Designation: D 6019 – 96

Test Method for Determination of Chromic Oxide in Basic Chromium Tanning Liquors (Ammonium Persulfate Oxidation)[1]

This standard is issued under the fixed designation D 6019; 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 covers the determination of chromic oxide in chrome tanning liquors, either simple, with added aluminum or zirconium, or with the usual masking complexing agents.

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. Summary of Test Method

2.1 The solution is oxidized with ammonium persulfate until the chromium is completely converted to the chromate ion. The solution is boiled to complete oxidation of organic materials and to destroy excess ammonium persulfate; it is then cooled and acidified. Potassium iodide is added, and the liberated iodine is titrated with standardized sodium thiosulfate.

3. Significance and Use

3.1 The procedure described is for the quality control for manufacturing liquors and specifications for the purchase of such liquors.

3.2 The chromium content of the liquors determines the amount to be used to obtain the desired degree of tannage, and hence may be a matter for specification in the purchase of leather.

4. Reagents and Materials

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, 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.[2] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean distilled water or water of equal purity.

4.3 *Ammonium Persulfate*—20 % solution 100 gm of persulfate into 500 mL water.

4.4 *Starch Indicator*, 1 %—Make a paste of 1 g of soluble starch in about 10 mL of water, add 90 mL water, and boil for 1 min with stirring. Cool and add one drop of chloroform. The solution is subject to decomposition and should be renewed if a deep blue color is not obtained on addition of one drop of indicator to a solution of 1 drop tincture of iodine in 100 mL of water.

4.5 *Sodium Thiosulfate Solution*, 0.1 *N*—Dissolve 24.85 g of $Na_2S_2O_3 \cdot 5H_2O$ in water, add 1 g Na_2CO_3, and dilute to 1 L.

4.5.1 *Standardization*—Dry potassium dichromate ($K_2Cr_2O_7$) in an oven at 130°C for 2 h and cool in a desiccator. Weigh into a glass stoppered Erlenmeyer flask (500 mL) about 0.2 g of potassium dichromate to an accuracy of 0.1 mg. Dissolve in 250 mL water, add 15 mL 1:4 hydrochloric acid, 20 mL of 10 % potassium iodide solution, stopper the flask, and allow to stand 5 min in the dark. Titrate with the sodium thiosulfate to be standardized. When the color of the solution has faded to a brownish-green, add 2 mL of 1 % starch solution, and continue titrating until the deep blue color changes to a clear green. Record the titration.

$$\textit{normality of sodium thiosulfate} = \frac{\text{weight potassium dichromate}}{0.04903 \times \text{mL titration}} \quad (1)$$

The thiosulfate solution is quite stable but should be restandardized at least once a month.

4.6 *Potassium Dichromate*, $K_2Cr_2O_7$, primary standard grade.

4.7 *Potassium Iodide*, 6 %—Dissolve 6 g KI in 100 mL water.

[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.

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[2] *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.