by Tension Loading

3. Terminology

3.1 *Definitions*—Many definitions in this test method are defined in Terminology D 907.

4. Summary of Test Method

4.1 This test method entails subjecting specimens with known physical properties to a controlled aging environment for specific time periods, then observing the physical properties again and noting any changes. The controlled environment consists of elevated temperature (70°C) (158°F) and oxygen at elevated pressure 2.07 MPa (300 psi).

4.2 Three types of test specimens are offered. The party requesting the adhesive evaluation will choose the type of specimen to be used.

Specimen Type	Configuration	Physical Property Tested
A	wood-to-wood lap	shear strength
B	wood-to-metal lap	shear strength
C	unsupported film	flexibility

4.3 Three different oxygen-pressure aging exposures are offered. Use any exposure with any of the above specimens. The party requesting the adhesive evaluation will choose the exposure to which the specimens are subjected.

4.3.1 Constant exposure for 500 h with a single test of the physical property at the end of 500 h.

4.3.2 Constant exposure for 1000 h with a single test of the physical property at the end of 1000 h.

4.3.3 Constant exposure for up to 1000 h with a series of tests of the physical property after 200, 400, 600, 800, and 1000 h.

5. Significance and Use

5.1 This test method is useful to the adhesive manufacturer in research and development or in manufacturing control. The results are also used for specification acceptance or as a guide in adhesive selection.

[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.

5.2 The provisions for testing bonded specimens as well as free films are made for two purposes. First, it is possible for an interaction to occur between oxygen and chemicals or degradation products that may affect the degradation of the bonded joints strength. Second, some increase in strength due to oxidative crosslinking may not be detrimental in a bonded assembly and in fact may be beneficial. Adhesives of this behavior are not satisfactorily tested by a film flexibility test.

5.3 Some users of this test method will be most interested in the performance of the bonded joint; some will be most interested in the performance of the adhesive. In the latter case, it is important to note that the true variance (error mean square) of the strength of the adhesive may be obscured when the tested control specimens or the tested aged specimens show wood failure.

5.4 *Conflict of Procedure*—If the procedures of this test method conflict with those of detailed product specifications or manufacturer's use instructions for a particular material, then use the latter.

6. Apparatus

6.1 *Oxygen-Pressure Vessel*—The specifications for the oxygen-pressure vessel described in 6.1.1.1 through 6.1.1.8 are the same as those described in Test Method D 572. Adequate safety provisions are important when heating oxidizable organic materials in oxygen, since the rate of reaction may become very rapid and very high pressures may develop. Heating these materials is especially dangerous when a large surface area is exposed. If the same equipment is used for the oxygen-pressure test as for the air-pressure heat test (Test Method D 454), be careful and check to see that the thermostatic controls are set properly because the specimens may react with oxygen very rapidly at the temperature of the air-pressure heat test. Fluids acceptable as heating media for one test may be hazardous when used for the other test.

6.1.1 Use on oxygen-pressure chamber consisting of a metal vessel designed to retain an internal atmosphere of oxygen gas under pressure, with provisions for placing specimens within it and for subjecting the entire chamber to controlled uniform temperature. Because of the superior temperature control and heat transfer, a metal vessel completely immersed in a liquid medium is recommended for referee tests. Ensure that the apparatus conforms to the following requirements:

6.1.1.1 The chamber can be any size; however, it must be large enough so that the specimens can be hung within it vertically, without crowding them, letting them touch each other, or letting them touch the sides of the chamber.

6.1.1.2 The source of heat is optional, but a location outside of the aging chamber itself is required.

6.1.1.3 The heating medium is optional. Water, air, or other fluids that will not ignite when oxygen is present may be used. Water has an advantage because it transfers heat rapidly and is noncombustible. When using air for the heating medium, thoroughly circulate the heated air by mechanically agitating it, and use baffles as needed to prevent local overheating and dead spots. Do not use oils or other combustible fluids as heating media for this test because they are extremely hazardous when oxygen is present.

