



Designation: D 6714 – 01

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

This standard is issued under the fixed designation D 6714; 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 %, calculated as chromic oxide expressed upon a dry basis otherwise referred to as moisture-free basis (mfb).

1.2 This test method is specific in that it applies to a sample or samples that are the resultant ash following the execution of Test Method D 6716.

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.* Specific hazards are given in Section 8.

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
- D 6716 Test Method for Total Ash in Wet Blue²
- 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 test 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 the ash obtained in Test Method D 6716. The resultant ash is dissolved

into acidic solution in a blend of nitric acid and a prepared oxidation mixture consisting of sulfuric and perchloric acids. Once completed, oxidation of all trivalent to hexavalent chromium 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 on fusion of the ash. However care must be taken because of potential hazards in the use of perchloric acid. The perchloric acid method also tends to give low results.

5. Significance and Use

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

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*, 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.
- 6.10 *Hot Plate*, with accurate variable temperature controls.

¹ This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.02 on Blue Stock.
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² *Annual Book of ASTM Standards*, Vol 15.04.

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