Designation: D4446/D4446M – 13

Standard Test Method for Anti-Swelling Effectiveness of Water-Repellent Formulations and Differential Swelling of Untreated Wood When Exposed to Liquid Water Environments[1]

This standard is issued under the fixed designation D4446/D4446M; 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 is designed to evaluate the effectiveness of water-repellent compositions for retarding dimensional changes in coated wood submerged in water. It can also be used to measure the differential swelling of untreated wood when exposed to liquid water environments. The compositions tested are designed to be mixed until uniform and applied by brush, roller, dip or spray to an exterior wood surface.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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. Summary of Method

2.1 Wood samples in the form of elongated slats that represent the timber species or product/treatment combination to be evaluated are exposed in soak containers. The elongated slats are immersed in the water-repellent formulation, conditioned with appropriate weighing, then subjected to immersion in distilled water for a prescribed period. The untreated slats omit the immersion in the water-repellent formulation. The swelling resulting from immersion for the selected time period is determined by reading a dial gage calibrated in increments of 0.025 mm [0.001 in.].

2.2 A water repellent efficiency of 60 % is required to pass this test.

3. Significance and Use

3.1 This test method is useful in determining the relative anti-swelling efficiency of various water-repellent formulations when applied to wood. It is the initial means of estimating the ability of water-repellent treated wood to perform satisfactorily when exposed to liquid water environments.

3.2 The swelling differences of untreated wood species when subjected to water immersion can also be determined by this test method.

3.3 This method is a basic screening test and thus provides an initial determination of the anti-swelling efficiency of water repellents. It is a qualitative method designed to provide a reproducible means of establishing: (*1*) the anti-swelling efficiency of water-repellent formulations, and (*2*) the relative swelling of untreated wood species when both are exposed to liquid water environments.

4. Apparatus

4.1 *Conditioning Room or Chamber,* having a controlled temperature of 23 ± 2°C [73.5 ± 3.5°F], and a controlled relative humidity of 50 ± 5 %. This room is used to establish a uniform moisture content in the test specimens.*In all studies the temperature and relative humidity selected by the investigator must be stated and must remain constant throughout a given conditioning and test period.*

4.2 *Balance,* sensitive to at least 0.01 g.

4.3 *Treating Tank* (Fig. 1).

4.4 *Swellometer,* as illustrated in Fig. 2, Fig. 3, and Fig. 4. Apparatus can be built to be used for both 5 in. and 10 in. wafers.

5. Test Specimens

5.1 Wood used for these tests must be straight-grained, flat-sawn, clear, kiln-dried Ponderosa pine sapwood or other suitable species. Cut the parent boards in a manner to give specimens 6 mm [¼ in.] in the longitudinal dimension, 38 mm [1½ in.] in the radial dimension, and 254 mm [10 in.] or 127 mm [5 in.] in the tangential dimension. Cut with a sharp,

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.42 on Architectural Coatings.

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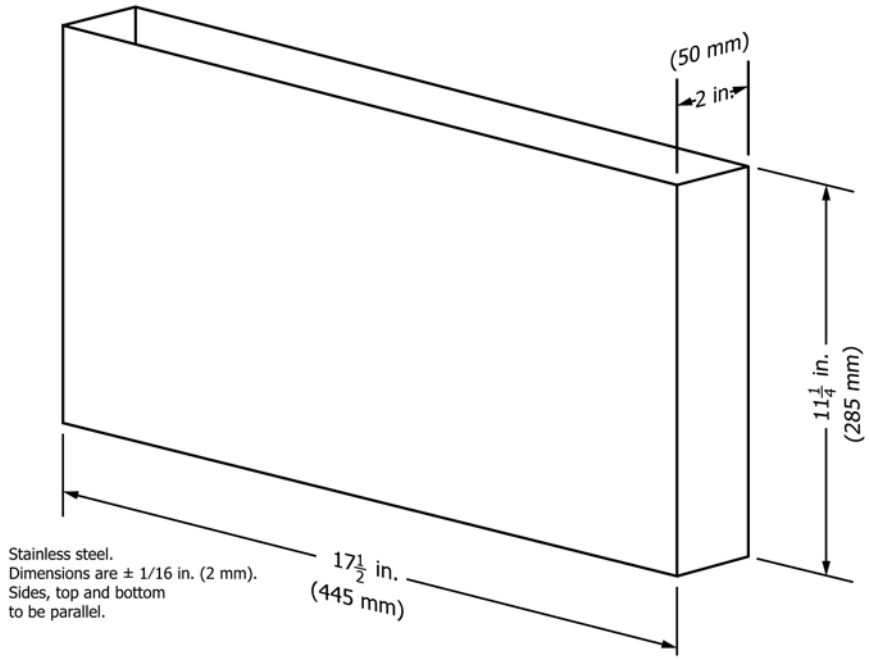

