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: D7477 – 16 (Reapproved 2021)

Standard Test Method for Determining the Area Stability of Wet Blue Submersed in Boiling Water[1]

This standard is issued under the fixed designation D7477; 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 provides a standard procedure for determination of the dimensional stability or area shrinkage of a specimen of Wet Blue that is submersed in boiling water for a specified time period. This test method is applicable to all types of Wet Blue.

1.2 The values given in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

1.3 This test method does not apply to Wet White.

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]

D6659 Practice for Sampling and Preparation of Wet Blue and Wet White for Physical and Chemical Tests

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Summary of Test Method

3.1 A sample cutting of Wet Blue is either taken directly out of the tanning drum or else is pre-soaked in water for 30 min or until it is completely re-hydrated (see 9.1 for details on re-hydration). The specimen to be tested is cut out from this thoroughly hydrated sample cutting. The test specimen is then totally submerged and suspended in boiling water. The test specimen is removed 3.0 min after the water temperature reaches 100 °C and begins to re-boil. As soon as the specimen has cooled sufficiently to allow comfortable handling the area loss is determined.

4. Significance and Use

4.1 Determination of the hydro-thermal area stability of Wet Blue provides information concerning the efficacy of the tanning process as well as the adequacy of the Wet Blue for intended end use applications where area stability is a particular requirement. Relative area stability of chrome-tanned leather is a requirement for many applications such as bookbinding, shoe and boot components, upholstery, seals and gaskets, etc.

4.2 This test method is suitable for use in development work and process control in the tannery and for specification testing of Wet Blue for domestic and international commercial purposes.

5. Apparatus

5.1 *Beaker,* standard, IL capacity. Other suitable containers may be used so long as the dimensions are sufficiently large to enable suspension of the completely immersed test specimen with no contact occurring with the sides and bottom of the container during the test. Particularly when non-standard test specimens are used, the size and shape requirements of the container are dependent on the dimensions of the specimen to be tested.

5.2 *Thermometer,* with a minimum scale reading to +110 °C, graduated in 1 °C, and having a 0.5 °C tolerance.

5.3 *Timer,* with minimum 3 min capacity and 1.0 s resolution.

5.4 *Metal die,* to cut specimens. The die should be constructed of highly corrosion resistant alloy metal and must be maintained in a clean and sharp condition to minimize distortion of the Wet Blue sample that may occur during the specimen cutting operation. The recommended specimen is a

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.02 on Wet Blue.

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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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square 76.2 by 76.2 mm (3.00 by 3.00 in.). However, other size and shape specimens can be used so long as the requirements of Section 8 are met.

5.5 *Measuring scale,* ruler longer than the greatest dimension of the test specimen, divided in millimeters (1⁄32 in.).

5.6 *Marking pen,* Suitable water-proof marker or pen for marking the indicator points, on the hydrated Wet Blue grain surface, to be used for measuring the specimen dimensions.

6. Reagents and Materials

6.1 *Water,* distilled or de-ionized laboratory water should be used, especially if there is any possibility of constituents in the available tap water affecting the results of this test.

6.2 *Glycerin (Glycerol),* technical grade is adequate.

6.3 *Salt (Sodium Chloride),* common table salt is adequate.

7. Hazards

7.1 All reagents and chemicals should be handled with care. Before using any chemical, read and follow all safety precautions and instructions on the manufacturer's label or MSDS (Material Safety Data Sheet).

8. Test Specimen

8.1 The original sample cutting to be pre-soaked and the test specimen cut from it shall be taken from the Wet Blue according to Practice D6659. Specifically, for a hide or side the cutting shall be taken from the "a" test area (the kidney area) of a hide or side. The number of samples to be tested shall be as described in Practice D6659 and need not be more than 12 per 50 000 ft^2 of Wet Blue stock.

8.2 The sample cutting taken from the Wet Blue should be large enough to permit the test specimen to be cut out with a fresh edge no closer than 13 mm (0.5 in) to an edge of the original cutting. The sample cutting from which the test specimen should be taken, should have minimum dimensions of approximately 101 by 101 mm (4 by 4 in.). A cutting taken straight out of the tanning drum prior to wringing need not be pre-soaked and the test specimen can be cut out directly from this sample cutting. A cutting taken from Wet Blue at any point from the wringing operation forward will need to be pre-soaked (see 9.1 for details on re-hydration). The standard test specimen shall be a square 76.0 mm (3.00 in.) on edge. Other size and shape specimens can be used. It is recommended that for non-standard test specimens the minimum dimension (for a side of a rectangle or diameter of a circle) be 51.0 mm (2.0 in.) and the maximum dimension (for a side or diameter) be 102 mm (4.0 in.). Before a test specimen of non-standard dimensions may be used with this test method it must be rigorously demonstrated that the non-standard specimen gives identical results to that of the standard specimen for the particular Wet Blue being tested.