6.1.1.4 Use a thermostat to control automatically the temperature of the heating medium.

6.1.1.5 Record the temperature automatically throughout the test period. If the pressure chamber is completely immersed, use the temperature of the heating medium as the temperature of the pressure chamber. Place the sensing element close to the temperature-measuring device but not touching the pressure chamber. If the pressure chamber is not completely immersed in the heating medium, place the sensing element in a thermometer well that extends into the pressure chamber. Fill the thermometer well with enough water to cover the element so that heat will transfer easily. If a comparison has been made and it has been confirmed that the temperature of the oxygen within the chamber is the same as the temperature of the heating medium, it is permissible to take the temperature in the heating medium instead of in the thermometer well. When using air as the heating medium, check the temperature in various parts of the oven to determine that the oven is heating evenly. In any case, verify the recorded temperature by checking with a temperature-indicating device whose sensing element is directly exposed to the oxygen within the pressure chamber.

6.1.1.6 Maintain positive, rapid, and complete circulation of the heating medium so as to ensure accurate, uniform heating.

6.1.1.7 The pressure chamber should have a reliable safety valve or rupture diaphragm set for release at 3.448 MPa (500 psi) pressure.

6.1.1.8 Do not expose any copper or brass parts to the atmosphere nor use them in the pressure chamber or the tubing or valves leading to it.

6.2 *Testing Machine for Lap Shear Strength Tests*—The testing machine specifications described in 6.2.1 are the same as those described in Test Method D 2339.

6.2.1 Use a testing machine capable of maintaining a constant rate of loading of 42 to 74 N/s (600 to 1000 lbf/min) or a constant rate of crosshead travel of 0.020 mm/s (0.050 in./min) ± 25 %. Use a testing machine with suitable grips and jaws so that the specimen can be gripped tightly and held in alignment as the load is applied. Fig. 1 shows grips and jaws that have been found satisfactory. Place the testing machine in an atmosphere that will not noticeably alter the moisture content of the specimens developed under the conditions prescribed in 10.2.

6.3 *Air Curing and Drying Oven*—Except for some modification, the oven specifications described (in 6.3.1 through 6.3.9) are the same as those described in Test Method D 573.

6.3.1 Use an oven whose interior size is (minimum) 0.40 m^3 (1 ft^3) to (maximum) 1.33 m^3 (36 ft^3) or any equivalent volume.

6.3.2 Suspend the specimens vertically without letting them touch each other or the sides of the oven.

6.3.3 The temperature variation in various parts of the oven shall not be allowed to exceed 2°C (4°F).

6.3.4 For the heating medium, use air circulated within the chamber at atmospheric pressure.

6.3.5 Any source of air may be used, if it is located in the air supply outside of the chamber itself.

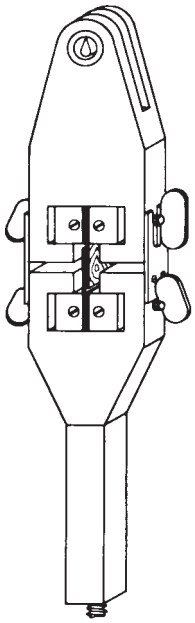


FIG. 1 Grips and Jaws

6.3.6 Install a thermometer in the upper central portion of the chamber, near the center of the specimens, to measure the actual temperature.

6.3.7 Use a thermostat to control the temperature automatically.

6.3.8 Circulate the heated air throughout the oven by mechanically agitating it. When using a motor-driven fan, the air must not come in contact with the fan motor brush discharge because there is danger of ozone forming.

6.3.9 Use baffles where they are needed to prevent local overheating and dead spots.

7. Materials

7.1 *Adhesive*, the adhesive to be tested.

7.2 *Wood* for wood-to-wood and wood-to-metal specimens, rotary cut, sliced or sawn and jointed veneers 3.2 to 6.4 mm (⅛ to ¼ in.) thick, shall be free of defects such as knots, cracks, short grain, or any discolorations or soft spots indicative of decay. The species to be used will be decided by the adhesive manufacturer or by the party requesting these tests. Generally a high-density wood such as Douglas-fir, hemlock, southern pine, or yellow birch is desirable.

7.3 *Metal* for wood-to-metal specimen dimensions be 1.6 to 3.2 mm (1⁄16 to ⅛ in.) thick. The metal will be selected by the adhesive manufacturer or by the party requesting the test, except that the metal used shall not be reactive as, for example, magnesium plate.

