Designation: D 2617 – 96

Standard Test Method for Total Ash in Leather[1]

This standard is issued under the fixed designation D 2617; 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 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:*

D 2807 Test Method for Chromic Oxide in Leather (Perchloric Acid Oxidation)[2]

D 2813 Practice for Sampling Leather for Physical and Chemical Tests[2]

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 D 2807.)

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, or longer if necessary 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 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.

[1] This test method is under the jurisdiction of ASTM Committee D-31 on Leather and is the direct responsibility of Subcommittee D31.06.01 on General Methods. This test method was developed in cooperation with the American Leather Chemists Assn. (Standard Method B 15 – 1969).

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

[3] Acceptable procedures are published in the *Journal of the American Leather Chemists Association*, Vol 51, 1956, p. 497; see Practice D 2813.