NOTE 1—As an example of a non-standard specimen, a convenient size for test specimens could be a square exactly 100 mm on edge. Then, on this specimen, a loss of 1 mm in each dimension is approximately (but not exactly) equal to 1 % loss in area. For instance, if a specimen experienced a loss of 4 mm in one dimension (actual measurement after the test was 96 mm determined by averaging the measured length of the two edges parallel to that dimension) and a loss of 6 mm in the other dimension (actual measurement after the test was 94 mm determined by averaging the measured length of the two edges parallel to that dimension) then the approximate area loss for that specimen would be 10 % (that is, 4 + 6 %). However, multiplication and subtraction followed by division [(10 000 – 9024 mm^2)/10 000 mm^2] yields 9.76 % for the actual area loss. For most applications using this method, the area loss determined by simply adding the dimensional loss values will give adequate results without going through the mathematical calculations.

8.3 Appropriate small holes may be punched in the test specimen to facilitate the suspension of the submerged specimen in the beaker of water during the test if J-hooks or S-hooks are used in conjunction with a rod across the top of the beaker. Alternative practices may provide suspension of the specimen by employing a net type structure attached to a rod across the top of the beaker.

9. Procedure

9.1 Cuttings that are taken directly out of the tanning drum prior to dumping and wringing need not be re-hydrated. Thorough hydration of the original sample cutting taken from Wet Blue after wringing shall be ensured by pre-soaking this cutting for a minimum 30 min or until the cutting is completely re-hydrated. All soaking for re-hydration should take place under ambient temperature conditions at the testing location. Cuttings taken from Wet Blue after wringing and that have not dried out usually re-hydrate within 30 min. Wet blue cuttings that have dried out somewhat may require significantly more than 30 min to become completely re-hydrated. Wet blue cuttings that have become significantly air-dried may require soaking periods as long as overnight to re-hydrate completely. Re-hydration of the sample cutting can be accomplished by soaking in a container or tray so long as the cutting is completely covered with water. Bending or flexing the cutting while it is completely immersed in water or application of a vacuum to facilitate removal of entrapped air may facilitate re-hydration. Complete hydration should be determined by weighing the sample at appropriate intervals during the soaking process until constant weight is achieved. Appropriate intervals between weighings could be 10-15 min for cuttings that have been wrung but not dried out and 30-60 min or more for cuttings that have dried out significantly. Constant weight is achieved when the difference between successive weighings is less than ±0.1 g. After the cutting has been completely re-hydrated, the test specimen can be cut out from it.

9.2 Before the test specimen is cut out from the completely hydrated cutting, the beaker of boiling water shall be prepared so it is ready when the test specimen is cut out. A 1L beaker or other container shall be filled with sufficient water to generously cover the test specimen when fully immersed, but leaving sufficient room to enable the immersion and removal of the test specimen without causing spillage of excess water onto the hotplate surface. The water shall be brought to boil. If the temperature is not exactly 100 ± 0.5 ºC sufficient glycerin or salt shall be added to bring the boiling point up to but not exceeding 100 ºC.

9.3 Prior to testing, the original area of the test specimen shall be determined. Measurement of the edge length of the test specimen should be made a distance back from the physical edge of the specimen in order to avoid problems with deformed

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edges that may occur as a result of the testing conditions. Mark the grain surface of the specimen with indelible ink (see 5.6) to indicate the points of the original measurements. Make two measurements of each dimension, back 13 mm (0.50 in.) from the edges of the specimen, and average them to calculate the area (A_0 = [(S_{1a} + S_{1b})/2 x (S_{2a} + S_{2b})/2]). The known dimensions of the cutting die may be used to calculate the original area of the test specimen; however, in order to facilitate the measurement determinations after testing, the grain surface of the specimen shall still be marked with indelible ink prior to testing to indicate the points of measurement as indicated above.

