Designation: D 5566 – 95 (Reapproved 2001)

Standard Test Method for Determination of Inorganic Salt Content of Sulfated and Sulfonated Oils[1]

This standard is issued under the fixed designation D 5566; 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 a sample of sulfonated or sulfated oil, or both, the inorganic sulfates, chlorides, and all other salts that are insoluble in a mixture of oleic acid and carbon tetrachloride.

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. Significance and Use

2.1 This test method is intended to be used for the determination of the inorganic salt content of sulfated and sulfonated fats and oils for the purpose of quality control.

3. Apparatus

3.1 *Gooch Crucible or Filter Paper*—Either may be used for filtering. Ignite the Gooch crucible in a larger crucible, supported by a ring and assembled as shown in Fig. 1. If filter paper is used, it may be a 9-cm general purpose ashless filter paper.

3.2 *Thermometer*.

4. Reagents

4.1 *Carbon Tetrachloride* (CCl_4).

4.2 *Ethyl Ether*.

4.3 *Oleic Acid*.

5. Procedure

5.1 The procedure consists of dehydrating the sample, dissolving in a solvent, filtering, igniting, and weighing the residue. In the presence of ammonium salts, the residue is not ignited but only dried to constant weight. The presence of sodium acetate does not interfere with this test method.

5.1.1 *In the Absence of Ammonium Salts*—Weigh 3 to 5 g of the sample and place in a 250-mL beaker, add an approximately equal amount of oleic acid, and heat the mixture on an oil bath, while stirring constantly with a thermometer, at a temperature of 105 to 110°C until practically free from water. Continue the heating until the temperature of the contents reaches 118 to 120°C and maintain at that temperature for about 5 min. If the dehydrated sample upon cooling does not remain liquid, add more oleic acid. Dissolve the dehydrated sample in 100 mL of CCl_4 warmed to 50 to 55°C, and filter through a counterpoised filter paper or a Gooch crucible. Pass 75 mL of CCl_4 through the crucible and again ignite, cool in a desiccator, and weigh. Repeat the process of washing with CCl_4 until there is no further loss in weight. Wash the residue with three 15-mL portions of a solution of oleic acid in CCl_4(2 %), then with six 15-mL portions of hot CCl_4, and finally with two 15-mL portions of ether or until the residue is free from oil. Take care that the top of the filter is thoroughly washed. Transfer the last traces of the residue to the filter by allowing the solvent to evaporate when the salts become free flowing. Dry the residue at 125 to 130°C for 45 min, cool in a desiccator, and weigh. Ignite the residue at a dull red heat for 15 min, weigh, and repeat the ignition until constant weight is obtained.

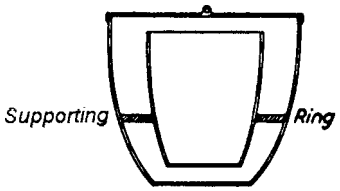

FIG. 1 Gooch Crucible Assembly for Determination of Inorganic Salts

5.1.2 *In the Presence of Ammonium Salts*—Proceed as described in 6.1.1 for the determination of inorganic salts in the absence of ammonium salts with the following exceptions: (*1*) in preparing the Gooch crucible, do not ignite but heat it at 105 ± 2°C for 45 min and repeat the heating until constant weight is obtained, and (*2*) heat the residue, whether in a Gooch crucible or on a filter paper as in (*1*), but do not ignite it.

6. Calculation

6.1 The method of calculation depends upon whether or not ammonium salts are present in the sample.

6.1.1 Calculate the inorganic sulfates and chlorides including ammonium salts as follows:

[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 Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 48-1957).

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Inorganic sulfates and chlorides, including ammonium salts, % = $(A/B) \times 100$ (1)

where:
A = weight of dried residue, g, and
B = weight of sample, g.

6.1.2 Calculate the nonvolatile inorganic sulfates and chlorides (in the absence of ammonium salts) as follows:

Nonvolatile, inorganic sulfates and chlorides; % = $(A/B) \times 100$ (2)

where:
A = weight of ignited residue, g, and
B = weight of sample, g.

NOTE 1—In the absence of ammonium salts, the difference between the percentage of the dried residue and the percentage of the ignited residue shall not be greater than 0.25 %.

7. Precision and Bias

7.1 This test method is adopted from the procedures of the American Leather Chemists Association, where it has long been in use and was approved for publication before the inclusion of precision and bias statements was mandated. The original interlaboratory test data are no longer available. 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.

8. Keywords

8.1 inorganic salts; leather; sulfated and sulfonated oils; sulfated oils