Designation: D5347 – 95 (Reapproved 2012)

Standard Test Method for Determination of the Ash Content of Fats and Oils[1]

This standard is issued under the fixed designation D5347; 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 the ash content of fats and oils used in the softening and stuffing of leather and in the manufacture of fatliquors and other softening and stuffing compounds. This test method was derived from Test Method D1951.

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

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.* See Section 5 for specific hazard statements.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1951 Test Method for Ash in Drying Oils and Fatty Acids (Withdrawn 2003)[3]

3. Significance and Use

3.1 This test method is intended to determine the ash content of fats and oils used in the softening and stuffing of leather, as well as those used in the manufacture of products for such purpose. The ash content of fats and oils is measured for the purpose of quality assurance.

4. Apparatus

4.1 *Crucible,* porcelain or high-silica glass (Note 1), 50-mL capacity.

NOTE 1—Platinum is not recommended. Boiled oils or oils contaminated with driers containing lead may ruin platinum by alloy formation.

4.2 *Electric Muffle Furnace.*

4.3 *Desiccator,* containing an efficient desiccant. Anhydrous calcium sulfate ($CaSO_4$), phosphorus pentoxide (P_2O_5) or concentrated sulfuric acid (H_2SO_4, sp gr 1.84) are satisfactory. (**Warning—**See 5.1 and 5.2 for specific hazards.)

NOTE 2—Magnesium perchlorate and barium perchlorate are also efficient desiccators and were previously listed in this section. However because of their explosive danger, and the availability of other safer materials, the recommendation for their use has been discontinued.

4.4 *Oil Sample Bottle,* 4-oz (120-mL).

4.5 *Triangle,* Nichrome or clay.

5. Hazards

5.1 *Phosphorus Pentoxide* is a strong oxidizer and reacts violently with water, reducing agents, and organic matter. Causes burns. Avoid contact with skin or eyes, or clothing, or inhalation as dust. Refer to supplier's Material Safety Data Sheet.

5.2 *Sulfuric Acid* is corrosive to skin, eyes and mucous membranes in the form of liquid, mist, or fumes. It causes severe burns. Take care to prevent the contact of the acid with eyes or skin or on clothing. In making dilute solutions, always add the acid to water with care. See supplier's Material Safety Data Sheet.

6. Procedure

6.1 Ignite the crucible in the muffle furnace at 550 to 650°C. Cool slightly, place in a desiccator for 1 h, and weigh to 0.1 mg.

6.2 Fill a 4-oz (120-mL) sample bottle with the sample and weigh to 0.05 g. Pour about 20 g of the sample from the bottle into the crucible supported on a triangle, using care so that no oil runs down the outside of the crucible or bottle.

6.3 Heat gently by moving a flame on the bottom and sides of the crucible until the oil ignites. Reduce the size of the flame until the heat is just sufficient to keep the sample burning. When the first batch of oil has burned out, add about 20 g more of the sample and continue in the same manner until all of the oil in the 4-oz bottle has been added. Reweigh the sample

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and OilsThis test method was developed in cooperation with the American Leather Chemists Assn. (Method H 27-1957).

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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] The last approved version of this historical standard is referenced on www.astm.org.

bottle to obtain the total weight of sample used, which will be somewhat over 100 g.

6.4 Continue heating the crucible until the oil is oxidized to a black char and transfer to a muffle furnace. Heat at 550 to 650°C for 1 h. Remove from the furnace, cool slightly, place in a desiccator, and cool to room temperature. Weigh and repeat heating in the furnace to constant weight (within 0.1 mg).

7. Calculation

7.1 Calculate the ash content, A, of the sample as follows:

$$A, \% = (R/S) \times 100 \quad (1)$$

or,

$$A, \text{ ppm} = (R/S) \times 1\,000\,000 \quad (2)$$

where:

R = residue (6.4), g, and
S = sample used, g.

8. Precision and Bias

8.1 Precision and bias were not established at the time this test method was written. An effort is being made to obtain the precision and, if obtainable, it will be published in future revisions. This test method has been in use for many years, and its usefulness has been well established.

9. Keywords

9.1 ash content; fatliquors; fats and oils; leather; softening and stuffing compounds