Designation: D4905 – 99 (Reapproved 2020)

Standard Practice for Preparation of Solution of Solid, Pasty and Powdered Vegetable Tannin Extracts[1]

This standard is issued under the fixed designation D4905; 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 practice covers a standard procedure for use in preparing the analytical solution required for the analysis of solid, pasty, or powdered vegetable tannin extracts.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

D6403 Test Method for Determining Moisture in Raw and Spent Materials

D6404 Practice for Sampling Vegetable Materials Containing Tannin

2.2 *ALCA Method:*

A11 Preparation of Solution of Solid, Pasty, and Powdered Extracts[3]

3. Summary of Practice

3.1 This practice describes a procedure to follow in the preparation of analytical strength solutions from samples of solid, pasty, and powdered vegetable tannin extracts.

4. Significance and Use

4.1 The concentration of tannins in solid, pasty, and powdered extracts needs to be reduced to analytical strength for tannin analyses.

4.2 Vegetable tannin extracts are heterogeneous mixtures of components with varying solubility.

4.3 The solubility of such extracts is influenced by temperature and concentration, which affect the degree of dispersion and size of the component particles.

4.4 Since extracts have greater solubility in hot water than cold, it is desirable to dissolve and disperse an extract in hot water and then let the solution cool slowly to standard room temperature.

4.5 It is often difficult to reduce samples of solid and particularly pasty extracts to specimen size and at the same time ensure representative sampling. Therefore, caution is advised in drawing conclusions on the precision and bias of the results obtained on such extracts; where difficulties in sample preparation are experienced, little confidence can be placed in the results.

5. Specimen

5.1 The specimen shall consist of a portion of the extract sample, prepared as described in Practice D6404, sufficient to

[1] This practice is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.01 on Vegetable Leather. This practice has been adapted from, and is a replacement for, Method A11 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.

give a solution containing as nearly as possible 4 g of tannin per L (not less than 3.75 g, nor more than 4.25 g, per L).

6. Apparatus and Reagents

6.1 *Formaldehyde*—40 % solution.

6.2 *Toluene*—Assay ≥99.5 %.

6.3 *Small Mortar and Pestle.*

6.4 *1-L Volumetric Flask,* M.C.A. type, mixing bulb style.

7. Procedure

7.1 Empty the sample onto a sheet of clean, glazed paper (or suitable clean, smooth foil material), roughly crush any large lumps, and quickly mix and quarter the whole. From each quarter, take a small representative portion, quickly crush, transfer to a small, glass stoppered weighing bottle, and take to the balance. Without delay, transfer the analytical specimen to a tared container of suitable size and weigh to the nearest 0.1 mg. Each sample shall be handled individually, and all operations carried out as quickly as possible up to this point, and all other precautions taken to minimize changes in moisture content.

7.2 *Pasty Extracts*—Where the extract has a moisture content such that it cannot be ground, weigh the sample, broken or cut, into small pieces (W_1) and bring to approximate equilibrium with the laboratory atmosphere by drying for several hours at a temperature not exceeding 60°C. Allow the sample to remain exposed to the air in the laboratory until the approximate equilibrium has been attained. Weigh the partially dried sample again (W_2). Calculate the original moisture content of the extract as in Test Method D6403, paragraph 9.3.3, except that the residual moisture in the air-dried material (B) shall be determined as in the Procedure section of Test Method D4903 instead of as in Test Method D6403, paragraph 9.3.1.

7.2.1 Treat the air-dried sample, prepared as above, as in paragraph 7.1, above. In such cases, calculate the results of the analysis to the basis of the original moisture before drying.

7.3 Pour approximately 200 mL of distilled water, at 95°C, into a 1-L volumetric flask and place the container with the specimen in a funnel resting in the neck of the flask. Add distilled water, at 95°C, to the extract, filling the container. Stir the contents of the container well and allow to stand for a few moments, so that any undissolved particles may settle to the bottom of the container. Decant the supernatant liquid into the flask, add more of the hot, distilled water to the contents of the container, stirred and decanted, and repeat the process until the soluble portion of the specimen has been completely dissolved and the whole of it washed into the flask. Mix the contents of the flask by swirling. Add sufficient distilled water, at 96°C, to bring the volume to approximately 900 mL and again mix the solution by swirling. The full operation of dissolving and decanting, etc., shall be carried out as quickly as possible, and at no time during the operation shall the temperature of the solution fall below 80°C. If necessary, apply heat to keep the solution above 80°C.

7.4 If the solution is likely to ferment (myrabolans or divi-divi), add 1 mL of 40 % formaldehyde and mix the solution again. In any event, the addition of 3 to 4 drops of toluene is recommended to ensure against mold growth during the overnight cooling.

7.5 Prepare duplicate solutions of 1 L each. It is permissible to prepare duplicate 2-L solutions, in which case all appropriate volumes shall be doubled.

7.6 The solution shall be cooled as directed in Practice D4904.

8. Keywords

8.1 analytical solution; tannin analysis; vegetable tannin analysis