7.3.1 The mill finish or chemical treatment of the surface should be the same as the material expected to be bonded in service with finish or surface treatment to be selected by the adhesive manufacturer or by the party requesting the test, except that lubricants or other combustible materials shall be removed from the surface by solvent cleaning before exposure in the oxygen-pressure vessel.

8. Sampling

8.1 *Sampling Method*—When several test specimen panels are made or films cast and groups of individual specimens are aged for different time intervals, mix all the specimens in a box and draw at random from the box for assignment to a given group.

8.2 *Sample Size*—Use at least five test specimens to determine the original physical properties of each sample. Also use five or more specimens of the same material for each exposure period of the test. But for purposes of statistical analyses described in a later section, the number of specimens in the control group and in each aged group should be the same. The user may increase the number of specimens in each group in response to the size of the property change to be detected, the degree of confidence desired, and the test result variability. This subject is further considered in Annex A1.

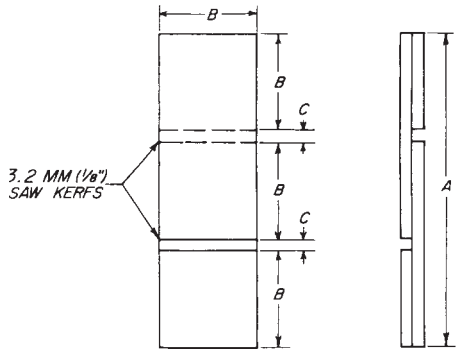
9. Test Specimens

9.1 Lap-shear test specimens must be made from the same adherends and adhesive materials that are actually used in service. A tension lap-shear specimen made like the ones in Fig. 2 and Fig. 3 is recommended. Methods for preparing wood-to-wood specimens are similar to those described in Test Method D 2339 with modifications to accommodate mastic consistency adhesives.

9.2 *Wood-to-Wood Test Panel Preparation*:

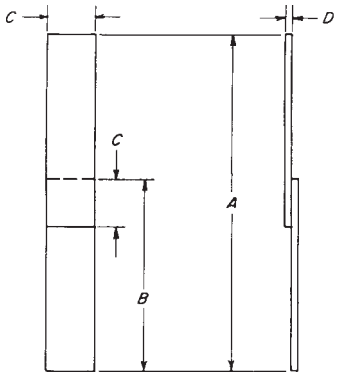
9.2.1 Cut the veneer into suitable sizes and assemble it in pairs with the grain direction of the two sheets parallel to each other. Fig. 4 shows a size that has been found convenient, and in this case the grain is parallel to the shorter dimension. Make sure the veneer is within ± 1 % of the moisture content recommended by the manufacturer of the adhesive. When the manufacturer does not give a recommendation, use a moisture content of 10 to 12 % based on ovendry weight in accordance with Sections 122 to 125 of Test Methods D 143.

9.2.2 If the specimens fail predominantly at the saw kerfs, prepare and test a new set of specimens with thicker veneers or smaller distances between the saw kerfs such as 12.7 mm (0.5 in.), as in Fig. 4.



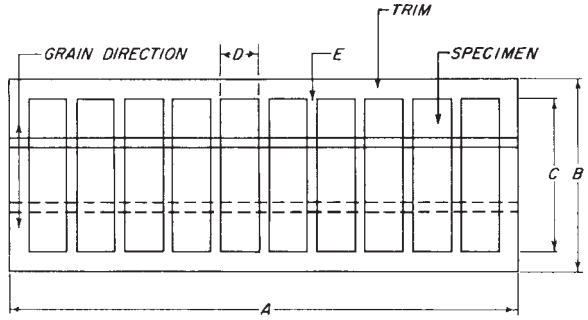
	Metric Equivalents			
	Dimension	Tolerance	Dimension	Tolerance
	mm		in.	
A	82.6	±0.25	3.25	±0.010
B	25.4	±0.25	1.00	±0.010
C	3.17	±0.10	0.125	±0.004

