



Designation: D 555 – 84 (Reapproved 1998)

Standard Guide for Testing Drying Oils¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This guide covers the selection and use of procedures for testing drying oils commonly used in paints, varnishes, and related products.

1.2 The test methods included are as follows:

Test Method	Section	ASTM Test Method
Acetone Tolerance	13	D 1950
Acid Value	6	D 1639
Ash	10	D 1951, D 564
Break	12	D 1952
Clarity	5	D 2090
Color	19	D 1544, D 1209
Color after Heating of Drying Oils	25	D 1967
Drying Properties	23	D 1640
Flash Point	24	D 93, D 1310, D 56
Foots Volumetric	11	D 1954
Foots Gravimetric	11	D 1966
Gel Time	14	D 1955
Hydroxyl Value	16	D 1957
Loss on Heating	17	D 1960, D 93
Matter Insoluble in Chloroform	18	D 1958
Preparation of Sample	4	...
Refractive Index	21	...
Sampling	3	D 1466
Saponification Value	8	D 1962
Specific Gravity	20	D 1963, D 1475
Tung Oil Quality Test	15	D 1964
Unsaponifiable Matter	9	D 1965
Unsaturation:		
Diene Value:		
Spectrophotometric Method	7	D 1358
Iodine Value:		
Rosenmund-Kuhn-henn Method	7	D 1541
Wijs Method	7	D 1959
Viscosity	22	D 1545, D 445

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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- D 56 Test Method for Flash Point by Tag Closed Tester²
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester²
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 564 Test Methods for Liquid Paint Driers³
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)⁴
- D 1259 Test Method for Nonvolatile Content of Resin Solutions³
- D 1310 Test Method for Flash Point and Fire Points of Liquids by Tag Open-Cup Apparatus³
- D 1358 Test Method for Spectrophotometric Diene Value of Dehydrated Castor Oil and Its Derivatives⁵
- D 1466 Test Method for Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials⁵
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products³
- D 1541 Test Method for Total Iodine Value of Drying Oils and Their Derivatives⁵
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)³
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble-Time Method⁵
- D 1639 Test Method for Acid Value of Organic Coating Materials⁵

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.04.

⁵ Annual Book of ASTM Standards, Vol 06.03.



- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature³
- D 1644 Test Methods for Nonvolatile Content of Varnishes³
- D 1950 Test Method for Acetone Tolerance of Heat-Bodied Drying Oils⁵
- D 1951 Test Method for Ash in Drying Oils and Fatty Acids⁵
- D 1952 Test Method for Quantitative Determination of Break in Drying Oils⁵
- D 1954 Test Method for Foots in Raw Linseed Oil (Volumetric Method)⁵
- D 1955 Test Method for Gel Time of Drying Oils⁵
- D 1957 Test Method for Hydroxyl Value of Fatty Oils and Acids⁵
- D 1958 Test Method for Chloroform-Insoluble Matter in Oiticica Oil⁵
- D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids⁵
- D 1960 Test Method for Loss on Heating of Drying Oils⁵
- D 1962 Test Method for Saponification Value of Drying Oils, Fatty Acids and Polymerized Fatty Acids⁵
- D 1963 Test Method for Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25°C⁵
- D 1964 Test Method for Tung Oil Quality⁵
- D 1965 Test Method for Unsaponifiable Matter in Drying Oils, Fatty Acids and Polymerized Fatty Acids⁵
- D 1966 Test Method for Foots in Raw Linseed Oil (Gravimetric Method)⁵
- D 1967 Test Method for Measuring Color After Heating of Drying Oils⁵
- D 1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters⁵
- D 2090 Test Method for Clarity and Cleanness of Paint and Ink Liquids⁵
- D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints³
- D 2800 Test Method for Preparation of Methyl Esters from Oils for Determination of Fatty Acid Composition by Gas Chromatography⁵
- D 3457 Test Method for Preparation of Methyl Esters from Fatty Acids for Determination of Fatty Acid Composition by Gas-Liquid Chromatography⁵
- D 3725 Test Method for Semiquantitative Determination of Fish Oil in Drying Oils and Drying Oil Fatty Acids by Gas-Liquid Chromatography⁵

3. Sampling

3.1 Sample the material in accordance with Test Method D 1466. This test method covers in considerable detail a procedure for obtaining representative samples of liquid oils and fatty materials from drums, barrels, casks, and tank cars. The test method gives instructions on obtaining representative samples from 4000, 6000, 8000, 10 000 and 12 000-gal (15, 23, 30, 38, and 45-m³) cars. Additional directions must be obtained for sampling cars of other capacities.

3.2 Test Method D 1466 takes into consideration the possible presence of settled solid or “footy” materials that may exist in the container. The test method requires that drums or casks be thoroughly mixed by rolling before a sample is taken.

