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Standard Test Method for Shrinkage Temperature of Leather[1]

This standard is issued under the fixed designation D6076; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the shrinkage temperature of all types of leather. 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 given 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

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*—to contract or become smaller.

3.2.2 *shrinkage*—the temperature at which noticeable shrinkage occurs when a leather specimen is gradually heated in an aqueous medium.

4. Summary of Test Method

4.1 A leather 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 a thoroughly wetted leather 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 leather. The shrinkage temperature of leather 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 leather is commonly used as an indicator of the type of tannage or the degree of tannage, or both, of that particular leather (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 *Theis 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

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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.

[3] A shrinkage meter apparatus setup meeting these specific requirements was formerly available from the Arthur H. Thomas Co., Philadelphia, PA. The minimum number of apparatus setups required for an order makes the cost of new apparatus of this type prohibitive. Used apparatus of this type may still be available from tanneries or laboratories that have gone out of business or from used equipment dealers.

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 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 ± 0.5°C.

6.8 *Thermometer,* with a white background, a scale from –1 or –10°C to +101 or +110°C or +150°C, graduated in 1°C, and with ±0.5°C tolerance.

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) or 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 *Suction Flask,* 1 L.

6.14 *Nylon String,* 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.

[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 Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

7. Reagents

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

8. Test Specimen

8.1 The test specimen for the test shall be a rectangle of leather 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 leather 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 When shrinkage temperatures at or below 98°C are specified, the heating medium shall be water.

9.2 When shrinkage temperatures over 98°C are specified, the heating medium shall be the glycerine-water solution described in 7.1.

9.3 Prepare the specimens as follows prior to mounting in the holding clamps. Place more than 15 specimens in a 1–L suction flask containing approximately 300 mL of the required heating medium at room temperature. Stopper the flask and apply a vacuum for 15 to 60 s and then release for 15 to 60 s. Continue an intermittent vacuum until all the specimens settle to the bottom of the flask. Gently shake the flask each time the vacuum is released to prevent specimens that are ready to sink from being entangled with other specimens. One hour after the last release of vacuum, remove the first specimen from the flask and mount in the shrinkage meter. Keep the other specimens in the flask (containing the heating medium) until tested. The time from when the first specimen is removed from the suction flask and the testing of the last specimen is completed shall not exceed 6 h.

9.4 Fasten the specimen in the two clamps of the shrinkage meter and completely immerse in the heating medium, which has been preheated to 50 ± 5°C. Adjust the indicator to a reference point on the dial by taking the slack out of the specimen. If the specimen should lengthen before it contracts as the temperature of the heating medium is increased at a rate of 3 to 5°C/min, the reference point at which the indicator comes to rest shall be the new reference point for observing shrinkage.

9.5 When a shrinkage temperature of 98°C or below is specified, increase the temperature of the water heating medium at the rate of 3 to 5°C/min until the specimen shrinks or until the specified shrinkage temperature is reached.

9.6 When the material specification requires the specimen not to shrink when subjected to a temperature of 98 ± 0.5°C for 30 s, increase the temperature of the water heating medium at the rate of 3 to 5°C/min until the temperature reaches 98°C. Keep the temperature at 98 ± 0.5°C for 30 s by adjusting the

transformer. If the specimen shrinks during the 30–s period, record the number of seconds that elapse between reaching the temperature of 98°C and shrinkage of the specimen. If the specimen shrinks before the temperature reaches 98°C, record the temperature at which shrinkage occurs.

9.7 When a shrinkage temperature of over 98°C is specified, increase the temperature of the glycerine-water heating medium at the rate of 3 to 5°C/min until the specimen shrinks or until a maximum temperature at 112°C is reached.

10. Report

10.1 Report the following information:

10.1.1 When the specified shrinkage temperature is 98°C or less:

10.1.1.1 If the specimen shrinks before the temperature reaches 98°C, report the temperature at which shrinkage occurs to the nearest 1°C, and

10.1.1.2 If the specified temperature is reached without shrinkage.

10.1.2 When the specification requires the specimen to not shrink for 30 s at 98°C:

10.1.2.1 If the specimen shrinks before the temperature reaches 98°C, report the temperature at which shrinkage occurs to the nearest 1°C,

10.1.2.2 If the specimen shrinks during the 30–s interval, report the number of seconds that elapsed between reaching the temperature of 98°C and shrinkage of the specimen to the nearest second, and

10.1.2.3 If shrinkage of the specimen does not occur during the 30–s interval.

10.1.3 When the specified shrinkage temperature is greater than 98°C:

10.1.3.1 The temperature at which shrinkage occurs to the nearest 1°C, and

10.1.3.2 If the temperature of 112°C is reached without shrinkage.

11. Precision and Bias

11.1 This test method is adopted from the Federal Test Method Standard No. 311, Method 7011.1. This method has long been in use and was approved for publication before the inclusion of precision and bias statements was mandated. 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 degree of tannage; leather; shrinkage; shrinkage temperature; tannage