FIG. 2 Wood-to-Wood Tension Shear Test Specimen



	Metric Equivalents			
	Dimension	Tolerance	Dimension	Tolerance
	mm		in.	
A	178	±0.25	7.0	±0.010
B	102	±0.25	4.0	±0.010
C	25.4	±0.25	1.0	±0.010
D	3.17	±0.10	0.125	±0.004

FIG. 3 Wood-to-Metal Tension Shear Test Specimen



	Metric Equivalents			
	Dimension	Tolerance	Dimension	Tolerance
	mm		in.	
A	305	±1.0	12.0	±0.040
B	102	±0.25	4.0	±0.010
C	83	±0.25	3.25	±0.010
D	25.4	±0.25	1.0	±0.010
E	3.17	±0.10	0.125	±0.004

FIG. 4 Bonded Wood-to-Wood Panel Showing Location of Saw Kerfs for Cutting Individual Specimens

9.2.3 Follow the directions of the adhesive manufacturer when applying the adhesive, but use a notched trowel to spread adhesives of mastic consistency. Use a trowel with notches shaped like equilateral triangles 3.2 mm (⅛ in.) on a side, and spaced 3.2 mm (⅛ in.) apart. During troweling, hold the trowel at approximately a 45-deg angle. Apply enough adhesive to fill the notches of the trowel as it passes the length of the 305-mm (12-in.) veneer panel. After the open time prescribed by the adhesive manufacturer has elapsed, assemble the veneers into two-ply panels so that the grain in the two plies is parallel.

9.2.4 Pressure will vary according to the viscosity of the uncured adhesive. Apply enough pressure to the joints to reduce the glueline to 0.4 to 0.8 mm (0.015 to 0.031 in.).

9.2.5 Cure the panels at room conditions, approximately 23°C (77°F) for 14 days; then further cure them at 60°C (140°F) for 12 h. These recommendations are not intended to override any special instructions by the adhesive manufacturer.

9.2.6 After curing the panels, cut individual specimens from them, as shown in Fig. 4 (wood-to-wood panels). Be careful when grooving the wood-to-wood specimens to ensure that the saw cut extends to, but not beyond, the glueline. This procedure can be accomplished by first cutting the individual test slips from the panels as shown in Fig. 4, and then grooving them individually to the proper width, depth, and location with a hollow-ground grooving saw. Use any other method of grooving that will give equally satisfactory results. Alternatively, the panels can first be grooved to the proper width, depth, and location before cutting the individual test specimens. Measure the width of each specimen and the distance between the grooves, to the nearest 0.25 mm (0.010 in.), to determine the shear area.

9.3 *Wood-to-Metal Test Panel Preparation*:

9.3.1 Prepare wood-to-metal specimens in panels as shown in Fig. 5, and cut them into specimens after they are cured. Machine the slots in the metal panels before bonding, using one or more saws on a milling machine arbor. Do not use any metal panels whose edges have burrs or bevels and make sure the edges are at right angles to the faces before the panels are bonded. Apply the adhesive according to the manufacturer's directions, but apply adhesives of mastic consistency in a 3.2-mm (⅛-in.) bead, along the edge of the metal panel to be bonded. Assemble the wood and metal panels so that they will be held rigidly and the length of the overlap is controlled at 25.4 mm (1.0 in.).

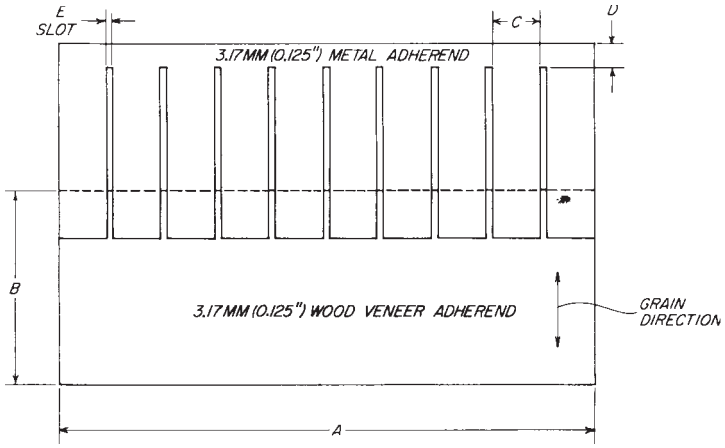
9.3.2 Pressure will vary in accordance with the viscosity of the uncured adhesive. Apply enough pressure to the joints to reduce the glueline 0.4 to 0.8 mm (0.015 to 0.031 in.).

