Designation: D4655 – 95 (Reapproved 2017)

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

This standard is issued under the fixed designation D4655; 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 U.S. Department of Defense.

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 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.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2813 Practice for Sampling Leather for Physical and Chemical Tests

D4654 Test Method for Sulfate Basicity in Leather

3. Significance and Use

3.1 These test methods are used to determine the basicity of leather when used in accordance with Test Method D4654.

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 D2813).

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

[1] These test methods are under the jurisdiction of ASTM Committee D31 on Leather and are the direct responsibility of Subcommittee D31.06 on Chemical Analysis. This test method was developed in cooperation with the American Leather Chemists Assn. (Method D20–1956).

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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.

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 10 mL of a 1 % barium chloride solution dropwise. Keep the covered beaker in a warm place at least for 2 h and preferably overnight.

7.2.1 Follow the directions for filtration given in 7.1.1. Record the value of the $BaSO_4$ as W_4.

8. Calculation of Results

8.1 Calculate the total sulfate content of the specimen as follows:

$$\%\ \text{total sulfate}\ (SO_4) = W_2 \times .4115/W_1 \times 250/200 \times 100 \qquad (1)$$

where:

W_1 = the weight of the specimen, and
W_2 = the weight of the $BaSO_4$.

8.2 Calculate the neutral sulfate content of the specimen as follows:

$$\%\ \text{neutral sulfate}\ (SO_4) = (W_4 \times .4115 - N \times A \times .048)/W_3 \times 250/250 \times 100 \qquad (2)$$

where:

W_3 = the weight of the specimen,
W_4 = the weight of the $BaSO_4$,
A = the millilitre of standard NaOH, and
N = the normality of the standard NaOH.

8.3 Calculate the combined acid sulfate content of the specimen as follows:

$$\%\ \text{combined acid sulfate}\ (SO_4) = \%\ \text{total sulfate} - \%\ \text{neutral sulfate}. \qquad (3)$$

8.3.1 The total, neutral and combined acid sulfates in the sample for test shall be the average of the test results obtained from the specimens tested.

9. Report

9.1 Unless otherwise specified in the detail specification, the results shall be reported to the nearest 0.1 %.

10. Precision and Bias

10.1 This test method is adopted from the procedures of the American Leather Chemists Association where it has long been in use and where it was approved for publication before the inclusion of precision and bias statements were mandated. The original interlaboratory test data is no longer available. The user is cautioned to verify by the use of reference material if available that the precision and bias of this test method is adequate for the contemplated use.

11. Keywords

11.1 combined acid sulfate; mineral tanned leather; neutral sulfates; total sulfates