Designation: D 4655 – 95

Standard Test Methods for Sulfates in Leather (Total, Neutral, and Combined Acid)[1]

This standard is issued under the fixed designation D 4655; 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 These test methods are intended for use in determining the total, neutral, and combined acid sulfate in mineral-tanned leather.

1.2 The values stated in SI units are to be regarded as the 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

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

D 4654 Test Method for Sulfate Basicity in Leather[2]

3. Significance and Use

3.1 These test methods are used to determine the basicity of leather when used in accordance with Test Method D 4654.

4. Apparatus

4.1 *Volumetric Flask*, 250 mL.

4.2 Filter paper, ashless, fine grained and porcelain crucible.

4.3 Crucible, Gooch, with porous porcelain filter (optional).

5. Reagents

5.1 *Ammonium Hydroxide Solution*, (0.1 *N*)—7 mL/L reagent grade concentrate NH_4OH. Optional: Potassium dihydrogen phosphate, 0.1 molar solution (13.6 g/L KH_2PO_4) or sodium dihydrogen phosphate, 0.1 molar solution (13.8 g/ L NaH_2PO_4-H_2O).

5.2 *Hydrochloric Acid Solution*, (1.5 *N*)—125 mL/L reagent grade concentrate hydrochloric acid.

5.3 *Barium Chloride Solution*—($BaCl_2 \cdot 2H_2O$), 1 %.

5.4 *Sodium Hydroxide Solution*, 0.01 *N*, 0.4 g/L.

5.5 *Mixed Indicator*, consisting of 60 mL of a 0.1 % solution of methyl red and 40 mL of a 0.1 % solution of methylene blue, both in 95 % alcohol.

6. Sampling, Test Specimens, and Test Units

6.1 The specimen for each determination shall consist of 1 g leather from the composite sample (See Practice D 2813).

6.2 Two specimens from the composite sample shall be tested for each determination.

7. Procedure

7.1 *Total Sulfates*—Weigh the specimen to the nearest milligram and record the value as W_1. Transfer the specimen to a 250-mL volumetric flask and add 200 mL of 0.1 *N* ammonium hydroxide or 0.1 molar potassium or sodium dihydrogen phosphate solution. Immerse the flask up to the neck in a bath of boiling water. Thoroughly wet all products by swirling occasionally. After 2 h cool the flask to room temperature, and make up to volume with distilled water, shake, and without delay filter through a folded filter paper. Discard the first 20 to 25 mL of the filtrate. Pipette 200 mL of the filtrate into a 600 mL beaker and add about 20 mL of 1.5 *N* hydrochloric acid. Heat the solution to boiling and while boiling and stirring the solution, add 20 mL of a 1 % solution of barium chloride dropwise. Keep the covered beaker in a warm place at least for 2 h and preferably overnight.

7.1.1 Filter the precipitate through a fine grained ashless filter paper and wash with hot water until free from chloride. A weighed Gooch crucible or a weighed porous crucible may be used as an alternative for the filtration. Transfer the paper with the precipitate to a weighed crucible and ignite gently, either over a gas burner or in a muffle oven at 900°C for 1 h. Cool the crucible in a desiccator, weigh, and record the value of the $BaSO_4$ as W_2.

7.2 *Neutral Sulfates*—Weigh the specimen to the nearest milligram and record the value as W_3. Transfer the specimen to a 250-mL volumetric flask and add 200 mL of distilled water. Immerse the flask up to the neck in boiling water. Thoroughly wet all particles by swirling occasionally. After 2 h cool flask to room temperature, make up to volume with distilled water, shake and without delay filter through a folded filter. Discard the first 20 to 25 mL of the filtrate. Pipette 200 mL of the filtrate into a 600-mL beaker and titrate with 0.01 *N* sodium hydroxide, using a few drops of the methyl red/methylene blue indicator. Record the results as mL 0.01 *N* NaOH. After addition of 4 to 5 mL of 1.5 *N* hydrochloric acid, heat the solution to boiling. While boiling and stirring the solution, add

[1] These test methods are under the jurisdiction of ASTM Committee D-31 on Leather and are the direct responsibility of Subcommittee D31.06 on Chemical Analysis—General Methods. This test method was developed in cooperation with the American Leather Chemists Assn. (Method D 20–1956).

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