Standard Practice for Extraction of Tannins from Raw and Spent Materials[1]

1. Scope

1.1 This practice covers extracting the tannin from raw and spent materials. The water extract obtained by this method is used to determine the tannin content of the original material. Analysis for total solids, soluble solids, and soluble non-tannins of the water extract from a material provides the information necessary to calculate the extractable tannin content of that material. The types of materials typically analyzed by this practice are products of plants such as woods, barks, leaves, nuts, fruits, roots, etc. and any of a wide variety of by-products (spent materials) from industrial processes utilizing plant products.

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]

D1517 Terminology Relating to Leather
D4903 Test Method for Total Solids and Water in Vegetable Tanning Material Extracts
D4904 Practice for Cooling of Analytical Solutions
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
D6403 Test Method for Determining Moisture in Raw and Spent Materials
D6404 Practice for Sampling Vegetable Materials Containing Tannin

2.2 *ALCA Methods:*

A5 Extraction of Raw and Spent Materials[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of general leather and tanning terms used in this practice refer to Terminology D1517.

3.1.2 *raw material*—any of the various parts of plants that are used as a source of vegetable tannins.

3.1.3 *spent material*—plant tissue by-products from industrial processes which may contain significant quantities of vegetable tannins.

3.1.4 *tannin*—an astringent substance found in the various parts of plants such as bark, wood, leaves, nuts, fruits, roots, etc.

3.1.5 *vegetable tannins*—mixtures of substances (natural products) obtained from plant tissues by water extraction which have the chemical and physical properties necessary to convert animal hides and skins into leather.

4. Summary of Practice

4.1 The sample of material to be analyzed for tannin content is first brought to moisture equilibrium with the laboratory atmosphere (that is, wet materials such as spent materials are first dried under ambient laboratory conditions) and then ground sufficiently to pass through a sieve. This ground sample is then extracted with water in a steam-jacketed extractor.

5. Significance and Use

5.1 This practice provides a standard procedure for obtaining the water-soluble materials (including tannins) from any material that can be prepared for and charged to the steam-jacketed extractor. The extraction solutions are then analyzed according to Test Methods D4903, D6401, and D6402.

6. Apparatus and Reagents

6.1 *Sieve,* with circular openings 1.27 cm (0.50 in.) in diameter.

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[1] This practice is under the jurisdiction of ASTM Committee D31 on Leather This test method has been adapted from and is a replacement for Method A5 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.

6.2 *Sieve,* with circular openings of 2 mm diameter (U.S. Std. Sieve Series No. 10).

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

6.4 *Borosilicate Glass Wool,* made of soft, pliable fibers.

6.5 *Steam-Jacketed, Borosilicate Glass Extraction Apparatus,* as described in Fig. 1. This apparatus may need to be special ordered and custom built by a laboratory glass-blowing manufacturer.

6.6 *Tubing,* flexible, chemical and heat resistant tubing of appropriate size and length to fit onto the outlet tubes of the extractor apparatus. The tubing from the extraction chamber outlet must fit into the receiving flask.

6.7 *Clamps,* Hoffman or similar type for use on the tubing extensions from the extractor outlets.

6.8 *Wiley Mill,* or similar grinder.

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

6.10 *Formaldehyde,* 40 % solution.

6.11 *Toluene,* assay ≥ 99.5 %

6.12 *Volumetric Flask,* 1-L capacity for spent materials or 2-L capacity for raw materials. Class A flasks with a bulb in the neck (M.C.A. type) are especially suitable for this work.

7. Preparation of Sample

7.1 All of the sample, drawn as directed in Practice D6404, shall first be ground in a rough grinder, or shredded, to pass a sieve having round openings 1.27 cm (0.50 in.) in diameter. After this grinding, the sample shall be mixed and quartered, as described in the Quartering Samples section of Practice D6404 until about 450 g (1 lb) remains. This material shall then be ground to pass a sieve having circular openings of 2 mm diameter (U.S. Standard Sieve Series No. 10). This grinding shall be conducted in a Wiley mill or similar grinder.

7.2 Where the original sample is too wet to be ground, or where such grinding is likely to cause significant changes in the moisture content, samples of raw material shall be weighed, spread out on (canvas) trays which permit free circulation of air all around the sample, and allowed to attain approximate moisture equilibrium with the laboratory atmosphere; the temperature at no time should exceed 60°C. Spent materials should be similarly treated except that temperatures up to 100°C may be used. The partially dried sample shall then be ground as described in 7.1.

7.2.1 In all cases, the loss of moisture during such drying must be determined and the results of the analysis calculated to the basis of the original moisture before drying. A second moisture shall be run on the finely ground sample. The method of determining these moisture values and calculating the original moisture content is found in Test Method D6403.