9.4 The test specimen shall be totally submersed in the boiling water and suspended either by a hook or a net type device to prevent it contacting the bottom and sides of the container. Start the timer when the water has again reached 100 ± 0.5 ºC and begins to re-boil with the same vigor it exhibited prior to immersion of the specimen. After 3.0 min ± 5 s remove the specimen from the water, cool it to a comfortable working temperature by holding it under running cold water, and place it on a flat non-corrosive surface.

NOTE 2—Shortly after the test specimen is placed into the boiling water air is released from the specimen and can cause the water to appear to be boiling again. Before the timer is started, it is imperative to make sure the temperature of the water is the same as it was before the specimen was introduced and that the water is boiling with the same intensity as it was initially.

9.5 Re-measure the dimensions at the marked points and calculate the area with the new values for the averaged dimensions (A_F = [(S_{1a} + S_{1b})/2 x (S_{2a} + S_{2b})/2]). A negative value for the change in area ($A_0 - A_F$) indicates that the specimen expanded or stretched rather than contracted or shrunk.

9.6 When multiple samples must be tested to a known quality control or specification requirement on a pass/fail basis it may be more efficient to construct a rigid template with an outline of the exact dimensions of the test specimen out of non-corrosive material such as poly (methyl methacrylate) (PMMA). Then make an additional outline on the template of the percent limit of the area loss acceptable for the specification. When using this measurement method option it is imperative to check the original dimensions of the specimen against the template outline for the original dimensions prior to testing. If the specimen edges do not match the template outline, that specimen must be discarded and another specimen cut until the edges do match the template outline. After testing, place the specimen on the template and determine whether the dimensions are greater than the limit outline (Pass) or less than the limit outline. If less than limit outline calculate the area loss as in Fig. 1.

10. Calculation

10.1 Calculate the area change as follows:

$$\text{Percent area change} = \left[\left(A_0 - A_F\right) \times 100\right]/A_0$$

where:

A_0 = original weight of the specimen, and
A_F = area of the specimen after testing.

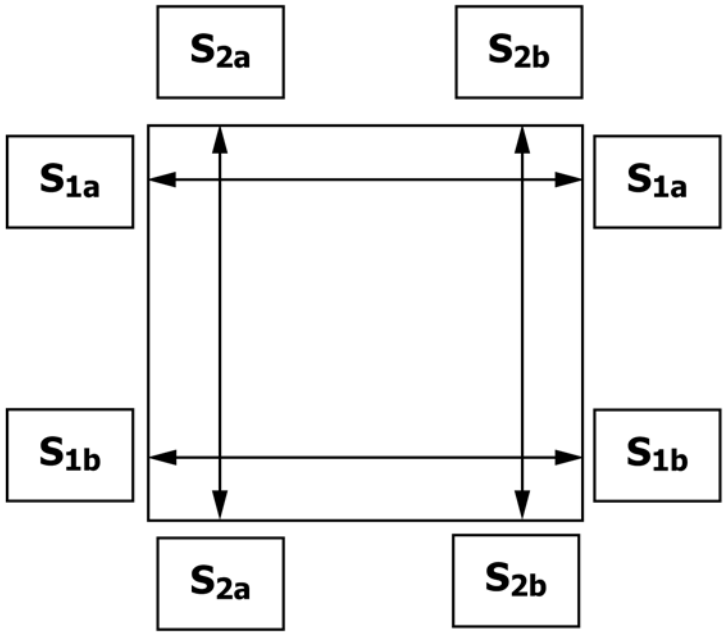

FIG. 1 Measure Area as:

11. Report

11.1 Report the area change to the nearest 0.1 %.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is based on a repeatability study of WK19533, New Test Method for Determining the Area Stability of Wet Blue Submersed in Boiling Water, conducted in 2007. One laboratory compared six different materials under two different conditions ("as received" and "dried out"), at one temperature. Every "test result" represents an individual determination. The laboratory was asked to submit five replicate test results, for each analysis and condition. Except for the limited number of reporting laboratories, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D31-1011.[3]

12.1.1 *Repeatability Limit (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.

12.1.1.1 Repeatability limits are listed in Table 1 and Table 2.

12.1.2 *Reproducibilty Limit (R)*—" *R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.2.1 Reproducibility limits were not addressed in this study.

[3] Available from ASTM International Headquarters. Request RR:D31-1011.