



Standard Test Method for Analysis of Tannery Liquors¹

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1. Scope

1.1 This test method covers the analysis of tannery liquors made up from vegetable tanning materials.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D4903 Test Method for Total Solids and Water in Vegetable Tanning Material Extracts

D4904 Practice for Cooling of Analytical Solutions

D6401 Test Method for Determining Non-Tannins and Tannin in Extracts of Vegetable Tanning Materials

D6402 Test Method for Determining Soluble Solids and Insolubles in Extracts of Vegetable Tanning Materials

D6404 Practice for Sampling Vegetable Materials Containing Tannin

D6410 Test Method for Determining Acidity of Vegetable Tanning Liquors

2.2 *ALCA Methods:*

A25 Analysis of Tannery Liquors³

3. Terminology

3.1 *Definitions:*

¹ This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.01. This test method has been adapted from and is a replacement for Method A25 of the Official Methods of the American Leather Chemists Association.

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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 Official Methods of the American Leather Chemists Association. Available from the American Leather Chemists Association, University of Cincinnati, P.O. Box 210014, Cincinnati, OH 45221-0014.

3.1.1 *tannery liquor*—water solutions containing vegetable tannin that are made up and used in a vegetable tannery.

3.1.2 *tannin*—an astringent substance found in the various parts of plants such as bark, wood, leaves, nuts, fruits, roots, etc. Also, quantitatively, tannins are operationally defined as the non-volatile materials present in tannin extracts and raw or spent materials that are dissolved or suspended in water, are part of the soluble solids determined by Test Method D6402, and do react with or bind to hide powder when mixed as in this test method.

3.1.3 *vegetable tannins*—mixtures of substances (natural products) obtained from plant tissues by water extraction which have the chemical and physical properties necessary to convert animal hides and skins into leather.

4. Summary of Test Method

4.1 An analytical solution is prepared from the sample of tannery liquor (Practice D6404). Specimen aliquots from this analytical solution are then analyzed for total solids (Test Method D4903), soluble solids and insolubles (Test Method D6402), non-tannins and tannin (Test Method D6401), and total acidity (Test Method D6410).

5. Significance and Use

5.1 This test method is used to determine the chemical properties of tannery liquors which are relevant for the vegetable tanning process and influence the astringency of vegetable tanning liquors. The astringency of liquors is dependent upon the solids and tannin content and the acidity. This method provides a standard procedure for determining these properties for any sample of vegetable tanning liquor.

5.2 The specimens are aliquots from the analytical solution prepared from the sample of tannery liquor collected for this purpose.

5.3 The total solids, soluble solids, and non-tannins content are determined and then the tannin content of the liquor sample is calculated. Because the amount of tannin per liter of analytical solution is less than that required for Test Method D6401, a table specifying the quantity of prepared hide powder to be used for solutions with lower tannin concentrations is included in this test method.

5.4 The total acidity of the liquor sample is determined by one of two titrametric procedures described in Test Method D6410.

5.5 The results of this test method are dependent on a great many variables but particularly upon: the temperature conditions under which the solutions were prepared and stored and the temperature at which the current analysis is performed; the uniformity and consistency of the Kaolin paste layer deposited onto the filter paper; the rate of solution run-out from the pipette; conditions related to the properties of the hide powder used to react with the tannin content of the solution; etc. It is, therefore, essential that the method be followed exactly in order to obtain reproducible results both among specimens within a laboratory and for analyses between laboratories.

6. Apparatus and Reagents

6.1 Analytical Solution:

6.1.1 *Flask*, 1 L volumetric. Class A flasks with a bulb in the neck (M.C.A. type) are especially suitable for this work.

6.1.2 *Hydrometer*, preferably with a Barkometer scale (i.e. a scale calibrated in °Bk). The three digits to the right of the decimal point on a standard specific gravity reading are equal to the Barkometer scale reading. That is, a sp.gr. of 1.200 equals 200°Bk and a sp.gr. of 1.020 equals 20°Bk.

6.2 Acidity Determination—Method 1:

6.2.1 *Graduated Cylinder*, glass-stoppered, graduated to contain 250 mL.

6.2.2 *Gelatin Solution*, 1 %, neutral to bromocresol purple. The addition of 25 mL of 95 % ethyl alcohol per litre is recommended to prevent frothing. The solution shall be adjusted to neutrality to bromocresol purple with 0.1 N acetic acid or 0.1 N sodium hydroxide.

