



Designation: D 6656 – 01

Standard Test Method for Determination of Chromic Oxide in Wet Blue (Perchloric Acid Oxidation)¹

This standard is issued under the fixed designation D 6656; 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 wet blue that has been partly or completely tanned with chromium compounds. In general, the samples will contain chromium content between 1 % and 5 % when calculated as chromic oxide expressed upon a dry basis otherwise referred to as moisture-free basis (mfb).

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.* See Section 9 for specific safety hazards.

2. Referenced Documents

2.1 ASTM Standards:

- D 6658 Test Method for Volatile Matter (Moisture) of Wet Blue by Oven Drying²
- D 6659 Practice for Sampling and Preparation of Wet Blue for Physical and Chemical Tests²
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

3. Terminology

3.1 *Definitions*—The terms and definitions employed within this method are commonly used in normal laboratory practice and require no special comment.

4. Summary of Test Method

4.1 The perchloric acid method may be applied to wet blue. Accurately weighed wet blue samples are digested in a blend of concentrated nitric acid and a prepared “oxidation mixture” consisting of sulfuric and perchloric acids. Once completed, oxidation of all tri-valent to hexavalent chrome is executed by controlled heating. Upon dilution, the chromium is indirectly (back) titrated volumetrically with standardized thiosulfate

using released iodine as the titrate. The perchloric acid method requires less manipulation than procedures based upon fusion of the ash. However, care must be taken because of potential hazards in the use of this reagent.

5. Significance and Use

5.1 The procedure described is specific for chromium in wet blue. Vanadium is the only common interfering element and is rarely present in quantity. The precision and accuracy of the methods are usually, at least, as good as the sampling of wet blue itself.

5.2 The chromium content of wet blue is related to the degree of tannage obtained, and hence may be a matter for specification in the purchase of wet blue. The procedure described provides adequate accuracy for this purpose.

6. Apparatus

- 6.1 *Analytical Balance*—accurate and calibrated to 0.001 g.
- 6.2 *Erlenmeyer Flasks*—250 ml capacity or equivalent.
- 6.3 *Burette*—50 ml capacity of suitable calibration grade, minimum calibration of 0.1 ml.
- 6.4 *Glass Anti-Bumping Beads*—or equivalent.
- 6.5 *Measuring Cylinders*—of 50 ml capacity or equivalent.
- 6.6 *Small Glass Filter Funnel*.
- 6.7 *Dessicator*—of suitable size and design and charged with fresh dessicant.
- 6.8 *Weighing Vessels*—of suitable size and design.
- 6.9 *Drying Oven*—with accurate variable temperature controls.

7. Reagents and Materials

7.1 *Purity of Reagents*—Analytical Reagent (AR) grade shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades

¹ This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.02 on Wet Blue.

Current edition approved May 10, 2001. Published May 2001.

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

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

⁴ *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, UK and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.