FIG. 1 Treating Tank—Five Samples Tested Simultaneously

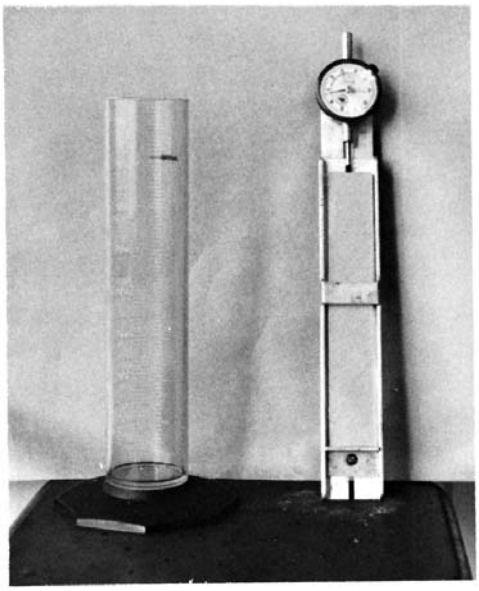

FIG. 2 Single Test Swellometer Tank and One Sample

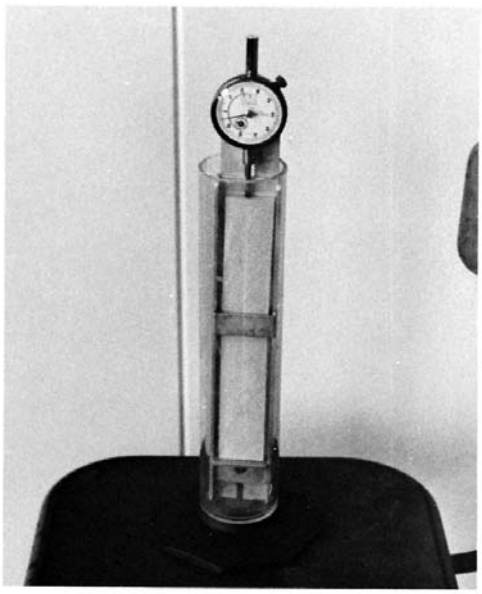

FIG. 3 Single Test Swellometer Tank—With One Sample In Place

fine-toothed saw to obtain as smooth a surface as possible without sanding. Number each specimen for identification and reference.

5.1.1 Selection of Ponderosa pine sapwood can be assured by the following quick chemical test: (*1*) prepare a solution of 5 g of benzidine in 25 g of hydrochloric acid and 970 g of water, and (*2*) prepare a second solution consisting of a 10 % concentration of sodium nitrite in water. When the test is to be made, pour equal amounts of the two solutions together and then apply to the wood surface by means of brushing or dipping. The sapwood immediately shows a yellowish color, while the heartwood turns dark red-brown. The colors remain distinct after drying.

5.2 Determine the swelling of an untreated specimen from each parent board before testing begins. Do this by equilibrating the specimen in accordance with 7.1 and then testing in accordance with 7.5.1. The swelling shall not be less than 8.25 mm [0.325 in.]. See Table 1.