6.2.3 *Kaolin*⁴, acid-washed kaolin clay which conforms to the following specifications:

6.2.3.1 Suspend 1.0 g kaolin in 100 mL distilled water. The pH value should be between 4.5 and 6.0 after 5 min.

6.2.3.2 A mixture of 2.0 g kaolin and 200 mL distilled water are shaken for 10 min and the mixture filtered through the standard filter paper (see 6.5). A 100 mL aliquot of the clear filtrate should have less than 0.001 g of residue after evaporation and oven-drying in a platinum dish.

6.2.4 *Alkaline Titrant*, 0.1 N sodium hydroxide solution.

6.3 Acidity Determination—Method 2:

6.3.1 *Pipet*, transfer pipette with 6 mL capacity.

6.3.2 *pH Meter*, with glass/calomel electrodes.

6.3.3 *Alkaline Titrant*, 0.1 N sodium hydroxide solution.

7. Test Specimen

7.1 The specimens for the tannin determination shall consist of 100 mL aliquots of the analytical solution prepared from the tannery liquor sample or the filtrates collected for particular methods.

⁴ The sole source of supply of Kaolin known to the committee at this time is L. H. Lincoln & Son, Inc., 203 Cherry Street, Coudersport, PA 16915. 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.

7.2 The specimen for determination of acidity shall be either a 25 mL aliquot (for titration - Method 1) or a 6.0 mL aliquot (for titration - Method 2) from the analytical solution prepared from the tannery liquor sample.

8. Procedure

8.1 Preparation of the Analytical Solution:

8.1.1 Collect the tannery liquor sample as described in Practice D6404 (11.10).

8.1.2 Prepare the analytical solution for this analysis by diluting a specimen aliquot from the tannery liquor sample to the mark in a 1 L volumetric flask with distilled water. The aliquot specimen shall be of such size that, after dilution the analytical solution shall contain as nearly as possible 7.0 g total solids per litre. Follow the above procedure except where such dilution would give more than 3.5 g tannin per litre. In this case select the aliquot specimen size such that after dilution the analytical solution shall contain as nearly as possible, but not exceed, 3.5 g tannin per litre, irrespective of the solids content.

8.1.3 Dilution Procedure:

8.1.3.1 When the liquor sample has a specific gravity of 65°Bk (that is, sp.gr. ≥ 1.065) or more, dilute the aliquot specimen with water at 40°C, and cool as in Practice D4904.

8.1.3.2 When the liquor sample has a specific gravity of less than 65°Bk (that is, sp.gr. < 1.065), dilute the aliquot specimen with water at 23°C.

8.2 Analytical Procedures:

8.2.1 Determine total solids as in Test Method D4903.

8.2.2 Determine soluble solids and insolubles as in Test Method D6402.

8.2.3 Determine non-tannins and tannin as in Test Method D6401, except adjust the quantity of prepared hide powder used (per Test Method D6401) according to Table 1.

8.2.4 In the calculation for the (%) non-tannins (Test Method D6401), calculate the factor F in the formula according to the weight of water contained in the actual amount of hide powder used for each specimen.

8.2.5 Determine the total acidity as in Test Method D6410.

9. Calculation

9.1 Calculate the results of the analyses as directed in each of the referenced test methods.

10. Report

10.1 Record the following information:

10.1.1 Tannin analysis results to the nearest 0.01 %, and

10.1.2 Total acidity to the nearest 0.1 % acetic acid.

TABLE 1 Quantities of Hide Powder

Tannin (grams per liter)	Dry Hide Powder (grams per 200 mL)
3.50	9.1
3.00	7.8
2.50	6.5
2.00	5.2
1.50	3.9
1.00	2.6

11. Precision and Bias

11.1 This test method is adopted from Method A25 of The Official Methods of the ALCA. This test method has long been in use and was approved for publication before the inclusion of precision and bias statements were mandated. The original inter-laboratory test data is no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this standard practice is adequate for the contemplated use.

11.2 The analytical results obtained by this method are operationally defined by each of the analytical procedures employed. There is no independent measure of the true analytical components of a sample. Therefore the bias cannot be related to the true component content of the sample.

12. Keywords

12.1 acidity; tannery liquor; tanning liquor; tannins; tannin analysis; vegetable tannin analysis

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