9.3.3 To ensure maximum solvent removal, cure the panels at room conditions, approximately 23°C (77°F) for 14 days, and then further cure them at 60 ± 3°C (140 ± 5°F) for 12 h. These recommendations are not intended to override any special instructions by the adhesive manufacturer.

9.3.4 After cure, cut individual specimens from the panels as shown in Fig. 3 (wood-to-metal specimen). In cutting the wood portion of the wood-to-metal panels to form individual specimens, use the same multiple sawmilling machine setup to ensure uniform width of metal and wood adherends. Remove adhesive squeeze-out after cutting the individual wood-to-metal specimens. Take care not to cut and weaken the wood adherends at the end of the lap, nor to pull adhesive from the interior of the joint. The latter problem can be avoided by using a very sharp blade in a slicing motion.

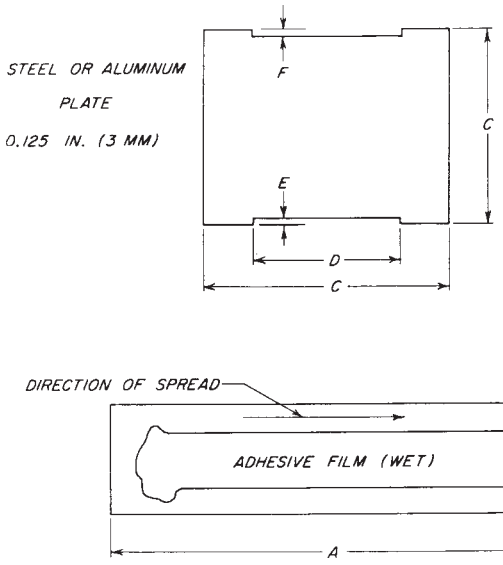
9.4 *Adhesive Film Preparation*:

9.4.1 Prepare specimens by casting wet films of adhesive on a release surface such as silicone-treated paper, TFE-fluorocarbon, or polyethylene sheet as shown in Fig. 6. Cast the first film of adhesive using the 1.27-mm (0.05-in.) gap of the spreader. Condition this film for 3 h at 21 ± 3°C (70 ± 5.4°F) and 50 ± 10 % relative humidity. Then cast a second wet film of adhesive on top of the first film, using the 2.54-mm (0.100-in.) gap of the spreader.



Metric Equivalents				
	Dimension	Tolerance	Dimension	Tolerance
	mm		in.	
A	282	±0.25	11.125	±0.010
B	102	±0.25	4.0	±0.010
C	25.4	±0.25	1.0	±0.010
D	12.7	±0.25	0.50	±0.010
E	3.17	±0.10	0.125	±0.004

FIG. 5 Bonded Wood-to-Metal Panel Showing the Presawn Metal Adherend



Metric Equivalents				
	Dimension	Tolerance	Dimension	Tolerance
	mm		in.	
A	305	±2.0	12.0	±0.08
B	152	±2.0	6.0	±0.08
C	127	±1.0	5.0	±0.040
D	76	±0.25	3.0	±0.010
E	2.54	±0.05	0.100	±0.002
F	1.27	±0.05	0.050	±0.002

FIG. 6 Adhesive Film Spreader (Top) and Film Preparation (Bottom)

9.4.2 For adhesives having solid contents in the range from 95 to 100 % by weight, the wet film of adhesive may be cast in one step using the 2.54-mm (0.100-in.) gap of the spreader.

9.4.3 Cure the completed casting of adhesive for 3 days at 21 ± 3°C (70 ± 5°F) and 50 % relative humidity, then for 2 days at 49 ± 3°C (120 ± 5°F) and relative humidity of 50 %. After cure, cut 25 by 76-mm (1 by 3-in.) specimens from the free film.

10. Conditioning Specimens

10.1 Specimens to be aged must be ovendried for 3 days at 60 ± 3°C (140 ± 5°F) under ambient relative humidity, and atmospheric pressure before being placed in the oxygen bomb. Specimens used to establish the initial strength (not aged in the oxygen bomb) must also be ovendried to maintain comparability.