6. Formulations

6.1 Treat the test specimens with the ready-to-use formulation as advocated by the manufacturer of the formulation.

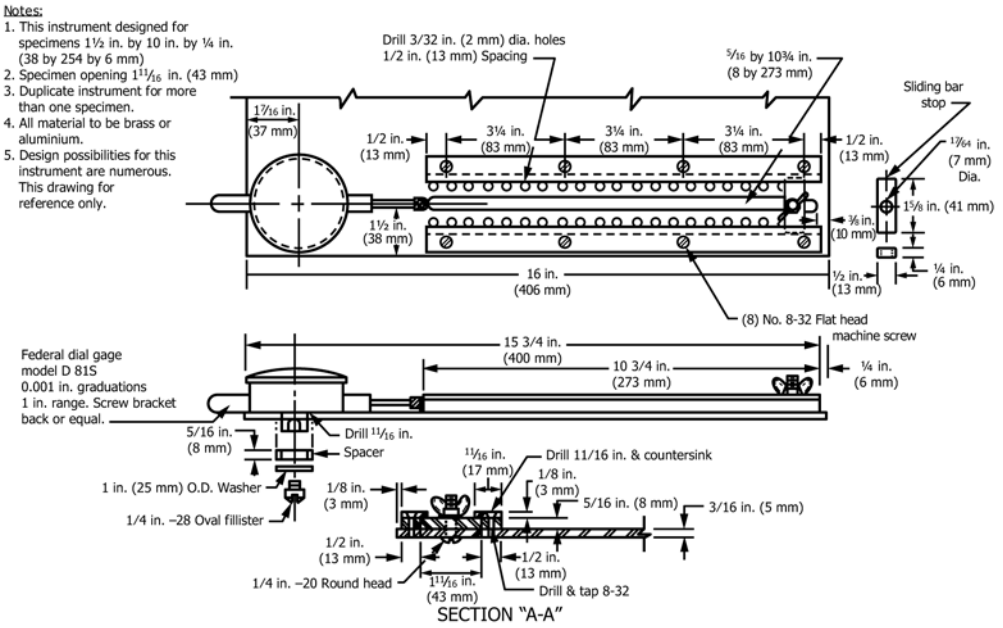

FIG. 4 Swellometer

TABLE 1 Natural Tangential Swelling of Selected Species[A]

Species	Tangential Swelling, %
Pine, ponderosa	6.2
southern yellow	7.5
Oak, red	9.5
white	10.5
Maple	8.3
Beech	11.9
Douglas-fir	7.6

[A] Source: "Wood Handbook," *Agriculture Handbook No. 72*, USDA, Forest Service, Washington, DC, 1974.

6.2 Test, experimental or control formulations may be added to the test as desired.

6.3 Establish adequate records, reference samples and identification to permit independent review and analysis.

7. Test Procedure

7.1 Cut two adjacent specimens, both either tangentially or longitudinally, from each board. Specimen pairs from ten different boards (20 test specimens in all) make a set (in accordance with 7.3 and 7.4) for testing the water-repellency of one formulation at one retention level. Each additional retention level requires ten additional samples. Store the test specimens with space between each one on a raised screen or rack in the conditioning room or chamber until they reach constant weight as determined by a uniform weight over a 48-h period.

7.2 In comparing different water-repellent formulations, it is necessary to determine the quantity of formulation picked up and retained by the test specimen. If the specimens are weighed before treatment and again immediately after being dipped in the water-repellent formulation the difference is the weight of the formulation absorbed. To remove the unabsorbed treatment from the specimens it is recommended for water-thin viscosity type solutions to be stood on edge and rotated to ensure a uniform film. For a thicker viscosity treatment it is recommended to use a treatment saturated brush or lint free cloth to remove excess treatment to ensure a uniform film. Then, if reweighed just before being placed in the swellometer, the difference between excess treatment weight and the final weight is the weight of the material actually retained by the specimen.

7.3 *First Test:*

7.3.1 Maintain the formulation to be tested at a minimum temperature of 21°C [70°F] and thoroughly mix immediately before immersing the test specimens.

7.3.2 Fully immerse in the Treating Tank (Fig. 1) *five* of the ten test specimens (one from each board) in the formulation to be tested. The immersion time for solvent-based formulations shall be 30 s. Because of the small size of the specimens (6 mm [¼ in.] thick) a 30-s immersion period is adequate for formulation pickup. This 30-s immersion period will reflect appropriate screening test results, but is not intended to reflect the proper treatment time for use on commercial products. The immersion time for water-based formulations shall be 3 min. Treat the test specimens while in the conditioning room/chamber or within 30 s after removal from the conditioning room/chamber. Five untreated specimens, one from each of the boards, serve as controls.

