



Designation: D 1467 – 89 (Reapproved 1995)^{ε1}

Standard Guide for Testing Fatty Acids Used in Protective Coatings¹

This standard is issued under the fixed designation D 1467; 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.

ε¹ NOTE—Keywords were added editorially in May 1995.

1. Scope

1.1 This guide covers the selection and application of procedures for testing fatty acids such as cottonseed, linseed, soybean, dehydrated castor, tall oil, etc. used in protective coatings.

1.2 The test methods included are listed as follows:

Test Method	Section	ASTM Test Method
Acid Value	4	D 1980
Appearance	13	D 1544
Ash	9	D 1951
Clarity	13	D 2090
Color:		
Gardner	14	D 1544
Platinum-Cobalt	14	D 1209
Color Change After Heating	6	D 1981
Fatty Acid Composition	12	D 1983
Hydroxyl Value	10	D 1957
Rosin Acids	11	D 1240
Sampling	3	D 1466
Saponification Value	7	D 1962
Titer	15	D 1982
Unsaponifiable Matter	8	D 1965
Unsaturation:		
Diene Value	5	D 1358
Iodine Value	5	D 1959

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:

- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)²
- D 1240 Test Method for Rosin Acids Content of Naval Stores, Including Rosin, Tall Oil and Related Products³
- D 1358 Test Methods for Spectrophotometric Diene Value

¹ These methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.32 on Drying Oils.

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² Annual Book of ASTM Standards, Vol 06.04.

³ Annual Book of ASTM Standards, Vol 06.03.

of Dehydrated Castor Oil and Its Derivatives³

D 1466 Test Method for Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials³

D 1541 Test Method for Total Iodine Value of Drying Oils and Their Derivatives³

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁴

D 1951 Test Method for Ash in Drying Oils and Fatty Acids³

D 1957 Test Method for Hydroxyl Value of Fatty Oils and Acids³

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids³

D 1962 Test Method for Saponification Value of Drying Oils, Fatty Acids, and Polymerized Fatty Acids³

D 1965 Test Method for Unsaponifiable Matter in Drying Oils, Fatty Acids and Polymerized Fatty Acids³

D 1980 Test Method for Acid Value of Fatty Acids and Polymerized Fatty Acids³

D 1981 Test Method for Measuring Color After Heating of Fatty Acids³

D 1982 Test Method for Titer of Fatty Acids³

D 1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters³

D 2090 Test Method for Clarity and Cleanness of Paint and Ink Liquids³

D 3457 Test Method for Preparation of Methyl Esters from Fatty Acids for Determination of Fatty Acid Composition by Gas-Liquid Chromatography³

2.2 American Oil Chemists' Society (AOCS) Methods:⁵

Tj-1a Polyunsaturated Acids

Cc-13b Color, Wesson Method (Lovibond)

Td-2a Photometric Index

3. Sampling

3.1 Sample the material in accordance with Test Method D 1466.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Available from American Oil Chemists Society, 508 S. 6th St., Champlain, IL 61820.



3.2 Fatty acids are frequently solid at room temperature and shall be completely liquified and blended prior to testing. Temperatures during liquefying of the fatty acids should not exceed 10 to 15°C above the melting point of the sample.

3.3 Samples should be stored preferably in the dark, in full glass containers. Partially filled containers should be filled with inert gas or carbon dioxide to prevent oxidation.

4. Acid Value

4.1 Acid value is a measure of the quantity of free fatty acids and is defined as the number of milligrams of potassium hydroxide required to neutralize the fatty acids in 1 g of sample. Acid value determination is useful in establishing the identity and purity of a fatty acid.

4.2 Determine the acid value in accordance with Test Method D 1980 which is the referee method. Other solvents should be checked out carefully before using for control purposes.

5. Unsaturation

5.1 Iodine value is a convenient test for expressing the degree of unsaturation of a fatty acid. Iodine value is the weight percent of iodine absorbed.

