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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Saponification Value of Drying Oils, Fatty Acids, and Polymerized Fatty Acids[1]

This standard is issued under the fixed designation D 1962; 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 Department of Defense.

1. Scope

1.1 This test method covers the determination of the saponification value of drying oils, bodied oils, fatty acids, and polymerized fatty acids.

1.2 *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.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 305 Test Method for Solvent-Extractable Material in Black Pigments[2]

D 1193 Specification for Reagent Water[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *saponification value*—a measure of the alkali reactive groups in oils and fatty acids and is expressed as the number of milligrams of potassium hydroxide that react with 1 g of sample.

4. Significance and Use

4.1 The saponification value of oils and fatty acids is a measure of the content of ester linkages. For an oil, provided it is not significantly oxidized, the number of ester linkages per molecule (for example, three in a triglyceride), can be used to calculate the molecular weight of the oil.

4.2 A saponification value higher than normal indicates that the oil has been oxidized (blown) or chemically modified, for example, with other acids such as maleic, fumaric, or phthalic.

4.3 Saponification value alone is not a measure of the quality of the oil.

4.4 Chemically modified oils may require saponification times longer than 1 h for complete reaction.

5. Apparatus

5.1 *Erlenmeyer Flasks*, wide-mouth, alkali-resistant, 250 or 300-mL capacity.

5.2 *Condenser Loop*.

NOTE 1—Suitable condenser loops are shown in Figs. 1 and 2 of Test Method D 305.

5.3 *Steam Bath*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the Specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I of Specification D 1193.

6.3 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %), methanol or isopropanol.

NOTE 2—A "masked phenolphthalein indicator" may be used with off-color materials. Prepare by dissolving 1.6 g of phenolphthalein and 2.7 g of methylene blue in 500 mL of alcohol. Adjust the pH with sodium hydroxide (NaOH) or KOH solution so that the greenish blue color is faintly tinged with purple. The color change is from green to purple when going from acid to alkali.

6.4 *Potassium Hydroxide, Alcoholic Solution*—Place 5 to 10 g of potassium hydroxide (KOH) (**Caution**—see 7.1) in a 2-L flask and add 1 to 1.5 L of ethyl alcohol (95 %) or denatured alcohol conforming to Formula No. 30 or 3A of the U. S. Bureau of Alcohol, Tobacco and Firearms. Boil on a

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

water bath under a reflux condenser for 30 to 60 min. Distill and collect the alcohol. Dissolve 40 g of KOH in 1 L of the distilled alcohol, keeping the temperature below 15°C while the alkali is being dissolved. This solution should remain clear.

6.5 *Sulfuric or Hydrochloric Acid, Standard* (0.5 *N*)—Add about 15 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) (**Precaution**—see 7.2) or 45 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) (**Precaution**—see 7.3) to about 900 mL of water, cool, and dilute to 1 L. Standardize against freshly standardized sodium hydroxide (NaOH) solution or by any other accurate method.

7. Hazards

7.1 *Potassium Hydroxide* and its strong solutions are caustic to the skin and eyes. Avoid all contact with skin and eyes. In case of contact, immediately flush eyes for 15 min and wash hands with plenty of cold water. Call a physician. Remove contaminated clothing and wash before reuse. See suppliers' Material Safety Data Sheet for further information.

7.2 *Sulfuric Acid* is corrosive to skin, eyes and mucous membranes in the form of liquid, mist, or fumes. It causes severe burns. Take care to prevent the contact of the acid with eyes, skin or on clothing. In making dilute solutions, always add the acid to water with care. In case of contact, immediately flush eyes with copious amounts of water for 15 min; flush skin with water (use shower if available); wash contaminated clothing before reuse. Immediately call a physician. See suppliers' Material Safety Data Sheet.

7.3 *Concentrated Hydrochloric Acid* is corrosive and may cause burns to the skin and eyes; the vapor is irritating to mucous membranes. Avoid contact with skin and eyes. In case of contact, wash skin and flush eyes with cold water for 15 min. Remove contaminated clothing. Call a physician. Wash clothing before reuse. See suppliers' Material Safety Data Sheet.

8. Procedure

8.1 To an Erlenmeyer flask, transfer a specimen weight of such size, weighed to 1 mg, that the back titration is 45 to 55 % of the blank. Add 25 mL of alcoholic KOH solution to the flask and to one or more additional flasks to be carried through as blanks. Place a condenser loop inside the neck of each flask and heat on the steam bath for 1 h.

NOTE 3—Certain synthetic oils are not completely saponified in 1 h. Run chemically modified drying oils in duplicate, using 1 and 2-h heating periods to establish completeness of saponification. If the 2-h heating gives appreciably higher results than the 1-h run, additional determinations using 4 and 6-h heating periods should be run to establish the time required for complete reaction.

8.2 Cool the solution, add phenolphthalein indicator (Note 2), and titrate with 0.5 *N* H_2SO_4 or HCl until the pink color has just disappeared.

9. Calculation

9.1 Calculate the saponification number, *P*, as follows:

$$P = [(B - V)N \times 56.1]/S$$

where:

V = H_2SO_4 or HCl required for titration of the specimen, mL,
B = H_2SO_4 or HCl required for titration of the blank, mL,
N = normality of the H_2SO_4 or HCl, and
S = specimen weight, g.

10. Precision and Bias

10.1 Precision and Bias have not been determined.

11. Keywords

11.1 drying oils; fatty acids; saponification value