Designation: D6017 – 97 (Reapproved 2015)

Standard Test Method for Determination of Magnesium Sulfate (Epsom Salt) in Leather[1]

This standard is issued under the fixed designation D6017; 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 This test method covers quantitatively determining the magnesium sulfate (epsom salt) in 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3790 Test Method for Volatile Matter (Moisture) of Leather by Oven Drying

2.2 *Federal Standards:*

Federal Test Standard No. 311, Method 6541 Magnesium Sulfate (Epsom Salt)[3]

3. Significance and Use

3.1 This test method distinguishes magnesium sulfate from other water soluble non-tanning salts found in leather.

4. Apparatus

4.1 *Platinum Crucible.*

4.2 *Filtering Crucible.*

4.3 *Suction Flask.*

4.4 *Muffle Furnace.*

5. Reagents

5.1 *Hydrochloric Acid,* specific gravity 1.19.

5.2 *Nitric Acid,* specific gravity 1.42.

5.3 *Ammonium Chloride.*

5.4 *Ammonium Hydroxide,* 1 to 1 and 1 to 9 parts by volume with distilled water.

5.5 *Ammonium Nitrate Solution,* 10 g dissolved in distilled water and made up to 100 mL.

5.6 *Ammonium Oxalate,* saturated solution.

5.7 *Ferric Chloride Solution,* 10 g dissolved in distilled water and made up to 100 mL.

5.8 *Methyl Orange Indicator,* 0.05 %.

5.9 *Sodium Ammonium Hydrogen Phosphate,* saturated solution.

6. Procedure

6.1 Unless otherwise specified in the material specification, determine the moisture content of the composite sample from which the test specimen is drawn, in accordance with Test Method D3790. Determine the weight of the composite specimens for moisture content at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

6.2 Place the specimen in a tared porcelain crucible, 5 g weighed to the nearest 0.001 g, and record the value as W_2. Place the weighed specimen in a cold muffle furnace or precarbonize over a burner prior to placing in a hot furnace. Gradually raise the temperature of the furnace to 600 ± 25°C and maintain at this temperature for 60 min. Remove the crucible and contents, cool in a desiccator, and weigh. Replace in the furnace at 600 ± 25°C for 30 min and repeat the cooling and weighing procedure until a constant weight is obtained (±0.01 g). If it is difficult to obtain a constant weight, leach the residue with hot distilled water and filter through an ashless filter paper. Place the filter paper in the crucible and ash. Add

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[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 from Federal Test Method Standard No. 311, Method 6541 in cooperation with the U.S. Army Natick Research Development & Engineering Center, Natick MA and the Defense Personnel Support Center Directorate of Clothing and Textiles, Philadelphia PA.

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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] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

the filtrate to the crucible and evaporate. The crucible shall then be put back in the muffle furnace and heated, cooled, and weighed as above until a constant weight (±0.01 g) has been obtained.

6.3 Moisten the ash with distilled water and add 15 mL of hydrochloric acid (specific gravity 1.19) to the crucible. Transfer the contents of the crucible to a 400 mL beaker and dilute to about 60 mL with distilled water. Add two drops of nitric acid (specific gravity 1.42) and heat the solution on a steam bath for about 15 min. Add ammonium hydroxide (1:1) dropwise, while stirring, until the solution is slightly alkaline to methyl orange or a faint odor of ammonia appears at this point. If the precipitate does not have the characteristic reddish-brown color of ferric hydroxide, dissolve the precipitate in hydrochloric acid, add a few drops of ferric chloride solution, and repeat the addition of a slight excess of ammonium hydroxide. Boil the contents of the beaker for a few minutes, then filter and wash free from chlorides with hot water, catching the filtrate and washings in a 400 mL beaker.

6.4 Add 3 g of ammonium chloride and evaporate the solution to about 175 mL; add 1 mL of ammonium hydroxide (1:1) and heat the solution to boiling. Add 10 mL of saturated ammonium oxalate solution slowly while stirring, then cover the beaker and set aside over a steam bath for at least 2 h. Transfer the solution and precipitate to a 250 mL volumetric flask, cool to room temperature, dilute to the mark, and mix well. Filter solution through a dry quantitative filter paper.

6.5 Transfer an aliquot of 50 mL to a beaker, dilute to about 150 mL with distilled water, add a few drops of methyl orange indicator, and add hydrochloric acid until slightly acid. Add a slight excess of saturated sodium ammonium hydrogen phosphate over that necessary to precipitate the magnesium (usually 5 mL is sufficient). While stirring vigorously, make the solution slightly ammoniacal by the dropwise addition of ammonium hydroxide (1:1) and set aside for 15 min. Add 5 mL of ammonium hydroxide (1:1) while stirring and set the solution aside overnight at room temperature.

6.6 Filter the solution through a filtering crucible, transfer the precipitate to the platinum crucible, and wash free from the chlorides with ammonium hydroxide (1:9) solution. Moisten the precipitate with a few drops of 10 % ammonium nitrate solution, dry cautiously, ignite for 1 h to a constant weight (±0.001 g) at 1000 to 1100°C, cool in a desiccator to room temperature, immediately weigh the value, and record as W_1.

7. Calculation

7.1 Calculate the magnesium sulfate (epsom salt, $MgSO_4 \cdot 7H_2$) in the specimen as follows:

$$\textit{magnesium sulfate}\ (MgSO_4 \cdot 7H_2O),\ \%\ (\textit{moisture} - \textit{free basis}) \quad (1)$$

$$= \frac{W_1 \times 2.2147 \times 5 \times 100}{W_2 \times \frac{(100 - M)}{(100)}}$$

where:

W_1 = weight of the ignited precipitate ($Mg_2P_2O_7$), g,
W_2 = weight of the specimen, g, and
M = moisture content of leather.

NOTE 1—See Test Method D3790.

8. Report

8.1 The magnesium sulfate in the specimen is the average of the results obtained from the specimens tested and reported to the nearest 0.1 %. Report individual results utilized in obtaining the average.

9. Precision and Bias

9.1 This test method is adopted from Federal Test Standard No. 311, Method 6541 where it has long been in use and was approved for publication before the inclusion of precision and bias statements was mandated. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this test method is adequate for the contemplated use.

10. Keywords

10.1 epsom salt; leather; magnesium sulfate; soluble salts