



Designation: D 3913 – 03

Standard Test Method for Acidity in Basic Chromium Tanning Liquors¹

This standard is issued under the fixed designation D 3913; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of the titratable acidity of chromium tanning liquors. By *titratable acidity* is meant not only free acidity, which is rarely present, but also anions combined with weakly basic cations such as Chromium (III), which can be titrated with base at the phenolphthalein end point.

1.2 *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:

D 3898 Test Method for Chromic Oxide in Basic Chromium Tanning Liquors²

E 180 Practice for Determining the Precision of ASTM Methods for the Analysis and Testing of Industrial Chemicals³

3. Summary of Test Method

3.1 A sample of suitable size is taken by diluting and aliquoting, if necessary. The sample is then heated to boiling and titrated hot to the first appearance of a pink color, stable to boiling in the supernatant solution, as determined by viewing the settled solution against the rim of a porcelain dish, or against the light in a tall beaker or Erlenmeyer flask.

4. Significance and Use

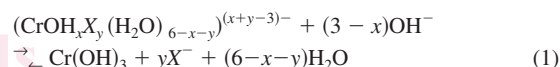
4.1 The acidity of a chrome tanning liquor, as determined by this test method, is rarely employed as such. This result is normally combined with the results of a chromium analysis (see Test Method D 3898) to determine a property of the liquor called basicity. This property, equal to the percentage by which anions in a normal chromic salt have been replaced by

hydroxyl in the solution being analyzed, is closely related to the tanning behavior of the solution.

5. Interferences

5.1 Although for most purposes, the results of this analysis are intended to give a measure of the acid anions associated with chromium, it should be mentioned that any weak basic cation will behave similarly. Hence, aluminum, zirconium, ferric iron, and weak organic bases will all yield salts with acidity titratable by this test method. A discussion of the effect of these interferences has been published.⁴

5.2 If there are anions which form very stable complexes with chromium present in the solution, the titration:



where:

X = stable complexes,

will not go to completion at the phenolphthalein endpoint and the acidity reported will be low. Large excess of oxalate or complexing anions of lesser affinity used for masking effect will introduce this error.⁵ To obviate this, an entirely different method has been developed in which the hydroxyl groups are titrated and the chromium is completely complexed by oxalate. This is generally referred to as the *Lehigh* method and may be used if complex ions are present in interfering amount.^{6,7}

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise specified, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁸ Other grades may be

⁴ Hartford, W. H., *JALCA*, Vol 56, 1961, p. 568.

⁵ Thorstensen, T. C., and Theis, E. R., *JALCA*, Vol 47, 1952, p. 583.

⁶ Theis, E. R., and Thorstensen, T. C., *Journal International Society Leather Trade Chemists*, Vol 31, 1947, p. 124.

⁷ Serfass, E. J., Theis, E. R., and Clark, A., Jr., *The Journal of the Leather Chemists Association*, Vol 42, 1947, p. 360.

⁸ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

¹ This test method is under the jurisdiction of ASTM Committee D-31 on Leather and is the direct responsibility of Subcommittee D31.06 on Chemical Analysis-General Methods. This test method was developed in cooperation with the American Leather Chemists Assoc.

Current edition approved May 10, 2003. Published July 2003. Originally approved in 1991. Last previous edition approved in 1993 as D 3913 – 93 (1998).

² *Annual Book of ASTM Standards*, Vol 15.04.

³ *Annual Book of ASTM Standards*, Vol 15.05.