10.2 Prior to the physical property tests, condition all specimens to approximate equilibrium moisture content at the conditions specified by the party requesting the test. In any case, do not allow more than 96 h to elapse between removal from the oxygen bomb and testing.

11. Adhesive Layer Thickness Measurement

11.1 After conditioning, measure the glueline thickness of each specimen to the nearest 0.02 mm (0.0008 in.) along one edge and record the measurement. A suitable device for measuring the glueline thickness is a low-power binocular microscope with calibrated eyepiece scale yielding approximately 100 divisions per millimetre (0.03937 in.) at a magnification of 80 times.

12. Tests of Unaged Specimens

12.1 Determine the tensile shear strength of bonded specimens, flexibility of free films or any other required properties of the original unaged specimens within 96 h of the start of the

aging period. The procedure for determining tensile shear strength and film flexibility is described in Section 14.

12.2 When properties are to be tested to determine compliance with specifications, it is permissible to determine the unaged properties required in 12.1 simultaneously with the aged properties after the first aging period even though the elapsed time exceeds 96 h.

13. Procedure for Accelerated Aging

13.1 Place the specimens to be aged in the aging chamber when it has been preheated to the operating temperature. No more than 10 % of the volume of the pressure chamber should be occupied by rubber or an oxidizable substance. If possible, avoid simultaneous aging of a mixed group of different compounds. For instance, do not age high-sulfur compounds with low-sulfur compounds, and do not age compounds containing antioxidants with those having no age-resistors. Some migration is known to occur. When starting a test, flush the air out of the oxygen-pressure chamber by releasing the oxygen pressure and refilling the chamber. Also check the chamber to make certain the apparatus does not leak.

13.2 Use an operating temperature of 70 ± 1°C (158 ± 1.8°F) determined as described in 6.1.1.5.

13.3 Use oxygen supplied to the aging chamber at 2.07± 0.10 MPa (300 ± 15 psi) pressure, as measured by a calibrated pressure gage.

13.4 Start the aging interval when the specimens are placed in the heated chamber. The selection of suitable intervals of aging depends on the rate of deterioration of the particular material being tested. Suggested time intervals are 200, 400, 600, 800, and 1000 h.

13.5 Do not use aging intervals that will cause deterioration so great that the final physical properties cannot be determined. In experimental work, it is desirable to use a range of periods, but for routine tests of known materials fewer intervals may be employed.

NOTE 1—**Caution:** For the evaluation of rubber compounds intended to be used at elevated temperatures, the above method may be used with an operating temperature of 80 ± 1°C (176 ± 1.8°F), employing time intervals suggested in 13.4 or otherwise agreed upon. Notice that when the aging temperature is increased to 80°C (176°F) from 70°C (158°F), the rate of oxidation may be expected to approximately double. If the rubber compound rapidly ages, or if it is contaminated by such materials as copper or manganese, the rate of oxidation may be catalyzed to the extent that it becomes violent.

14. Procedure

14.1 Place the tension shear strength test specimens in the jaws of the grips in the testing machine and grip them. Align the jaws so that the pairs of jaws are directly above each other and an imaginary straight vertical line would pass through the glueline and the points of suspension (Fig. 1). Place the specimens in the jaws alternately so that in one case the upper notch is to the left and in the next case toward the right. Apply the load at a rate of 42 to 74 N/s (600 to 1000 lbf/min) ± 25 % to failure.

14.2 Test the flexibility of the free film by bending the film strips 180° around a 6.35-mm (0.25-in.) mandrel.

15. Test of Aged Specimens

15.1 Determine the physical property of the specimens, aged for different intervals, as the intervals terminate. Disregard the fact that more specimens may still be aging. In determining the physical properties after aging, use the average of results from the five or chosen number of specimens as the final result. If one or more values do not meet the specified requirements when testing for compliance with specifications, then make an exception. Expose and test three additional specimens and use the average of the values for the eight specimens as the final result.

15.2 After completing the tests, examine the tested specimens visually and manually, and record descriptions of their condition. (Examples: wood failure of shear test specimens, cracking of adhesive layer, color, texture, tackiness.)