5.2 Determine the iodine value in accordance with Test Method D 1959 which gives fairly good accuracy and precision (Note 1) when applied to normal fatty acids. When Test Method D 1959 is applied to fatty acids containing conjugated double bonds such as dehydrated castor or tung an empirical figure is obtained giving only a relative measure of the total unsaturation. Refer to Test Method D 1541 for a more accurate procedure for the determination of the total unsaturation of tung oil acids. When Test Method D 1959 is used for dehydrated castor acids, a specimen weight of 0.11 to 0.13 g of fatty acids must be used to get comparative results between laboratories.

NOTE 1—The analyst is cautioned to follow the test method exactly as written since small deviations in sample size, reagents, and time of reaction will definitely affect the accuracy and precision of the test method.

5.3 When Test Methods D 1358 is applied to dehydrated castor acids an empirical value is also obtained due to the high linoleic acid content. In this method the conjugated diene content is derived from spectrophotometric measurements. Determine the amount of conjugated diene content of fatty acids in accordance with Test Methods D 1358, noting the empirical nature of the result for dehydrated castor acids.

5.4 The iodine value of tall oil fatty acids containing appreciable amounts of rosin acids is not a reliable measure of the fatty acid unsaturation because of the unsaturation of the rosin acids.

6. Color Change After Heating

6.1 The color stability of fatty acids when subjected to elevated temperatures is of importance to many commercial users of fatty acids. The conditions under which the color stability is determined such as temperature, time of heating, time of upheal, exposure to air, etc, all affect the final results.

6.2 Measure the color of fatty acids after heating by Test Method D 1981, which is applicable to all normal fatty acids.

It has been shown to have good precision within and between laboratories and should be used as the referee test method.

7. Saponification Value

7.1 Saponification value is a measure of the alkali-reactive groups in fatty acids and is expressed as the number of milligrams of potassium hydroxide that will react with 1 g of sample. The saponification value on a good grade of vegetable fatty acids is normally only 1 or 2 points above the acid value as determined in accordance with Test Method D 1980. The difference between the saponification value and the acid value is frequently referred to as the ester value. Saponification value can be used to calculate the average molecular weight of the fatty acid.

7.2 Determine the saponification value in accordance with Test Method D 1962.

8. Unsaponifiable Matter

8.1 The term unsaponifiable matter includes all of those substances that are not saponified by alkali and that are soluble in the ordinary fat solvents. Included are the higher aliphatic alcohols, sterols, and hydrocarbons.

8.2 Determine unsaponifiable matter in accordance with Test Method D 1965. Normal fatty acids contain small amounts of unsaponifiable matter, usually under 2 %. When excessive amounts of unsaponifiable matter are found, low quality is indicated.

9. Ash

9.1 The ash in fatty acids is determined by igniting the acids under specified conditions and determining the amounts of noncombustible material. Carry out this procedure in accordance with Test Method D 1951. The ash content of normal fatty acids should be very small.

10. Hydroxyl Value

10.1 Hydroxyl value is defined as the number of milligrams of potassium hydroxide equivalent to the hydroxyl (OH) content of 1 g of sample. Hydroxystearic acid is the only common fatty acid to which this test is applicable.

10.2 Determine the hydroxyl value in accordance with Test Method D 1957.

11. Rosin Acids

11.1 Fatty acids derived from tall oils will normally contain varying amounts of rosin acids. When the amount of the rosin acids does not exceed 15 %, determine their content in accordance with Test Method D 1240.

11.2 In Test Method D 1240 the rosin acids are determined by titration after esterification of the fatty acids with methanol in the presence of sulfuric acid as a catalyst, followed by separation of the acids. The test method requires use of an experimentally determined correction factor, dependent upon the amount of rosin acids present.

12. Fatty Acid Composition

12.1 Fatty acid composition can be determined in accordance with Test Method D 1983 after esterification of the fatty acids in accordance with Test Method D 1457.

