Designation: D5565 – 95 (Reapproved 2011)

Standard Test Method for Determination of the Solidification Point of Fatty Acids Contained in Animal, Marine, and Vegetable Fats and Oils[1]

This standard is issued under the fixed designation D5565; 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 determination of the solidification point of fatty acids contained in animal, marine, and vegetable fats and oils.

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.* See 5.2 and 5.7 for additional information.

2. Significance and Use

2.1 This test method is intended to cover determination of the solidification point of fatty acids contained in animal, marine, and vegetable fats and oils used in the softening and stuffing of leather, as well as those used in the manufacture of products for such purpose.

3. Apparatus

3.1 *Griffin Low-Form Beaker,* 2-L capacity.

3.2 *Wide Mouth Bottle*—Capacity of 450 mL, height of 190 mm, and inside diameter of neck of 38 mm.

3.3 *Test Tubes*—Length of 100 mm and diameter of 25 mm, with or without rim. These tubes may have an etched mark extending around the tube at a distance of 57 mm from the bottom to show the height to which the tube is to be filled.

3.4 *Saponification Vessel*—A750- or 1000-mL flask.

3.5 *Stirrer,* 2- to 3-mm outside diameter, with one end bent in the form of a loop of 19-mm outside diameter. Glass, nichrome, stainless steel, or monel wire shall be used. The upper end can be formed to accommodate hand stirring or for attachment to a mechanical stirrer.

3.6 *Laboratory Thermometer,* 0 to 150°C.

3.7 *Titer Test Thermometer*—Specifications for thermometer used in titer test determinations:

3.7.1 *Type*—Etched stem glass.

3.7.2 *Liquid*—Mercury.

3.7.3 *Filling Above Liquid*—Evacuated or nitrogen gas.

3.7.4 *Temperature Range*—2° to +68°C.

3.7.5 *Subdivisions*—0.2°C.

3.7.6 *Total Length*—385 to 390 mm.

3.7.7 *Stem Diameter*—6 to 7 mm.

3.7.8 *Stem Construction*—Plain or lens front. The cross section of the lens front type shall be such that it will pass through an 8 mm ring gage but will not enter a 5 mm slot gage.

3.7.9 *Bulb Diameter*—5.5 mm to not greater than that of stem.

3.7.10 *Bulb Length*—15 to 25 mm.

3.7.11 *Bulb Construction*—Corning normal or equally suitable thermometric glass.

3.7.12 *Distance from Bottom of Bulb to − 2° Mark*—50 to 60 mm.

3.7.13 *Distance from 68° Mark to Top of Thermometer*—20 to 35 mm.

3.7.14 *Length of Unchanged Capillary Between the Highest Graduation and the Expansion Chamber*—10 mm.

3.7.15 *Expansion Chamber*—To permit heating to at least 85°C.

3.7.16 *Top Finish*—Glass ring.

3.7.17 *Longer Graduation Lines*—At each 1° mark.

3.7.18 *Graduations*—Numbered at zero and each multiple of 2°.

3.7.19 *Immersion*—45 mm, a line shall be etched around the stem 45 mm from the bottom of the bulb.

3.7.20 *Special Marking on Thermometer*—A.O.C.S. Titer Test.

3.7.21 *Maximum Scale Error Permitted at any Point*—0.2°C.

3.7.22 *Marking on Case*—A.O.C.S. Titer Test, −2° to +68° in 0.2°C.

3.7.23 *Standardization*—The thermometer shall be standardized at the ice point and at intervals of approximately

[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. (Methods H 10 and H 17-1957).

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20°C, for the condition of 45 mm immersion, and for an average stem temperature of the emergent mercury column of 25°C.

3.8 *Filter Paper,* qualitative, rapid filtering grade.

4. Reagents

4.1 *Glycerol-Caustic Solution,* prepared by dissolving, with the aid of heat, 250 g of solid potassium hydroxide in 1250 g of glycerin (dynamite or C. P. grade). To avoid foaming, heating shall not exceed 135 to 145°C. (Sodium hydroxide can not be substituted for potassium hydroxide.)

4.2 *Sulfuric Acid,* 30 % by weight.

5. Procedure

5.1 *Preparation of the Fatty Acids*—Weigh approximately 110 g of glycerol-caustic solution into the saponification vessel and heat to 150°C with stirring. Then add approximately 50 mL of oil or melted fat sample and reheat the whole to 140 to 150°C. A little additional solution may be necessary to ensure complete saponification in some cases.

5.2 Continue stirring until the saponification is complete. (**Warning—**Do not heat to above 150°C.)

5.2.1 (The committee has investigated a number of tests for complete saponification, but none has been found up to the present time that is reliable under all circumstances. Familiarity with the changes that occur in the appearance and character of the mass usually enables determination of the proper end point. Saponification is usually indicated by a change in the appearance of the mass and is often accompanied by an increase in the viscosity, or thickening. When this occurs, the solution thins out again after the reaction is complete and assumes a homogeneous appearance. The most common characteristic is that of soap bubbles forming and rising from the surface. There are cases in which none of these criteria can be depended on, so that considerable care must be exercised at all times to ensure complete saponification.)

5.3 After cooling slightly, add 200 to 300 mL of distilled water and stir and heat the mass well until the soap is dissolved. Then, with stirring, add 50 mL of dilute H_2SO_4 and boil the whole until the fatty acids are melted completely and clear. Additional water may be added before or during boiling if desired.

5.4 Remove the aqueous layer, containing the H_2SO_4, add water again, and repeat boiling for 2 to 3 min or until the fatty acids are melted entirely and clear. Since acids of high melting point fats are sometimes slow to melt and clear, inspect the fatty acid layer while it is quiet to ensure that all has melted.

5.5 Remove the water again, and, if necessary, repeat the washings described in 5.4 until the wash water is neutral to a methyl orange indicator.

5.6 Transfer the fatty acids to a filter paper carefully, so as not to include any water. The filter paper may be supported on a small beaker without a funnel. The acids must remain completely melted until entirely filtered.

5.7 After heating the filtered acids on a hot plate to 130°C to remove traces of moisture, fill the titer test tube to a height of 57 mm from the bottom. (**Warning—**The sample shall not be held at 130°C or reheated to this temperature more than once. If excessive moisture is present, the water shall be allowed to settle and the fatty acids decanted and then refiltered and reheated. The acids must be thoroughly dry.)

5.8 *Solidification of the Fatty Acids*—Adjust the water bath to a temperature of 20 ± 1°C for all samples having titers of 35°C or higher, and 15 to 20°C below the titer point for all samples with titers below 35°C.

5.9 Place the test tubes, containing the fatty acids, in the water bath assembly. Insert the titer thermometer to the immersion mark so that it will be equidistant from the sides of the tube. Stir, with the stirring rod in a vertical manner, at a rate of 100 complete up-and-down motions/min. (Stirring may be performed mechanically by attaching a small motor with a suitable speed-reducing mechanism to the stirring rod.) The stirrer shall move through a vertical distance of approximately 38 mm, with agitation starting while the temperature is at least 10°C above the titer point.

5.10 Continue stirring at the directed rate until the temperature remains constant for 30 s or begins to rise in less than a 30-s interval. Discontinue stirring immediately, remove or raise the stirrer out of the sample, and observe the increase in temperature. The titer point is the highest temperature indicated by the thermometer during this rise. Expect duplicate determinations to agree within 0.2°C.

6. Report

6.1 Report the results in degrees Centigrade or Fahrenheit. Reference this test method as the procedure used in the test report.

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 fat liquors; fatty acids; leather; solidification point; titer test