Designation: D5558 – 95 (Reapproved 2017)

Standard Test Method for Determination of the Saponification Value of Fats and Oils[1]

This standard is issued under the fixed designation D5558; 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 the saponification value of 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.*

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. Significance and Use

2.1 This test method is intended for use in the determination of the saponification value of fats and oils used in the manufacture of fat liquors for the purpose of quality assurance.

3. Apparatus and Reagents

3.1 *Erlenmeyer Flasks,* Corning[2] alkali resistant, Kimble resistant, or equivalent, 250 or 300 mL.

3.2 *Air Condensers,* minimum length 650 mm.

3.3 *Water Bath or a Hot Plate,* with variable heat control.

3.4 *Hydrochloric Acid,* 0.5 *N*, accurately standardized.

3.5 *Alcoholic Potassium Hydroxide,*[3]added to a 2-L flask from 1 to 1.5 L of 95 % ethyl alcohol (U.S.S.D. Formula 3A[2] is permitted) and a few grams (5 to 10 g) of potassium hydroxide. After boiling under a reflux condenser on a water bath for 30 to 60 min, the alcohol shall be distilled and collected. For the preparation of alcoholic potassium hydroxide, 40 g of potassium hydroxide, low in carbonate, shall be dissolved in 1 L of the distilled alcohol, keeping the temperature below 15.5°C (60°F) while the alkali is being dissolved. This solution should remain clear.

4. Procedure

4.1 Melt the sample, if not already liquid, and filter through filter paper to remove any impurities and the last traces of moisture. The sample must be completely dry.

4.2 Accurately weigh a sample, of such size (usually 4 to 5 g) that the back titration is 45 to 55 % of the blank, and 50 mL of the alcoholic KOH is added with a pipette, allowing the pipette to drain for a definite period of time.

4.2.1 Prepare a blank determination and conduct simultaneously with the sample.

4.3 Connect air condensers to the flask and boil the solution gently but steadily until the sample is completely saponified. This usually requires approximately 1 h for normal samples. Take care that the vapor ring in the condenser does not rise to the top of the condenser or there may be some loss.

NOTE 1—Some samples particularly difficult to saponify may require more than 1 h. This can only be determined by trial. Clarity and homogeneity of the test solution are partial indicators of the complete saponification, but they are not necessarily absolute criteria.

4.4 After the flask and condenser have cooled somewhat, but not sufficiently to jell the contents, wash down the inside of the condenser with a little distilled water. Then disconnect the flask, add approximately 1 mL of indicator, and titrate the solution with 0.5 *N* HCl until the pink color has just disappeared.

5. Calculation and Report

5.1 Calculate the saponification number as follows:

$$\textit{saponification value} = \frac{28.05(A - B)}{\textit{weight of sample}} \quad (1)$$

where:

A = titration of blank, and
B = titration of sample.

[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 31-1957).

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[2] Product is widely available commercially.

[3] Alternately, commercially available potassium hydroxide solution 0.5 Nm methanol is said to work satisfactorily.

5.2 Reference this test method as the procedure used in the test report.

6. Precision and Bias

6.1 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.

7. Keywords

7.1 fats and oils; leather; saponification value