Designation: D6401 – 99 (Reapproved2009)

Standard Test Method for Determining Non-Tannins and Tannin in Extracts of Vegetable Tanning Materials[1]

This standard is issued under the fixed designation D6401; 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 is intended for use in determining the quantity of soluble non-tannins and tannin in solutions of tannin extracts, water extracts of vegetable tanning materials, or tanning liquors. The method is applicable to the analysis of liquid, solid, pasty, and powdered extracts and to extracts of raw or spent materials.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units 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]

D2617 Test Method for Total Ash in Leather

D4901 Practice for Preparation of Solution of Liquid Vegetable Tannin Extracts

D4902 Test Method for Evaporation and Drying of Analytical Solutions

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

D4905 Practice for Preparation of Solution of Solid, Pasty and Powdered Vegetable Tannin Extracts

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

D6405 Practice for Extraction of Tannins from Raw and Spent Materials

2.2 *ALCA Method:*

A22 Non-Tannins and Tannin[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *soluble non-tannins*—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 not react with or bind to hide powder when mixed as in this test method.

3.1.2 *tannins*—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.

4. Summary of Test Method

4.1 An aliquot of the analytical solution prepared from tannin extracts (Practices D4901 or D4905) or of the water extract from raw or spent materials (Practice D6405) is dried overnight in a forced-air oven (Test Method D4902) and the solid residue remaining is determined and defined as the total solids for that sample (Test Method D4903). Another aliquot of the same solution is passed through a specified filtering procedure and the quantity of solid residue remaining in the filtrate is determined and defined as the soluble solids for that sample; the calculated difference between the total solids and the soluble solids is defined as the insolubles for that sample (Test Method D6402). Another aliquot of the original analytical solution or water extract is mixed with a prepared hide powder material with the resulting solution being passed through the same filtering procedure as for the soluble solids analysis. The quantity of solid residue remaining in the filtrate is determined and defined as the soluble non-tannins for that sample; the calculated difference between the total soluble solids and the soluble non-tannins is defined as the tannin content for that sample.

[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 method has been adapted from and is a replacement for Method A22 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.

5. Significance and Use

5.1 This test method is used to determine the proportion of the total soluble solids which are soluble non-tannins and the proportion which are tannins in a solution of tannin extract or in the water extract from raw or spent materials prepared for tannin analysis.

5.2 The specimens are aliquots from the analytical solutions prepared from tannin extracts or the water extract solutions prepared from raw or spent materials.

5.3 The soluble non-tannins are defined as the portion of the soluble solids which are not absorbed or bound by a prepared hide powder material.

5.4 The tannins are defined as the portion of the soluble solids which are absorbed or bound by a prepared hide powder material.

5.5 The results of this test method are dependent on a great many variables but particularly upon:

5.5.1 The temperature conditions under which the solutions were prepared and stored and the temperature at which the current analysis is performed;

5.5.2 The uniformity and consistency of the Kaolin paste layer deposited onto the filter paper;

5.5.3 The rate of solution run-out from the pipette;

5.5.4 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 *Tannin Dish,* crystallizing dish, borosilicate glass, 50 mm tall, 70 mm outside diameter. The bottom corner shall be rounded to a radius of 6 mm, the bottom shall be flat and not cupped in the center, and the top edge shall be rounded and polished.

6.2 *Watch Glass,* a suitable size (approximately 150 mm diameter) to be used as a cover for the funnel and filter paper and a suitable size (75 mm) to be used as a cover for the tannin dishes.

6.3 *Pipet,* 100 mL capacity, preferably with a wide orifice approximately 2.4 mm (3⁄32 in.) diameter and 15-25 s delivery time.

6.4 *Volumetric Flasks,* graduated to deliver 200 mL borosilicate flask (Pyrex No. 5840 works well).

6.5 *Filter Paper*[4], 21.5 cm diameter, pleated to contain 32 evenly divided creases.

6.6 *Funnel,* 100 to 125 mm top diameter, 60° angle bowl, and 150 mm stem length.

6.7 *Shake Bottles,* 0.95 L (32 oz), with rubber stoppers. The bottles shall be approximately 21.6 cm (8.5 in.) overall height and 8.9 cm (3.5 in.) diameter. One quart canning jars (Mason-type) with plastic screw-on lids work well.

6.8 *Shaking Machine,* rotating type, equipped to hold 0.95 L (32 oz) bottles for end-over-end agitation of hide powder and analytical solution. The speed of rotation shall be 60 ± 2 rpm, and the machine shall be so constructed that the side of the shake bottle adjacent to the rotating shaft shall be not less than 5.1 cm (2 in.) nor more than 7.6 cm (3 in.) from the center of the shaft.

6.9 *Cloth,* The cloth shall be of cotton, with a nominal (unbleached) thread count of 48 by 48 and a weight of 1.65 yards per 454 g (1 lb). When bleached, the thread count will be approximately 52 to 53 by 43 to 48.

6.10 *Kaolin*[5], acid-washed kaolin clay which conforms to the following specifications:

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

6.10.2 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). A100 mL aliquot of the clear filtrate should have less than 0.001 g of residue after evaporation and oven-drying in a platinum dish.

6.11 *Chrome Alum Solution,* a 3 % solution, prepared by dissolving $CrK(SO_4)_2$ 12 H_2O in distilled water at room temperature (3 g per 100 mL of solution). The solution shall not be more than 30 days old when used.

6.12 *Hide Powder*[5]—The hide powder shall meet the following specifications:

6.12.1 The hide powder shall be finely ground and uniform in texture with no lumps present.

6.12.2 The moisture content should be 12 to 15 % as determined by Test Method D4902.

6.12.3 The ash content shall be less than 0.3 % as determined by Test Method D2617.

6.12.4 A mixture prepared from 7.0 g of air-dry hide powder suspended in 100 mL of 0.1 *N* KCl and set aside for 24 h with occasional shaking shall have a pH value not less than 5.0 nor greater than 5.4.

6.12.5 A sample of the hide powder shall be carried through the analytical procedure for the determination of non-tannins described below using distilled water in place of the analytical solution. The water-soluble content of the resulting filtrate shall not exceed 0.004 g.

6.12.6 Each new shipment of hide powder should be tested against the hide powder of the previous shipment using certain standard extracts. The new hide powder shall give an average non-tannin percentage which differs by less than ± 0.2 from the comparable non-tannin percentage obtained with the hide powder from the previous shipment.

[4] The sole source of supply of S&S No. 610 filter paper known to the committee at this time is Schleicher & Schuell, 10 Optical Avenue, P.O. Box 2012, Keene, NH 03431. 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.

[5] The sole source of supply of Kaolin and hide powder 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.