Designation: D7476 – 08 (Reapproved 2020)

Standard Test Method for Brine Saturation Value of Cured (Salt-Preserved) Hides and Skins[1]

This standard is issued under the fixed designation D7476; 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 covers the estimation of degree of saturation of the brine content of cured (salt-preserved) hides and skins containing 40 % or more moisture.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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:*[2,3]

D6715 Practice for Sampling and Preparation of Fresh or Salt-Preserved (Cured) Hides and Skins for Chemical and Physical Tests

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

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

3. Terminology

3.1 *Definitions:*

3.1.1 The terms and definitions employed within this test method are commonly used in normal laboratory practice and require no special comment.

4. Summary of Test Method

4.1 The moisture (volatile matter by oven drying) and ash content of a composite hide or skin sample are determined. For purposes of calculation the ash is assumed to be equivalent to sodium chloride and the moisture is assumed to be equivalent to water. The percent ratio of ash to moisture is calculated. This ratio times 100 is divided by 35.9 (which is the percent salt to moisture ratio in a saturated sodium chloride (brine) solution at 20°C (68°F)) to estimate the percent of saturation in the brine solution in the hides or skins.

NOTE 1—A saturated solution of sodium chloride at 20°C (68°F) contains 26.4 % by weight of NaCl. Therefore, in 100 g of that saturated solution only 73.6 g (100 – 26.4 g) is contributed by water. As such on a weight basis, 100 parts by weight of water will dissolve 35.9 parts [(100 x 26.4)/73.6] by weight of NaCl.

5. Significance and Use

5.1 This test method is intended to evaluate whether sufficient salt (NaCl) has been retained by the hides or skins to slow down degradation by bacterial (enzymatic) action, and to slow down autolysis by inherent enzymatic action, until they are preserved by a tanning process.

NOTE 2—Osmosis occurs during brine curing because moisture is drawn out by salt through the epidermis.[4] Low moisture (< 40 %) in the hides does not necessarily indicate poor cure. A hide may have low moisture due to osmosis; to increased fat content; or to drying out conditions. In all instances the hides would still show adequate cure if the moisture was saturated to approximately 85 % salt, because the ash: moisture ratio would be adequate.

6. Apparatus

6.1 *Crucible,* 30 to 50 mL, high-form, platinum or porcelain. Prepare crucible by heating it for 15 min in a muffle furnace set at 600 ± 25°C. Cool in a desiccator before use.

[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.

[3] "Trade Practices for Proper Packer Cattlehide Delivery," issued by Leather Industries of America and U.S. Hide, Skin & Leather Association (printed May 1985).

[4] JALCA, 1963, p. 143.

6.2 *Electric Muffle Furnace,* with controller or rheostat and pyrometer, capable of maintaining a temperature of 650 ± 25°C.

6.3 *Desiccator,* of appropriate size and charged with fresh desiccant.

6.4 *Analytical Balance,* capable of weighing to 0.001 g.

6.5 *Oven,* forced air, capable of maintaining 100 – 105°C.

7. Reagents and Materials

7.1 Distilled or de-ionized water.

8. Hazards

8.1 The crucibles will be extremely hot when they are removed from the muffle furnace. Therefore, it is suggested that the analyst handle the hot crucibles using furnace tongs or equivalent.

9. Sampling

9.1 Sampling shall be per Practice D6715.

9.2 Time is crucial in the preparation of salt-preserved hides for chemical testing. Samples should not be prepared if analysis can not be done immediately. The following procedures should be executed with a minimum of hide exposure to either air or moisture or salt-absorbent materials (including human skin). Ensuring this will prevent or minimize any significant or salt loss from the samples.

9.3 During all stages of preparation, samples shall always be cut on a non-porous, non-absorbing hard surface using a clean sharp cutting tool, preferably a new razor or scalpel blade. Avoid excess pressure on the sample that could force liquid (moisture) from the hide sample.

9.4 *De-hairing, Cleaning, and Dicing:*

9.4.1 Without damaging the hide surfaces, carefully remove all the hair and manure from each hide sample using appropriate equipment (clippers, scissors, razor, knife, an so forth),

9.4.2 Remove any loose surface salt or residual undesired material (manure, dirt, and so forth) from each hide sample and flesh each sample down to firm corium.

9.4.3 Using appropriate equipment (scalpel, single-edged razor blade, cutting die, and so forth), cut a 1 in. (25.4 mm) diameter circular plug (disc) from each original hide sample.

9.4.4 Uniformly dice each plug into ¼ in. (6 mm) cubes.

9.4.5 Place all the diced-up samples into a suitably sized airtight glass or plastic container that has minimal airspace once all diced hide samples have been added.

9.4.6 Repeat the dicing procedure for each of the individual hide sample plugs, until the composite sample is complete.

9.4.7 Thoroughly mix the composite sample.

10. Procedure

10.1 Weigh duplicate samples accurately into tared crucibles 2 to 5 g (±0.001 g) of cured (salt-preserved) hide or skin prepared as described in 9.1 to 9.4, and record the weights. Perform this step with minimum delay and avoid prolonged exposure of the sample to air so that it does not gain mass (moisture) or lose mass (moisture) at a significant rate.

10.2 Pre-dry the crucible and sample at 60 – 65°C (140 – 149°F) for 3 h ± 15 min to prevent gelation.

10.3 Place the pre-dried crucible and sample in the oven and maintain at 100 – 105°C for 16 ± ½ h.

10.4 Remove crucible from the oven, cool in desiccator, weigh and record to the nearest ±0.001 g.

10.5 Place the crucible and dried sample in a cool muffle furnace (≤ 100°C) and raise the temperature gradually to 600 ± 25°C. Leave the crucible and sample in the hot furnace for a minimum of 3 h up to 16 h at 600 ± 25°C.

10.6 To prevent any loss of ash, very carefully remove the crucible from the furnace. **Warning—**The crucible is extremely hot!

10.7 Check the ash condition:

Ash condition	Action
White/Gray-white (complete ashing)	Proceed to 10.9
Dark gray or Black (incomplete ashing)	Proceed to 10.8

10.8 Cool the crucible to room temperature. Moisten the ash with 10–15 mL of DI water, then place the crucible in the oven until visibly dry. Transfer the crucible to a cool muffle furnace (≤ 100°C) and repeat steps 10.5 to 10.7.

10.9 Cool the crucible to room temperature in a desiccator.

10.10 Weigh the crucible immediately and record the weight.

11. Calculation

11.1 Calculate the percentage of moisture (volatile matter by oven drying) as follows:

$$D = \text{moisture, } \% = [(A - B)/(A - C)] \times 100$$

where:

A = original weight of sample and crucible,
B = weight of dried sample and crucible, and
C = weight of crucible.

11.2 Calculate the percentage of ash as follows:

$$F = \text{Ash, } \% = [(E - C)/(A - C)] \times 100$$

where:

E = weight of ash and crucible, and A and C have the same meaning as in 11.1.

11.3 Calculate the ash to moisture ratio in percent as follows:

$$G = \text{ash to moisture ratio} = (F/D) \times 100$$

11.4 Calculate the brine saturation value in percent as follows:

$$\text{Brine Saturation Value, } \% = (G/35.9) \times 100$$

12. Report

12.1 Report the Brine Saturation Value to the nearest whole number %.

12.2 Report the percentage of ash to the nearest 0.1 %. The ash value reported shall be the average of duplicate analysis.