Designation: D6402 – 99 (Reapproved 2014)

Standard Test Method for Determining Soluble Solids and Insolubles in Extracts of Vegetable Tanning Materials[1]

This standard is issued under the fixed designation D6402; 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 solids and insolubles in solutions of tannin extracts, water extracts of vegetable tanning materials, or tanning liquors. This test method is applicable to the analysis of liquid, solid, pasty, and powdered tannin extracts and to the water extracts of raw or spent 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]

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

D6403 Test Method for Determining Moisture in Raw and Spent Materials

D6405 Practice for Extraction of Tannins from Raw and Spent Materials

2.2 *ALCA Methods:*

A21 Soluble Solids and Insolubles[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *insolubles*—non-volatile materials present in tannin extracts and raw or spent materials that are dissolved or suspended in water and do not pass through a filtering process described in this method.

3.1.2 *soluble solids*—non-volatile materials present in tannin extracts and raw or spent materials that are dissolved or suspended in water and pass through a filtering process described in this method.

4. Summary of Test Method

4.1 An aliquot of the analytical solution prepared from tannin extracts (Practices D4901 or D4905) or the water extract from raw or spent materials (Practice D6405) is dried overnight in a forced-air oven (Test Method D4902) and the quantity of solid residue remaining is determined and used to calculate 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 used to calculate the soluble solids for that sample. The difference between the total solids and the soluble solids is defined as the insolubles for that sample.

5. Significance and Use

5.1 This test method is used to determine the proportion of the total solids which are soluble solids and that proportion which are insoluble solids 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.

[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 A21 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.

5.3 The soluble solids are defined as the portion of the total solids which are dissolved in the water and pass through a filter prepared by depositing a layer of Kaolin paste onto a standard filter paper.

5.4 The insolubles are defined as the portion of the total solids which do not pass through the standard filter paper prepared with the layer of Kaolin paste.

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; 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 second delivery time.

6.4 *Graduated Cylinder,* standard laboratory grade with at least 225 mL capacity.

6.5 *Glass Rods,* soft glass stirring rods with rounded, fire-polished ends.

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

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

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

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

6.8.2 A mixture of 2.0 g kaolin and 200 mL distilled water is shaken for 10 min and the mixture filtered through the standard filter paper. 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.

6.9 *Balance,* analytical balance which will weigh up to 100 g with an accuracy of ± 0.1 mg (± 0.0001 g).

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

6.11 *Thermometer,* accurate to ± 0.2°C used to check and monitor the oven set point.

6.12 *Dessicator,* any convenient form or size, using any normal dessicant.

7. Test Specimen

7.1 The specimen shall consist of 100 mL of the solution prepared as described in Practices D4905, D4901, or D6405 and after passing through the filtering process described in this test method.

8. Procedure

8.1 Place a pleated filter paper in a 100 to 125 mm funnel.

8.1.1 Fold the filter paper to contain 32 evenly divided pleats.

8.2 Add to 2.0 g of kaolin in a clean glass container (a 250 mL beaker works well) 25 mL of the well-mixed analytical solution (which has been prepared as described in Practices D4901, D4905, or D6405) and stir the mixture with a glass stirring rod to form a smooth paste. Then add an additional 200 mL of the well-mixed analytical solution (making 225 mL total) and again stir the mixture to a uniform suspension. Immediately pour the suspension onto the pleated filter paper in the funnel and collect the filtrate in the container in which the solution and kaolin were mixed. After approximately 40 mL of the filtrate has been collected, swirl it, to pick up kaolin remaining on the sides and bottom of the container, and return to the funnel. During this and subsequent operations, keep funnels and containers covered with watch glasses to avoid changes due to evaporation, and maintain the temperature of the solution and of the filtrates between 23 and 25°C. Repeat the operation of collecting and repouring 40 mL of filtrate as many times as is necessary until the solution has been in contact with the filter paper for exactly 1 h. At the end of the hour, siphon the solution out of the filter as completely as possible, taking care not to disturb the kaolin film on the filter paper. Discard this liquid.

8.3 Immediately, refill the kaolin-lined paper in the funnel with 225 mL of the well-mixed analytical solution which has been kept at a temperature of 23 to 25°C. Disturb the kaolin film as little as possible during this operation and best results are obtained by carefully pouring the solution into the center of the cone along a glass stirring rod contacting the edge of the beaker spout as the liquid is carefully transferred. Pour no additional solution to this 225 mL onto the filter paper. When 40 mL of the filtrate have passed through, collect the next portion in a clean, dry, glass container and, when about 125 mL have been collected, remove the container from beneath the funnel.

[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 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.

8.4 Check this 125 mL for clarity by swirling and viewing against the light.

8.4.1 If not clear it shall be rejected, and the determination re-run using a new filter.

8.4.2 If the filtrate is clear, mix it with a clean glass rod and withdraw a 100 mL specimen with a pipet and transfer into a tared tannin dish prepared as in Test Method D6403. The pipet should be the one used for transferring the specimen used for determining total solids (Test Method D4903) and rinse with a portion of the filtrate before withdrawing and measuring the specimen.

8.5 Place the dish containing the specimen, together with the other tannin dishes containing the specimens for total solids and non-tannins, in the drying oven and evaporate and dry as specified in Test Method D4902.

9. Calculation

9.1 Calculate the amount of soluble solids in the specimen as follows:

$$\text{soluble solids, } \% = \{[(W_2 - W_1) \times 10]/(W_3)\} \times 100 \qquad (1)$$

where:

W_1 = weight (grams), tare weight of tannin dish,
W_2 = weight (grams), tannin dish plus oven-dried specimen, and
W_3 = weight (grams), specimen used to prepare 1 L of the analytical solution in Practices D4901, D4905, or D6405.

9.2 Two specimens of each sample material were taken in preparing the solutions (Practices D4901, D4905, or D6405); therefore two values for soluble solids will be obtained for each extract or tanning material. The average (mean) of these values shall be taken as the percentage of soluble solids in the sample under test.

9.2.1 Duplicates are considered to be in good agreement when the percent soluble solids differ by no more than 0.2.

9.3 The amount of insolubles in the sample shall be calculated as follows:

$$\text{insolubles, } \% = \text{total solids } (\%) - \text{soluble solids } (\%) \qquad (2)$$

where:
total solids (%) is determined as in Test Method D4903, and soluble solids (%) is determined as in 9.1.

10. Report

10.1 Record the soluble solids and insolubles results to the nearest 0.01 %.

11. Precision and Bias

11.1 This test method is adopted from Method A21 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 test method is adequate for the contemplated use.

11.2 The soluble solids content obtained by this test method is operationally defined as the dried solids weight of the specimen collected after filtering through the specially constructed filter system. The insolubles content is defined as the difference between the total solids (Test Method D4903) and the soluble solids of the sample. There is no independent measure of the true soluble solids content or insolubles content of a sample. Therefore the bias cannot be related to the true soluble solids or insolubles content of the sample.

12. Keywords

12.1 insolubles; soluble solids; tannin analysis; vegetable tannin analysis