Designation: D6716 – 08 (Reapproved 2013)

Standard Test Method for Total Ash in Wet Blue or Wet White[1]

This standard is issued under the fixed designation D6716; 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 total ash in wet blue and wet white.

1.2 For total ash in wet white, the procedure is identical; substitute wet white for wet blue in the standard.

1.3 Total ash in wet blue may be reported upon a number of different bases (for example, fat-free, moisture-free, as received, excluding chromium, and so forth). Before proceeding with any tests, it is very important to determine upon which basis that the total ash is to be reported and to identify all other test methods that will be required to be executed in order to achieve the determined reporting method.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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]

D3495 Test Method for Hexane Extraction of Leather

D6658 Test Method for Volatile Matter (Moisture) of Wet Blue by Oven Drying

D6659 Practice for Sampling and Preparation of Wet Blue for Physical and Chemical Tests

D6714 Test Method for Chromic Oxide in Ashed Wet Blue (Perchloric Acid Oxidation)

3. Terminology

3.1 *Definitions:*

3.1.1 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 weighed sample is ignited in air at 600 ± 25°C until constant mass is attained. The weighed residual matter is termed “ash” and is calculated as a percentage of the original sample.

5. Significance and Use

5.1 This test method is useful in determining the approximate amount of nonvolatile inorganic material in wet blue. This may be in the form of salts or oxides of the elements. In a mixed-chrome tannage, the approximate percentage of other elements in the wet blue may be determined by subtracting the chromic oxide that may be conveniently determined on the ash. (See Test Method D6714.)

5.2 The specified temperature of 600°C is high enough to produce a reproducible result but it does not completely dehydrate such oxides as aluminum oxide (Al_2O_3) and chromic oxide (Cr_2O_3). Likewise, such salts as sulfates and phosphates may be incompletely dehydrated, and if alkalis and chromium are present simultaneously, oxidation to chromate may occur. Therefore, caution is advised in drawing conclusions based on quantitative relations of the elements.

6. Apparatus

6.1 *Crucible,* 30- to 50-mL, high-form, platinum or porcelain.

6.2 *Electric Muffle Furnace,* with controller or rheostat and pyrometer, capable of maintaining a temperature of 600 ± 25°C.

6.3 *Dessicator,* of appropriate size and charged with fresh dessicant.

6.4 *Analytical Balance,* capable of accurate weighings to within 0.001 g.

7. Test Specimen

7.1 The specimen shall consist of 2 to 10 g of wet blue from the composite sample, prepared in accordance with Practice D6659.

[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.

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[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.

NOTE 1—Typically, wet blue is a combination of organic hide substance in conjunction with inorganic chromium tanning salts. However, under some circumstances, silicones or other solvent-soluble organo-metallic complexes (including electrolyte-stable fat liquors) are added during manufacture and may be present within the sample. It may be desirable to calculate ash upon an extracted (fat free) basis, and if so, this should be indicated within the final report. To report ash upon an extracted basis, it will be necessary to execute Test Method D3495 on a portion of the same sample and weighed out at the same time as the specimen for total ash determination.

8. Procedure

8.1 Weigh accurately into a tared crucible 2 to 10 g (±0.001 g) of wet blue, prepared as described in 7.1, and preferably at sufficiently close equilibrium with the laboratory humidity that it does not gain or lose mass (moisture) at a significant rate.

8.2 Place the crucible and sample in the muffle furnace and maintain at 600 ± 25°C for at least 4 h, or longer if necessary, to destroy carbonaceous matter (Note 2).

8.3 To prevent any loss of ash, very carefully remove the crucible from the furnace, cool in a desiccator, and weigh (Note 3). Ensure that the transfer of the crucible from oven to dessicator is smooth and that there are no external air draughts that could cause loss of ash.

8.4 Replace in the furnace and maintain at 600 ± 25°C for another 15 min.

8.5 Repeat the weighing operation.

8.6 Continue heating for 15 min and weighing as described above until a mass constant within 0.002 g (2 mg) is obtained.

8.7 Record the final weight.

NOTE 2—The procedure in Section 8 is satisfactory with most wet blue. However, with wet blue that is known or suspected to have a very high grease content or has been heavily treated with fats and oils during processing, start with a cold muffle and raise temperature gradually to 600°C, or burn off the oil carefully over a gas burner before placing the crucible in the hot furnace.

NOTE 3—If it is difficult to burn off the carbon, as evidenced by inspection or failure to achieve constant mass, moisten the ash with a few drops of 1:1 nitric acid, dry carefully over a low flame, and then transfer to the muffle furnace and heat as before. If this procedure is unsuccessful, digest the ash in the crucible with 15 to 20 mL of hot water for a few minutes, and filter the suspension through an ashless high-retention filter paper. Transfer the paper and insoluble residue to the crucible and ignite at 600 ± 25°C as described above. Cool, add the filtrate to the crucible, evaporate carefully to dryness, then ignite at 600 ± 25°C to constant mass as described previously.

9. Calculation

9.1 Calculate the percentage of ash in the wet blue weighed as follows:

$$\text{Ash, \%} = [(A - B) \div (C - B)] \times 100$$

where:

A = weight of the ash and crucible,
B = weight of the crucible, and
C = weight of the sample and crucible.

9.2 If it is desired to convert the percentage obtained above to the dry basis, perform a moisture determination (Test Method D6658) on a portion of the same sample weighed under the same conditions as in Section 8. If the percentage moisture found is D, then calculate the percentage of ash on a dry basis as follows:

$$\text{Ash, dry basis, \%} = [(A - B) \div (C - B)] \times [100/(1 - D/100)]$$

where A, B, and C have the same meaning as in 9.1.

10. Precision and Bias

10.1 *Precision*—The precision of this test method is largely limited by the homogeneity of the sample in a complex natural material such as wet blue.

10.2 *Repeatability*—At the 95 % confidence level, duplicate determinations by the same operator should not differ by more than 0.14 % ash.

10.3 *Reproducibility*—At the 95 % confidence level, the average of duplicate determinations in each of two laboratories by different operators should not differ by more than 0.20 % ash.

10.4 *Bias*—Inasmuch as all wet blue contains an unknown amount of natural or inherent ash, no meaningful statement can be made with respect to bias.

11. Keywords

11.1 aluminum oxide; ash; blue stock; chrome content; chromic oxide; total ash; wet blue; wet white