Designation: D4903 – 99 (Reapproved 2016)

# Standard Test Method for Total Solids and Water in Vegetable Tanning Material Extracts[1]

This standard is issued under the fixed designation D4903; 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 total solids and water in extracts of vegetable tanning materials. The test method is applicable to solutions of liquid, solid, pasty, and powdered extracts, and to extracts of raw or spent materials.

1.2 *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]

D4901 Practice for Preparation of Solution of Liquid Vegetable Tannin Extracts

D4902 Test Method for Evaporation and Drying of Analytical Solutions

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

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

D6405 Practice for Extraction of Tannins from Raw and Spent Materials

2.2 *ALCA Methods:*

A20 Total Solids and Water[3]

## 3. Summary of Test Method

3.1 An aliquot of the prepared analytical solution is pipetted into an evaporating dish and evaporated to dryness in a forced air oven.

## 4. Significance and Use

4.1 This test method is useful in determining the total solids and water in analytical solutions.

## 5. Apparatus

5.1 *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.

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

5.3 *Drying Oven*—A forced-air convection oven (or mechanical-convection draft oven) capable of maintaining a temperature of 100 ± 2.0° C.

5.4 *Thermometer*—Accurate to ±2.0° C, used to check and monitor the oven set point.

5.5 *Dessicator*—Any convenient form or size.

## 6. Test Specimen

6.1 The specimen shall consist of 100 mL of a solution prepared as described in Practices D4901, D4905, and D6405.

## 7. Procedure

7.1 Thoroughly mix the solution, prepared as described in Practices D4901, D4905, and D6405, by inverting and shaking

[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, Method A20 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.

8 to 10 times, taking care to ensure that all insoluble matter is uniformly dispersed and none is left adhering to the walls of the flask. Immediately pour a 150-mL to 250-mL portion (approximately) into a bottle or other suitable container, stoppered or covered, and retain until the soluble solids and non-tannins are ready to be pipetted (see Practice D6404). Where the analysis is being performed in a constant temperature room, however, the specimen may be pipetted from this portion at any convenient time. At the proper time, thoroughly mix this secondary portion by shaking, or stirring, so that the insoluble matter is uniformly distributed throughout the liquor and none remains adhering to the walls of the container. Immediately rinse the 100-mL pipet with the liquor rejecting the rinsings. Then stir the liquor well, avoiding production of air bubbles and foam, and while stirring immerse the tip of the rinsed pipet and fill pipet quickly by suction. Bring the level to the mark as quickly as possible, and transfer the 100-mL specimen into a tared evaporating dish. (Since tan liquors are very liable to foam and since they may contain large amounts of rapid-settling insoluble matter, conduct the final mixing so as to avoid foam formation. Fill and bring the pipet to the mark as quickly as possible.) Place the dish containing the specimen, together with the other dishes containing the specimens for soluble solids and non-tannins (Test Methods D6401 and D6402), in the drying oven and evaporate and dry as specified in Practice D4902.

## 8. Results

8.1 The amount of total solids in the sample shall be calculated as follows:

$$\text{total solids, \%} \frac{(W_2 - W_1) \times 10}{W_3} \times 100 \quad (1)$$

where:

$W_1$ = is the tare of the evaporating dish,
$W_2$ = is the weight of the dish plus dry residue, and
$W_3$ = is the weight of the specimen used in 1000 mL of the solution (Practices D4901, D4905, and D6405).

8.2 Since two specimens of each sample were taken in preparing the solutions (Practices D4901, D4905, and D6405), two values for total solids will be obtained for each extract or tanning material. The average of these shall be taken as the percentage of total solids in the sample under test. (Duplicates are considered to be in good agreement when the percent total solids differ by no more than 0.2.)

8.3 Record the results to the nearest 0.01 %.

## 9. Water

9.1 The amount of water in the sample shall be calculated as follows:

$$\% \text{ water} = 100 - \% \text{ total solids} \quad (2)$$

where:
percent total solids is determined as in 8.1.

## 10. Precision and Bias

10.1 This test method is adopted from the procedures of the American Leather Chemists Association,[3] where it has long been in use and where it was approved for publication before the inclusion of precision and bias statements was mandated. The original interlaboratory test data are no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias of this test method is adequate for the contemplated use.

10.2 The total solids content obtained by this test method is operationally defined as the dried solids weight of the specimen collected after drying the specimen. The water content is defined as the difference between 100% and the total solids of the sample. There is no independent measure of the true total solids and water content of a sample. Therefore the bias cannot be related to the true total solids or water content of the sample.

## 11. Keywords

11.1 solids; tannin analysis; total solids; vegetable tannin analysis; water