Designation: ~~D6407 – 99 (Reapproved 2009)~~ D6407 – 99 (Reapproved 2014)

Standard Test Method for Analysis of Iron and Copper in Vegetable Tanning Materials[1]

This standard is issued under the fixed designation D6407; 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 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
D6404 Practice for Sampling Vegetable Materials Containing Tannin
D6405 Practice for Extraction of Tannins from Raw and Spent Materials
2.2 *ALCA Methods:*
A31 Method for Copper and Iron in Tanning Materials[3]

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 D4901, D6404, or D6405.

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.

---

[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.

Current edition approved ~~April 1, 2009~~Nov. 1, 2014. Published ~~July 2009~~December 2014. Originally approved in 1999. Last previous edition approved in ~~2004~~2009 as D6407 – 99 ~~(2004).~~(2009). DOI: ~~10.1520/D6407-99R09.~~10.1520/D6407-99R14.

[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.

5.7 *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 [$FeSO_4$•~~(NH·~~$(NH_4)_2SO_4$•~~6H·~~$6H_2O$] 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 ($CuSO_4$•$5H_2$ 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.

5.11.1 *Standard Copper Solution,* 10 mL of the stock copper solution shall be diluted to 1 L with distilled water and the pH adjusted to between 5.5 and 6.0. 1 mL of this standard solution is equivalent to 0.00001 g Cu. The standard solution shall be freshly prepared for each analysis.

5.12 *Xanthate Solution,* 1.0 g of potassium ethyl xanthate shall be dissolved in distilled water and diluted to 1 L with distilled water. The solution shall be freshly prepared for each analysis.

5.13 *Matched Nessler Tubes and Supporting Rack.*

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

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

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

5.17 *Dessicator,* any convenient form or size, using any normal desiccant.

5.18 *Glazed, Porcelain Dish or Crucible of Suitable Size.*

5.19 *Muffle Furnace,* capable of maintaining a temperature of 600° ± 25°C.

5.20 *Hotplate,* ordinary lab grade.

5.21 *Steam Bath,* ordinary lab grade.

5.22 *Volumetric Flasks,* 200 and 250 mL capacity.

5.23 *Beakers,* 250 mL.

5.24 *Filter Paper,* quantitative, Whatman grade 40 or 52 or similar.

5.25 *Buret,* 10 mL capacity is sufficient.

6. Test Specimen

6.1 The sample of material from which the test specimens are taken shall be prepared as described in Practice D6404 for extracts and tannery liquor and as in the Preparation of Sample section of Practice D6405 for raw and spent materials.

6.2 The specimen shall consist of 5 g of solid extract or its equivalent (that is 10 g of liquid extract; 25 to 50 g of tannery liquor; 5 g of raw or spent materials).

7. General Instructions

7.1 The distilled water shall be distilled from a glass, tin-lined, or block tin still and shall be stored in glass, tin-lined, or block tin containers.

7.2 All apparatus used in this analysis shall be cleaned with hot hydrochloric acid solution (diluted 1 to 1) and rinsed with distilled water before use.

7.3 Blank determinations shall be made to minimize errors due to iron or copper either present in the reagents used or picked up during the analysis.

7.4 Duplicate determinations are recommended whenever possible.