8. Test Specimen

8.1 The specimen shall consist of a quantity of the sample, prepared as described in Section 7 above, sufficient to give as closely as possible 4 g tannin per litre of solution (not less than 3.75 nor more than 4.25 g/L). In the case of materials low in tannin, the specimen shall consist of the maximum quantity of the sample which can be extracted in the steam-jacketed extractor (see 6.3 and Fig. 1).

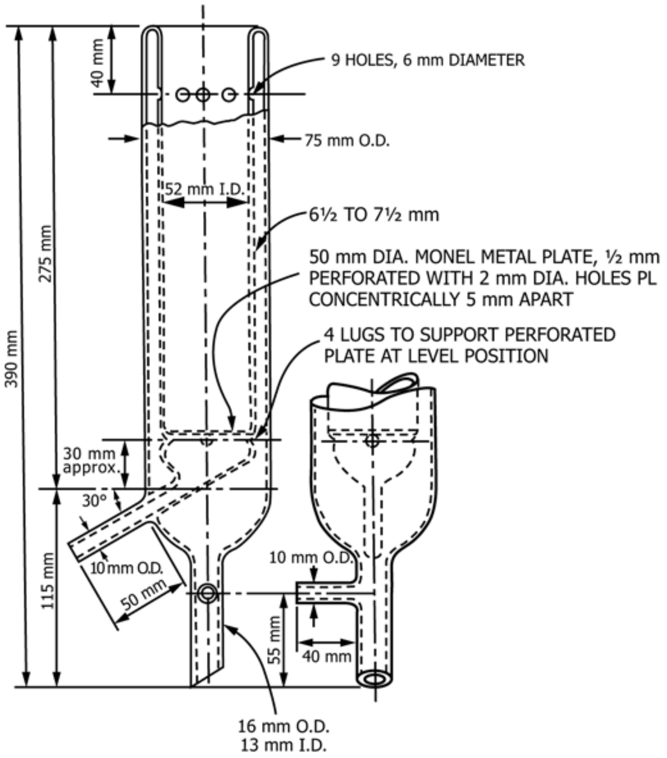

FIG. 1 Extraction Apparatus

9. Procedure

9.1 Prepare duplicate extract samples. Extract raw materials to a volume of 2 L. Extract spent materials to a volume of 1 L.

9.2 Attach the extractor to a boiling-flask, which furnishes steam by the boiling of distilled water. Attach a reflux condenser to the top of the extractor and connect to a source of cold water. Close the outlet tube on the bottom stem of the apparatus just above the steam generating flask by attaching a section of tubing and a clamp which then can be used to help regulate the amount of steam passing into the extractor; this may be particularly helpful at the beginning of an extraction. Close the outlet tube of the extraction chamber by using a section of tubing (with a clamp) leading into the receiving vessel. Prepare a layer of borosilicate glass wool, 0.64 cm (0.25 in.) thick, and place in the bottom of the extraction chamber, over the monel disc. Transfer the specimen to a tared container and weigh to the nearest 1 mg (0.001 g), taking every precaution to avoid changes in moisture content. Then wet the specimen with a minimum of distilled water, at 95°C, and stir the mixture with a glass rod to ensure complete wetting. Then transfer the wetted specimen quantitatively, to the extractor, through a transfer funnel, using a minimum quantity of the hot distilled water. Remove entrapped air in the charge by stirring with a glass rod, taking care not to disturb the layer of glass wool. Open the outlet tube of the extraction chamber slowly and allow the liquid to drain into the appropriate receiving flask. At this stage, the water in the boiling-flask shall be boiling and the steam freely condensing in the jacket. As the charge settles, rinse the stirring rod and the sides of the extractor with the hot, distilled water. Return any solid material which comes through with the first extract, when the outlet tube is opened, to the extraction chamber. When the water has drained from the surface of the charge, cover the specimen with a layer of borosilicate glass wool about 0.64 cm (0.25 in.) thick, and connect the condenser to the extractor. Open the outlet tube wide and continue the extraction for 7 h at a uniform rate. To assist in obtaining uniform extraction, it is recommended that the receiving flask be marked with seven or more equal volume graduations, so that the rate of extraction can be easily gaged, and adjusted by regulating the generation of steam in the boiling-flask. Shake the receiving flask every half hour, to mix the contents, and protect it from the heat of the burner during the extraction. Continue the extraction until the extract volume reaches the final mark on the flask.

9.3 The addition of three to four drops of toluene to the extract solution is recommended to ensure against mold growth during the overnight cooling. If the extracted solution is likely to ferment (for example, myrabolams or divi-divi), add 1 mL of 40 % formaldehyde and the extract solution again mixed.

9.4 Cool the solution as directed in Practice D4904.

9.5 Where, as in 8.1, the tannin content of the material is so low that the maximum quantity of sample which can be extracted if the apparatus fails to give a solution containing 4.00 ± 0.25 g of tannin per litre, reduce the quantity of hide powder to be used in the determination of non-tannins in Test Method D6401 proportionately.

10. Keywords

10.1 extraction; tannin; tannin analysis; vegetable tannin; vegetable tannin analysis