16. Calculations (Tension Shear Test Specimens Only)

16.1 Calculate the mean shear strength of unaged and aged groups as follows:

$$\bar{X}_i = \frac{\Sigma X_i}{r} \quad (1)$$

where:

$\bar{X}_i$ = mean shear strength,
X_i = individual shear strengths of specimens in a group, and
r = number of specimens in the group.

16.2 For an analysis of variance of control and aged groups for differences, see A1.4.

16.3 For Dunnett's *least significant difference* for comparing aged group means with the unaged group mean see A1.5.

17. Report

17.1 Report the following:

17.1.1 Adhesive,

17.1.2 Type of specimen,

17.1.3 Adherends (if lap shear specimens are used),

17.1.4 Adherend surface preparation,

17.1.5 Wood moisture content at bonding,

17.1.6 Cure conditions,

17.1.7 Type of aging test,

17.1.8 Maximum duration of exposure,

17.1.9 Intermediate test intervals,

17.1.10 Aging temperature,

17.1.11 Dates of original and final determinations of physical properties,

17.1.12 Adhesive layer thickness,

17.1.13 Results calculated in accordance with Section 16 (tension shear specimens only),

17.1.14 Amount of wood failure before aging and after every aging interval, expressed as a percent of the bonded area (tension shear specimens only),

17.1.15 Appearances and feel of the adhesive before aging and after each interval of aging, and

17.1.16 All observed and recorded data on which the calculations are based.

18. Precision and Bias

18.1 At the present time there is no basis for a statement concerning the repeatability or reproducibility of this test method. Such information may be available at some future date since the practice provides for statistical analyses and reporting of strength test variability.

18.2 No means have yet been devised for assessing the accuracy of predictions of long-term performance from a short-term accelerated exposure without actual long-time service test results. An indication of bias is possible by comparing the short-term aging result of an unproven material with the short-term aging result of a material with a proven service record.

19. Keywords

19.1 accelerated aging; lapshear; oxygen pressure; shear strength

ANNEX

(Mandatory Information)

A1. METHOD FOR DETERMINING SIGNIFICANT LOSS OF STRENGTH IN BONDED SPECIMENS EXPOSED OXYGEN-PRESSURE AGING

A1.1 Symbols

i = a number indicating one of the aged groups i | 1, 2, ... , t
t = the number of aged groups
j = a number representing one of the replicates within an aged group
r = the number of replicates in any aged group
$\bar{X}_{ij}$ = the shear strength of the jth replicate in the ith group
ΣX_{ij} = the sum of the shear strengths of the j replicates in the ith group, for example, ΣX_{1j} the sum of the shear strength of the j replicates in the first aged group
ΣX_{ij} = the sum the shear strength of the j replicates in all the i groups
ΣX_{ij}^2 = the square of the shear strength of each replicate totaled for every replicate in all the groups
$(\Sigma X_{ij})^2$ = the total of the shear strengths of all the replicates in all the groups, squared
$\bar{X}_{i\cdot}$ = the mean shear strength of the j replicates in the ith group
$\bar{X}_{c\cdot}$ = the mean shear strength of the j replicates in the unaged control group

A1.2 Group Means

$$\bar{X}_{i\cdot} = \frac{\sum_j X_{ij}}{r} \tag{A1.1}$$

A1.3 Difference of Aged Group Means from the Control Group Mean

$$D = |\ \bar{X}_{i\cdot} - \bar{X}_{c\cdot}\ | \text{ all } i \text{ except } i = c \tag{A1.2}$$

A1.4 Analysis of Variance

A1.4.1 *Correction Term*:

$$C = \frac{(\sum_{ij} X_{ij})^2}{rt} \tag{A1.3}$$

A1.4.2 *Total Sum of Squares*:

$$TSS = \sum_{ij} X_{ij}^2 - C \tag{A1.4}$$

A1.4.3 *Group Sum of Squares:*

$$GSS = \frac{(\sum_j X_1 \cdot)^2 + (\sum_j X_2 \cdot)^2 + \ldots . + (\sum_j X_t \cdot)^2}{r} \tag{A1.5}$$