7.3.3 Place both the five treated and the five untreated specimens separately on a raised screen or other suitable rack that will allow free access of air to them, and place the specimens in the conditioning room or chamber. After 24 h, remove all specimens from the conditioning room or chamber and allow them to volatilize under normal laboratory conditions for 3 days. Then place all ten specimens back into the conditioning room or chamber until they reach constant weight.

TABLE 2 10 in. Wafer Swelling (%)

Fluid	Average $\bar{X}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
A	76.20	1.80	2.96	5.03	8.30
B	63.17	1.92	3.50	5.36	9.80
C	73.84	3.25	4.10	9.09	11.49
D	9.45	1.25	3.71	3.50	10.38

TABLE 3 5 in. Wafer Swelling (%)

Fluid	Average $\bar{X}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
A	83.26	2.42	3.69	6.78	10.32
B	66.18	11.52	13.25	32.25	37.11
C	72.35	10.32	11.27	28.88	31.54
D	7.79	3.24	3.96	9.08	11.08

7.4 *Second Test:*

7.4.1 Pour the formulation remaining after treating (7.3.2) into a sealable glass container and allow to stand for 2 months at a temperature of 21 to 38°C [70 to 100°F]. After this 2-month period, decant and again test the formulation without shaking in accordance with 7.3. The formulation must pass both tests.

7.5 *Testing the Water-Repellency:*

7.5.1 Compare the swelling of the untreated controls with the swelling of the matched treated specimens after each has been submerged in distilled water. Measure the swelling by means of the Swellometer (illustrated in Fig. 2, Fig. 3, and Fig. 4). Insert a specimen in the guides of the swellometer, placed so that one end bears firmly on the adjusted base and the other end contacts the plunger of the dial. Adjustments can be made to the sliding bar to allow for use of either the 5 in. or 10 in. wafer. Take a reading of the dial before immersing the sample in the water. Arrange the instrumented sample in the container of distilled water maintained at 24 ± 3°C [75 ± 5°F] so that the specimen is completely submerged (but the dial and dial stem are dry) for 30 min. At the end of the 30-min period, take a second dial reading and note the difference. Test the group of five untreated controls and the group of five treated specimens separately using fresh distilled water for each group.

8. Evaluation of Results

8.1 Results from the first (ten samples, that is, five pairs of treated specimens and controls) and second tests (ten samples) shall be separately recorded and computed. The difference between the swelling of each treated specimen and the swelling of its matching untreated control specimen is divided by the swelling of the untreated control specimen and multiplied by 100. The average of the five percentages calculated for each test represents the percent effectiveness of the water-repellent formulation per test. The average of the results from both tests represents the final percent of water-repellent effectiveness of the formulation.

8.2 Historically a minimum water-repellent efficiency of 60 % is required to pass this test method.

9. Precision and Bias[2]

9.1 The precision of this test method is based on an interlaboratory study of D4446, Test Method for Anti-Swelling Effectiveness of Water-Repellent Formulations and Differential Swelling of Untreated Wood When Exposed to Liquid Water Environments, conducted from 2006–2007. Each of six laboratories tested the effectiveness of four different water repellent formulations on 10 in. and 5 in. wafers. Every "test result" represents an individual determination. All laboratories obtained two replicate test results for every material.[2] (See Table 2 and Table 3.)

9.1.1 *Repeatability (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

9.1.2 *Reproducibility (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

9.1.3 Any judgment in accordance with these two statements would have an approximate 95 % probability of being correct.

9.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

9.3 The precision statement was determined through statistical examination of 94 results, from six laboratories, on four materials. These four fluids were described as the following:

[2] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1175. Contact ASTM Customer Service at service@astm.org.

Material A: 8.4 % solids oil based clear water repellent

Material B: clear 5 % paraffin wax petroleum distillate solution

Material C: 10 % solids water based clear water repellent

Material D: 39 % solids paraffin wax emulsion

9.3.1 To judge the equivalency of two test results, it is recommended to choose the water repellent material closest in characteristics to the test material.

10. Keywords

10.1 anti-swelling; liquid water; water repellent