



Test Method for Shrinkage Temperature of Wet Blue and Wet White¹

This standard is issued under the fixed designation D8042; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of the shrinkage temperature of all types of Wet Blue and Wet White. The heating medium is water when the shrinkage temperature is at or below 98°C. The heating medium is a glycerine-water solution when the shrinkage temperature is above 98°C.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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

[D1517 Terminology Relating to Leather](#)

[D6076 Test Method for Shrinkage Temperature of Leather](#)

3. Terminology

3.1 *Definitions*—For definitions of general leather terms used in this test method, refer to Terminology [D1517](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *shrink*, *v*—to contract or become smaller.

3.2.2 *shrinkage*, *n*—the temperature at which noticeable shrinkage occurs when a Wet Blue or Wet White specimen is gradually heated in an aqueous medium.

4. Summary of Test Method

4.1 A Wet Blue or Wet White specimen is thoroughly soaked in the aqueous medium that will be used to heat the specimen for this test method. This specimen is then fastened between two clamps (one fixed and one movable) and immersed in the aqueous medium. The aqueous medium is

gradually heated until the specified temperature is reached without shrinkage or until shrinkage is indicated by a deflection of the dial needle which is attached to the movable clamp.

5. Significance and Use

5.1 This test method is designed to determine the temperature at which the Wet Blue or Wet White specimen experiences shrinkage. In this test method, shrinkage occurs as a result of hydrothermal denaturation of the collagen protein molecules which make up the fiber structure of the Wet Blue or Wet White. The shrinkage temperature of Wet Blue or Wet White is influenced by many different factors, most of which appear to affect the number and nature of crosslinking interactions between adjacent polypeptide chains of the collagen protein molecules. The value of the shrinkage temperature of Wet Blue or Wet White is commonly used as an indicator of the type of tannage or the degree of tannage, or both, of that particular Wet Blue or Wet White (especially for the more hydrothermally stable tannages such as chrome tannage).

6. Apparatus

6.1 *Stand*, for supporting the testing equipment.

6.2 *Indicating Device*, either of the two devices listed as follows:

6.2.1 *This Shrinkage Meter Dial*—a dial indicator having a face divided into 360 to 380°, with one revolution of the dial hand corresponding to 12.7 mm (0.5 in.) of specimen movement, and with leeway for four revolutions of the dial hand.³

6.2.2 *AGD (American Gage Design) Dial Indicator*, a mechanical device capable of registering on a scale a reading of the shrinkage or swelling movement of the specimen in a ratio of at least 25 units of dial movement (or greater) to 1 unit of specimen movement, and shall accurately detect by movement of the dial needle the point at which shrinkage begins. The

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.

Current edition approved June 1, 2016. Published June 2016. DOI: 10.1520/D8042-16.

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 A shrinkage meter apparatus setup meeting these specific requirements was formerly available from the Arthur H. Thomas Co., Philadelphia, PA. Used apparatus of that type may still be available from tanneries or laboratories that have gone out of business or from used equipment dealers. The sole source of supply of parts and dials to build a meter meeting the specifications known to the committee at this time is Schap Specialty Machine, Inc., 17309 Taft Road, Spring Lake, MI 49456. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

indicator shall have a spindle capable of attachment to the specimen and to the adjustable weight assembly.⁴

6.3 *Immersion Heater*, 250 to 500 W, with a heating element not longer than 140 mm (5½ in.) and a depth of immersion not in excess of 152 mm (6 in.).

6.4 *Variable Speed Stirrer*, with small blades.

6.5 *Spring-loaded Clamps*, (alligator type), two, mounted vertically one above the other and sufficiently far apart for gripping the full width of the specimen at the ends. The bottom clamp shall be fastened to the end of a U-shaped rod that can be turned sideways. The upper clamp shall be movable sideways as well as up and down.

6.6 *Metal Supporting Plate*, with apertures for the stirrer, thermometer, immersion heater, and the rod fastened to the upper specimen clamp.

6.7 *Variable Transformer*, capable of controlling the rate of heating (3 to 5°C/min) of the aqueous medium and capable of maintaining a temperature of $98 \pm 0.5^\circ\text{C}$.

6.8 *Thermometer*, capable of reading from 0 to 100°C in 1°C increments with a tolerance of $\pm 0.5^\circ\text{C}$.

6.9 *Tall-form 1-L Beaker*, for holding the aqueous heating medium.

6.10 *Lead Shot*.

6.11 *Indicating Device*, attached to the upper (movable) clamp and equipped with an adjustable weight assembly mounted over a pulley. The weight assembly shall consist of a small bottle (with a lid) containing lead shot that shall be attached to the string running over the pulley. The weight assembly shall be approximately 178 g to counterbalance the weight of the clamp, to overcome any inertia or friction of the indicating device, and to maintain the specimen under a slight tension. The weight shall be adjusted accordingly, so that it will not cause an elongation (prior to shrinkage) of more than 10 %. The device shall register a ratio of 25:1 (or greater) between the scale reading and the shrinkage or elongation of the specimen, and shall accurately detect the point at which shrinkage begins.

6.12 *Metal Die*, for cutting the specimen to the required dimensions.

6.13 *String*, (nylon or polyester) for connecting the upper clamp (or the rod connected to the upper clamp) to the weight assembly and passing over the pulley on the indicating dial.

7. Reagents

7.1 *Glycerine-water Solution*—When glycerine-water medium is specified for use (that is, when the shrinkage tempera-

ture is specified or expected to be greater than 98°C) the solution shall consist of 75 parts glycerine to 25 parts water. The specific gravity of the solution shall be adjusted to 1.19 at 23°C. The glycerine-water solution shall not be heated over 112°C.

7.2 De-ionized water may be used for Wet White which normally shrinks at less than 98°C.

8. Test Specimen

8.1 The test specimen for the test shall be a rectangle of Wet Blue or Wet White 76.0 ± 0.5 mm (3.0 in.) in length by 12.5 ± 0.5 mm (0.5 in.) in width, die cut from the sample unit of Wet Blue or Wet White with the long dimension perpendicular to the backbone line.

8.2 Unless otherwise specified in the material specification, one test specimen shall be tested from each sample unit.

9. Procedure

9.1 Vertically fasten the top and bottom of the specimen in the two clamps of the shrinkage meter, adjusting the specimen to remove any slack. Completely immerse the clamped sample into the heating solution as described in 7.1 or 7.2. The solution may be preheated to 50°C ($\pm 5^\circ\text{C}$). Mark or take note of the location of the indicator needle on the dial as the starting point.

9.2 Increase the temperature of the heating solution at a rate of 3 to 5°C per minute. Shrinkage is indicated when the dial needle moves clockwise. Record the shrinkage temperature at this point. If the needle moves counterclockwise, this is an indication the sample is stretching. Wait until the needle stops moving and mark this location on the dial as the new starting point. Monitor the dial for clockwise movement and record the shrinkage temperature.

10. Report

10.1 Report the following information:

10.1.1 Heating solution used: glycerine/water medium, or water.

10.1.2 The temperature at which shrinkage occurs to the nearest 1°C.

10.1.3 If the temperature of 110°C is reached without shrinkage.

11. Precision and Bias

11.1 This test method is adopted from Test Method D6076. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this test method is adequate for the contemplated use.

12. Keywords

12.1 boil test; degree of tannage; shrinkage; shrinkage temperature; tannage; Wet Blue; Wet White

⁴ The sole source of supply of the dial indicator (Model 2282PCC) known to the committee at this time is B.C. Ames, Inc., Melrose, MA 02176, www.bcames.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

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