A1.4.4 *Error Sum of Squares*:

$$ESS = TSS - GSS \tag{A1.6}$$

A1.4.5 *Degrees of Freedom*:

$$\begin{aligned} &\text{Groups} && df_G = t - 1 \\ &\text{Error} && df_E = t(r-1) \\ &\text{Total} && bdf_T = rt - 1 \end{aligned} \tag{A1.7}$$

A1.4.6 *"F" Test—See Table A1.1.* Find the mean square for each source of variation by dividing the sum of squares by the appropriate degrees of freedom for each source.

$$\text{Mean square} = \frac{\text{sum of squares}}{df} \tag{A1.8}$$

Calculated F

$$F = \frac{\text{group mean square}}{\text{error mean square}} \tag{A1.9}$$

Tables of "F" can be found in most textbooks on statistics. The correct value is found by locating the number in the table corresponding to the greater and lesser mean square's degrees of freedom. Generally in this test method the greater mean square has 5 degrees of freedom and the lesser mean square has

TABLE A1.1 "*F*" Test

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Square	Calculated "*F*"	Tabular "*F*" Confidence Level 95 %	Tabular "*F*" Confidence Level 99 %
Groups						
Error						
Total						

24 degrees of freedom. Entering the table of "*F*'' with 5 and 24 degrees of freedom "*F*'' = 2.62 at the 95 % level of probability and "*F*'' = 3.90 at the 99 % level of probability.

A1.4.7 *Comparing Calculated and Tabular" F'' Values*—If the calculated "*F*'' value exceeds the tabular "*F*'' value at either the 95 or 99 % level of probability, then it is reasonable to say that a significant difference (as opposed to a difference due to error alone) exists somewhere between one or more pairs of the groups with a level of confidence on the statement of either 95 or 99 %.

A1.5 Dunnett's Multiple Comparisons Test[3]

A1.5.1 Where the "*F*'' test indicates that differences exist among the groups it does not indicate where the difference occurs. To determine where the difference occurs, a multiple comparison test should be used to control the overall confidence level and protect against making erroneous statements about the totality of significant differences among a set of means. (Note that "Student's *t*'' test does not provide this control.) Dunnett's multiple comparison test is especially well adapted to comparing all means with a control.

A1.5.2 *Dunnett's Least Significant Difference*:

$$d' = t_{\text{Dunnett}} \times \sqrt{\frac{2\ (\text{error mean square})}{r}} \qquad \text{(A1.10)}$$

where $t_{\text{Dunnett}}$is selected from a table of "*t*'' values for onesided comparisons between *t* (Dunnett uses *p*) treatment means and a control for a joint confidence coefficient of 95 or 99 %. For this particular test method, with five means (excluding the control) and with 24 error degrees of freedom, Dunnett's *t* values are 2.76 and 3.45 at confidence levels of 95 and 99 %, respectively. The table is published in Dunnett's original article and in Steel and Torie.[4] Once Dunnett's *d′* has been calculated the means are compared as below:

Exposure	Mean	Difference from the Control Mean	Dunnett's *d′* 95 %	99 %
Control	433			
200 h	409	24	141	174
400 h	291	142[a]	141	174
600 h	232	201[b]	141	174
800 h	122	311[b]	141	174
1000 h	71	362[b]	141	174

Differences larger than *d′* are significantly different from the control mean.[a] and[b] indicate these differences are significantly different from the control mean at the 95 and 99 % level of confidence, respectively. If joint strength is highly variable, the calculated *least significant difference* may be larger than desired. For example, one may wish to detect a 10 % difference in strength between the unaged control specimens and aged specimens after some interval, but because the strength variability is large, the calculated *least significant difference* is greater than 10 % at the desired level of confidence. Increasing the number of specimens will reduce the size of the *least significant difference* and thus the minimum strength loss which can be termed significant.

[3] Dunnett, C. W., "A Comparison Procedure for Comparing Several Treatments with a Control," *Journal of American Statistical Association*, JSTNA, Vol 50, 1955, pp. 1096–1121.

[4] Steel, R. G. D., and Torie, J. H., *Principles and Procedures of Statistics*, McGraw-Hill Book Co., New York, NY, 1960.