



# Standard Test Method for Brine Saturation Value of Cured (Salt-Preserved) Hides and Skins<sup>1</sup>

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 ( $\epsilon$ ) 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 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*:<sup>2,3</sup>

**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

<sup>1</sup> 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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<sup>2</sup> 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.

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

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.<sup>4</sup> 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.

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.

<sup>4</sup> JALCA, 1963, p. 143.

## 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 ( $\pm 0.001$  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  $\pm$  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  $\pm$  ½ h.

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

10.5 Place the crucible and dried sample in a cool muffle furnace ( $\leq 100^\circ\text{C}$ ) and raise the temperature gradually to 600  $\pm$  25°C. Leave the crucible and sample in the hot furnace for a minimum of 3 h up to 16 h at 600  $\pm$  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 <b>10.9</b>
Dark gray or Black (incomplete ashing)	Proceed to <b>10.8</b>

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 ( $\leq 100^\circ\text{C}$ ) 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.

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

12.4 Report the ash to moisture ratio to the nearest 0.1 %.

## 13. Precision and Bias

13.1 *Precision*—The precision of this test method is based on a repeatability study of WK16710, New Test Method for Brine Saturation Value of Cured (Salt Preserved) Hides and Skins, conducted in 1985. One laboratory compared specimens from five different lots of hides for percent moisture, percent ash, and brine saturation. Every “test result” represents the

average of two individual determinations (except for brine saturation). The laboratory was asked to submit two replicate test results for each analysis. Except for the use of only one laboratory, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D31-1010.<sup>5</sup>

13.1.1 *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.

13.1.1.1 Repeatability limits are listed in Table 1, Table 2, and Table 3.

13.1.2 *Reproducibility 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.

13.1.2.1 Reproducibility limits were not addressed in this study.

13.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

13.1.4 Any judgment in accordance with statements 13.1.1 would normally have an approximate 95% probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are

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**TABLE 1 Moisture (%)**

Material	Average $\bar{x}$	Repeatability Standard Deviation $s_r$	Repeatability Limit $r$
1	40.54	1.35	3.78
2	41.55	0.69	1.92
3	45.56	1.64	4.59
4	43.92	1.46	4.10
5	46.55	0.48	1.35

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**TABLE 2 Ash (%)**

Material	Average $\bar{x}$	Repeatability Standard Deviation $s_r$	Repeatability Limit $r$
1	14.47	0.28	0.77
2	15.00	0.79	2.22
3	12.14	0.01	0.02
4	13.55	0.06	0.16
5	11.10	0.05	0.14

**TABLE 3 Brine Saturation (%)**

Material	Average $\bar{x}$	Repeatability Standard Deviation $s_r$	Repeatability Limit $r$
1	89.5	4.9	13.9
2	98.0	2.8	7.9
3	74.0	2.8	7.9
4	86.0	2.8	7.9
5	67.0	1.4	4.0

applicable to all circumstances and uses. The limited number of laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95% probability limit would imply. The repeatability limit should be considered as a general guide, and the associated probability of 95% as only a rough indicator of what can be expected.

13.2 *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.

13.3 The precision statement was determined through statistical examination of the analytical results from one laboratory, on samples from five different material lots.

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

## 14. Keywords

14.1 ash; brine saturation; curing; hide; moistureskin