



Standard Test Method for Analysis of Iron and Copper in Vegetable Tanning Materials¹

This standard is issued under the fixed designation D 6407; 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 iron and copper content in vegetable tanning materials. This test method is applicable to liquid, solid, pasty and powdered extracts, to raw and spent materials, and to tannery liquors.

1.2 The values stated in SI units are to be regarded as the 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

- D 4901 Practice for Preparation of Solution of Liquid Vegetable Tannin Extracts
- D 4902 Test Method for Evaporation and Drying of Analytical Solutions
- D 4905 Practice for Preparation of Solution of Solid, Pasty, and Powdered Vegetable Tannin Extracts
- D 6404 Practice for Sampling of Vegetable Materials Containing Tannin
- D 6405 Practice for Extraction of Tannins from Raw and Spent Materials

2.2 ALCA Methods:

- A31 Method for Copper and Iron in Tanning Materials³

3. Summary of Test Method

3.1 A specified quantity of the tanning material is analyzed for iron and copper and content.

4. Significance and Use

4.1 This test method is used to determine the quantity of iron and copper present in vegetable tanning materials or vegetable tannin extracts prepared using Practices D 4901, D 6404, or D 6405.

4.2 Because of the possibility of errors in this test method it is essential that the method be followed exactly in order to obtain reproducible results both among specimens within a laboratory and for analyses between laboratories.

5. Apparatus and Reagents

5.1 *Sulfuric Acid*, concentrated (96 %).

5.2 *Sulfuric Acid Solution*, diluted 1:20 with distilled water.

5.3 *Nitric Acid*, fuming.

5.4 *Hydrochloric Acid*, concentrated (36 %).

5.5 *Hydrochloric Acid Solution*, 0.1 N.

5.6 *Bromine Water*, saturated solution.

5.7 *Ammonium Hydroxide Solution*, concentrated diluted 1:1 with distilled water.

5.8 *Potassium Permanganate Solution*, 0.1 N.

5.9 *Potassium (or Ammonium) Thiocyanate Solution*, 10 g shall be dissolved in distilled water and diluted to 100 mL with distilled water.

5.10 *Stock Iron Solution*, This may be a purchased iron standard solution or may be prepared as follows:

5.10.1 0.70 g of crystallized ferrous ammonium sulfate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] shall be dissolved in 50 mL of distilled water and 20 mL of dilute sulfuric acid (diluted 1:4).

5.10.2 This solution shall be titrated with 0.1 N potassium permanganate solution until a faint pink persists for 1 minute and the iron is completely oxidized.

5.10.3 Dilute this solution to 1 L with distilled water. 1 mL of this solution is equivalent to 0.0001 g Fe. This solution shall be stored in brown bottles and be protected from light.

5.10.4 *Standard Iron Solution*, 10 mL of the prepared stock solution, or its equivalent of purchased iron standard solution, shall be diluted to 100 mL with distilled water. 1 mL of this standard solution is equivalent to 0.00001 g Fe. The standard solution shall be freshly prepared for each analysis.

5.11 *Stock Copper Solution*, 3.9283 g of copper sulfate crystals ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) shall be dissolved in distilled water and diluted to 1 L with distilled water. 1 mL of this solution is equivalent to 0.001 g Cu.

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 A31 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.