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Standard Test Method for Total Ash in Leather[1]

This standard is issued under the fixed designation D2617; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of total ash in leather.

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:*[2]

D2807 Test Method for Chromic Oxide in Leather (Perchloric Acid Oxidation)

D2813 Practice for Sampling Leather for Physical and Chemical Tests

3. Summary of Test Method

3.1 The 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.

4. Significance and Use

4.1 This test method is useful in determining the approximate amount of nonvolatile inorganic material in leather. 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 leather may be determined by subtracting the chromic oxide that may be conveniently determined on the ash. (See Test Method D2807.)

4.2 The temperature of 600°C specified 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 alkalies and chromium are present simultaneously, oxidation to chromate may occur. Therefore, caution is advised in drawing conclusions based on quantitative relations of the elements.

5. Apparatus

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

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

6. Test Specimen

6.1 The specimen shall consist of 1 to 5 g of leather from the composite sample prepared in accordance with an accepted procedure.[3]

NOTE 1—In some leathers, silicones or other organometallic complexes that are solvent, soluble, and ash producing are used. It may be desirable to obtain ash on an extracted sample, and if so, it should be indicated on the report.

7. Procedure

7.1 Weigh accurately (to 1 mg) into a tared crucible 1 to 5 g of leather, prepared as described in 6.1, and preferably at sufficiently close equilibrium with the laboratory humidity that it does not gain or lose mass at a significant rate. Place the crucible and sample in the muffle furnace and maintain at 600 ± 25°C for at least 15 min after the absence of char or glowing embers, to destroy carbonaceous matter (Note 2). Remove the crucible from the furnace, cool in a desiccator, and weigh (Note 3). Replace in the furnace and maintain at 600 ± 25°C for another 15 min. Repeat the weighing operation. Continue heating for 15 min and weighing as described above until a mass constant within 0.2 mg is obtained. Record the final mass.

NOTE 2—The above procedure is satisfactory with most leathers. With heavily oiled or stuffed leather, 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

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.06 on Chemical Analysis. This test method was developed in cooperation with the American Leather Chemists Assn. (Standard Method B15 – 1969).

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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.

[3] Acceptable procedures are published in the *Journal of the American Leather Chemists Association*, Vol 51, 1956, p. 497; see Practice D2813.

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drops of 1+1 nitric acid, dry carefully over a low flame, and then transfer to the muffle 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 above.

8. Calculations

8.1 Calculate the percentage of ash in the leather weighed as follows:

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

where:

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

8.2 If it is desired to convert the percentage obtained above to the dry basis, perform a moisture determination on a portion of the same sample weighed under the same conditions as in Section 7. If the percentage moisture found is D, then calculate the percentage of ash on a dry basis as follows:

$$\text{Ash, dry basis, \%} = [(A - B)/(C - B)] \times [100/(1 - D/100)] \quad (2)$$

where A, B, and C have the same meaning as in 8.1.

8.3 The ash may be determined on a sample conditioned as to temperature and relative humidity. In this case, weigh the sample under identically controlled temperature and humidity, and report the results in such a manner as to indicate this fact, for example:

$$\text{Ash } (X\ ^\circ C, Y\ \% \text{ relative humidity}) = Z\ \% \quad (3)$$

9. Precision and Bias [4]

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

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

9.1.2 *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.

9.2 *Bias*—Inasmuch as all leathers contain an unknown amount of natural or inherent ash, no meaningful statement can be made with respect to bias.

10. Keywords

10.1 aluminum oxide; ash; chromic oxide

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D31 -0089.

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