



# Standard Test Method for Determination of Organically Combined Sulfuric Anhydride Ash-Gravimetric, Test Method C<sup>1</sup>

This standard is issued under the fixed designation D5352; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of the organically combined sulfuric anhydride existing in a sample of sulfonated or sulfated oil, or both, by extracting the undecomposed sulfonated or sulfated fat and other fatty matter over an acidulated, concentrated salt solution, and ashing the purified extract. This test method is applicable to all types of sulfonated and sulfated oils, including true sulfonic acid oils and those containing sodium acetate or similar partially titratable compounds. This test method was derived from Test Methods D500, Sections 25 through 28.

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:*<sup>2</sup>

D500 Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils

## 3. Significance and Use

3.1 This test method is intended for the determination of organically combined sulfuric anhydride in sulfonated and sulfated fats and oils.

## 4. Reagents

4.1 *Ethyl Ether.*

4.2 *Hydrogen Peroxide (30 %)*—Concentrated hydrogen peroxide ( $H_2O_2$ ).

4.3 *Methyl Orange Indicator Solution (1 g/L)*—Dissolve 0.1 g of methyl orange in 100 mL of water.

4.4 *Sodium Chloride (NaCl)*, solid.

4.5 *Sodium Sulfate (Na<sub>2</sub>SO<sub>4</sub>)*, anhydrous.

## 5. Procedure

5.1 The procedure consists of isolating and purifying the fatty matter as it exists in the original oil by dissolving the sample in a solvent, acidifying and washing with saturated brine, and ashing the purified extract. If the sample contains ammonia, the ammonia shall first be expelled before the determination is made on the sample.

5.1.1 *In the Absence of Ammonia*—Proceed as described in the separation of purified oil (6.1.1 Method B), combining the ether layers in the first funnel rather than in the decomposition flask. Carefully remove any water that may settle and dehydrate the ether layer as follows: add 5 g of anhydrous  $Na_2SO_4$ , shake vigorously for 5 min, and filter directly into a 150-mL beaker placed in a bath of warm water. Wash the flask and filter with ether until free from fat (absence of oil stains on the filter paper after drying) and add the filtrate to the beaker. To avoid creeping of the oil, the volume in the beaker should at no time during the filtering and washing exceed 50 mL (one third full). Evaporate the ether solution until the volume has been reduced to about 20 mL and transfer the residue to a tared 50-mL crucible (high form). Immerse the crucible in a 100-mL beaker containing warm water until practically all of the ether has evaporated. Rinse the beaker with two 10-mL and three 5-mL portions of ether, or until all of the oil has been transferred to the crucible; allow each portion of rinsing ether to evaporate before the next rinsing is made. Burn gently the solvent-free residue, and finally ignite at a dull red heat to constant weight. To prevent creeping of the oil and to hasten evaporation, stir the solution with a glass rod; before the oil is burned, wipe the rod clean with ashless filter paper and add the paper to the crucible. To oxidize traces of carbon or sodium sulfide that might form, moisten the ash with  $H_2O_2$  and again carefully ignite to constant weight. Calculate the percentage of ash as follows:

<sup>1</sup> 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 Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 44-1957).

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<sup>2</sup> 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.

$$\text{Extracted ash, \%} = (A/B) \times 100 \quad (1)$$

where:

$A$  = weight of ash, g, and

$B$  = weight of sample, g.

5.1.2 *In the Presence of Ammonia*—Dissolve 5 to 8 g of the sample in 80 mL of water in a 300-mL beaker. Add 10 mL of 1 *N* NaOH solution and boil the solution gently until wet litmus paper no longer indicates ammonia. Cool the solution, transfer to a 300-mL separatory funnel, and add about 35 g of solid NaCl or enough to make the final solution a 25 % NaCl solution. Add five drops of methyl orange solution and complete the neutralization, extraction, etc., in accordance with the procedure described in 5.1.1.

## 6. Calculation

6.1 Calculate the percentage of organically combined sulfuric anhydride as follows:

$$\text{Organically combined sulfuric anhydride, \%} = 1.1267 A \quad (2)$$

where:

$A$  = extracted ash, %, and

1.1267 = molecular ratio of  $2\text{SO}_3:\text{Na}_2\text{SO}_4$ .

## 7. Precision and Bias

7.1 Although this test method is widely used, precision and bias information is not available at this time.

## 8. Keywords

8.1 ash-gravimetric; leather; sulfated fats and oils; sulfonated fats and oils; sulfuric anhydride

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