Designation: D6410 – 99 (Reapproved 2014)

Standard Test Method for Determining Acidity of Vegetable Tanning Liquors[1]

This standard is issued under the fixed designation D6410; 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 determining the acidity 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]

D4904 Practice for Cooling of Analytical Solutions

D6404 Practice for Sampling Vegetable Materials Containing Tannin

2.2 *ALCA Methods:*

A25 Analysis of Tannery Liquors[3]

3. Terminology

3.1 *Definitions:*

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

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 acidity by one of two titrametric methods.

5. Significance and Use

5.1 This test method is used to determine one of 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 test method provides a standard procedure for determining the total acidity 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 acidity of the liquor sample is determined by one of two titrametric procedures described in this test method.

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 (that is, 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 I:*

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:

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

Current edition approved Nov. 1, 2014. Published December 2014. Originally approved in 1999. Last previous edition approved in 2009 as D6410 – 99 (2009). DOI: 10.1520/D6410-99R14.

[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.

[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.

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 II:*

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 specimen for determination of acidity shall be either a 25 mL aliquot (for titration - Method I) or a 6.0 mL aliquot (for titration - Method II) 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.

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 Test Method 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 *Determination of Total Acidity of the Tannery Liquors:*

8.2.1 *Method I:*

8.2.1.1 Transfer a 25 mL aliquot of the analytical solution to a 250 mL graduated and stoppered cylinder. Then add 50 mL of the gelatin solution to the cylinder. Dilute this mixture to 250 mL with distilled water. Then add 15 g of kaolin, the stopper cylinder, and shake vigorously. Then allow the mixture to settle for at least 15 min.

8.2.1.2 Then withdraw a 30 mL aliquot of the supernatant solution from the cylinder, dilute with 50 mL of distilled water, and titrate with 0.1 *N* sodium hydroxide using bromocresol purple as the indicator.

8.2.2 *Method II:*

8.2.2.1 A 6 mL aliquot of the analytical solution is transferred to a suitable vessel for titration using a pH meter and pH electrodes. The specimen is diluted with 75 mL of distilled water and titrated with 0.1 *N* sodium hydroxide to pH = 6.0.

9. Calculation

9.1 Report the results as percent acetic acid and are calculated as follows:

9.1.1 For Method 1:

$$\text{total acidity}\,(\%-\text{acetic acid}) = V_B \times (0.2) \quad (1)$$

where:

V_B = the mL of 0.1 *N* sodium hydroxide used in the titration.

9.1.2 For Method 2:

$$\text{total acidity}\,(\%-\text{acetic acid}) = V_B \times (0.1) \quad (2)$$

where:

V_B = the mL of 0.1 *N* sodium hydroxide used in the titration.

10. Report

10.1 Report the total acidity to the nearest 0.1 % acetic acid.

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 test method are operationally defined by the analytical procedures employed. There is no independent measure of the true acidity component of a sample. Therefore the bias cannot be related to the true acidity content of the sample.

12. Keywords

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