This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D3632 – 98 (Reapproved 2019)

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

This standard is issued under the fixed designation D3632; 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 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D143 Test Methods for Small Clear Specimens of Timber

D454 Test Method for Rubber Deterioration by Heat and Air Pressure

D572 Test Method for Rubber—Deterioration by Heat and Oxygen

D573 Test Method for Rubber—Deterioration in an Air Oven

D907 Terminology of Adhesives

D2339 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 D907.

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.

[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.

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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.

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 D572. 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 D454), 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 D2339.

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 D573.

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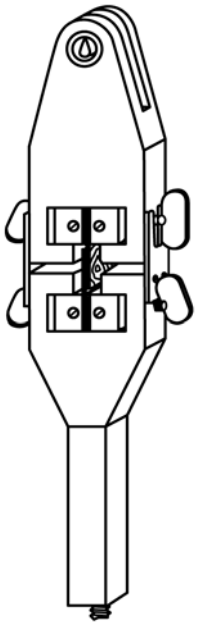


FIG. 1 Grips and Jaws

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.

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

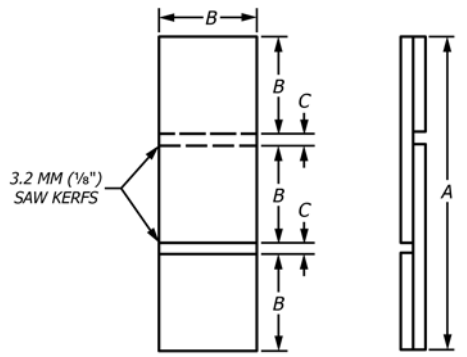
8.1 *Sampling Method*—When several test specimen panels are made or films cast and groups of individual specimens are aged for different time intervals, mix all the specimens in a box and draw at random from the box for assignment to a given group.

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

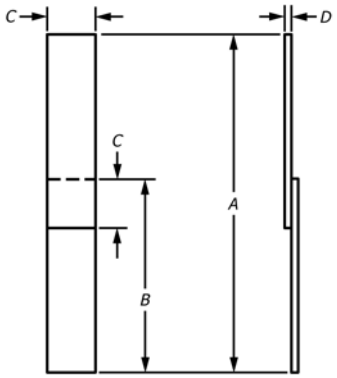
9.1 Lap-shear test specimens must be made from the same adherends and adhesive materials that are actually used in service. A tension lap-shear specimen made like the ones in Fig. 2 and Fig. 3 is recommended. Methods for preparing wood-to-wood specimens are similar to those described in Test Method D2339 with modifications to accommodate mastic consistency adhesives.

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



	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

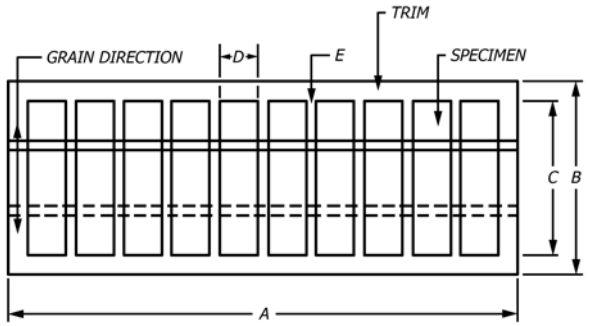


	Metric Equivalents			
	Dimension	Tolerance	Dimension	Tolerance
	mm		in.	
A	178	±0.25	7.0	±0.010
B	102	±0.25	4.0	±0.010
C	25.4	±0.25	1.0	±0.010
D	3.17	±0.10	0.125	±0.004

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

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 D143.

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	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° 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