However, with regard to tank cars, the procedure, if followed carefully, will yield representative samples from cars that contain considerable quantities of settled solid material. If it is known that there is no settled material in a tank car, any one of a number of established liquid samplers may be used with good results.

4. Preparation of Sample

4.1 Melt the sample if it is not already completely liquid. The temperature during melting should not exceed 10 to 15°C above the melting point of the sample.

4.2 Mix the laboratory sample thoroughly by shaking, stirring, or pouring from one vessel to another. Take the specimens for the individual tests from this thoroughly mixed sample.

5. Clarity

5.1 This requirement provides for the quick rejection of natural oils that are obviously contaminated by solid matter, such as dirt, or water in excess of the solubility limit.

5.2 Most natural oils contain some saturated glycerides, which may crystallize out at low temperatures giving a cloudy appearance. If the cloudiness disappears on warming, it is probably due to these saturated glycerides and should be disregarded.

5.3 Some processed oils are naturally hazy, as a result of the processing methods used, and a clarity requirement should not be included in specifications for processed oils unless it is known that properly processed oils of the type desired will meet the requirements.

5.4 Determine the clarity in accordance with Test Method D 2090.

6. Acid Value

6.1 The acid value of an oil is an indication of the condition of the seed from which the oil has been extracted and of the refining to which it has been subjected. It is not useful for the identification of the type of oil.

6.2 Test Method D 1639 is generally most satisfactory as to precision. There is no choice between sodium and potassium hydroxides except personal preference.

6.3 If the percent of free fatty acids calculated as oleic is required, the following equation may be used for the transformation:

$$\text{Free fatty acids, \%} = 0.503 \times \text{acid value} \quad (1)$$

7. Unsaturation

7.1 The drying properties of fats and oils are indicated by the amount and nature of unsaturation they contain. The amount is conventionally expressed as the iodine value, that is, centigrams of iodine absorbed per gram of sample (weight percent of iodine absorbed). The iodine value is a fairly satisfactory measure of the relative drying time and speed of heat-polymerization among a group of oils of the same type. However, because both drying time and heat-polymerization are affected by the kind and distribution of fatty acids in the oil, these methods are not so useful in comparing oils of different types. The measurement of unsaturation is an auxiliary method.



determination of the individual fatty acids for the identification of natural oils, since each natural oil has its own range of unsaturation values.

7.2 Determine the unsaturation of natural drying oils that do not contain conjugated double bonds by the Wijs method as described in Test Method D 1959, which gives fairly good accuracy and precision. It has largely superseded the Hanus and other methods that tend to give high results. When the Wijs method is applied to oils containing conjugated double bonds, such as tung oil and dehydrated castor oil, an empirical figure is obtained that is indicative of the relative amount of unsaturation present, but is not a measure of the total unsaturation. With careful control of the reaction conditions, however, reproducible and useful results may be obtained. Where the total unsaturation is required, make the determination using a modification of the Rosenmund-Kuhnhehn method as described in Test Method D 1541. This method gives an accurate measure of total unsaturation on conjugated oils and is also satisfactory for nonconjugated oil, although somewhat more difficult to run than the Wijs method. Quantitative hydrogenation will also yield an accurate measure of the total unsaturation. There is no standard procedure for this method. It is the only satisfactory method for oils containing acetylenic bonds, such as isano oil.

7.3 The iodine value is useful for the identification of linseed, soybean, safflower, and similar natural oils. The amount of conjugated diene (or triene) is useful for identifying tung and oiticica oils, as well as dehydrated castor oil. Conjugated diene is a measure of the quality of dehydrated castor oil, although it is not the only measure that should be used. Determine the amount of conjugated diene by spectrophotometric measurement using Test Methods D 1358.

8. Saponification Value

8.1 The saponification value is, essentially, a measure of the molecular weight of the fatty acid portion of the glyceride, varying inversely with the weight, except for certain modified oils. It is not a measure of the quality or identity of the oil. The value is useful for certain calculations in the use of the oil, such as in the manufacture of alkyd resins.

8.2 Determine the saponification value in accordance with Test Method D 1962, which is satisfactory for all normal oils and for many special and synthetic products. As indicated in the test method, longer saponification times are required for certain synthetic oils, and some special products may require the use of a higher-boiling solvent, such as ethylene glycol, for complete saponification.

8.3 Saponification value is not changed significantly by polymerization but increases rapidly with oxidation. A saponification value significantly higher than normal indicates the presence of oxidized or blown oils or else modification with chemicals such as maleic or fumaric acids.

9. Unsaponifiable Matter

9.1 The unsaponifiable matter is a measure of the materials present in the oil that are oil-soluble and are not converted to water-soluble soaps by the saponification conditions used. A small amount of unsaponifiable matter is characteristic of all natural oils, varying with the extraction and refining condi-

tions. Within the limits of the individual oil specifications, the amount of unsaponifiable matter is no measure of the quality or identity of the oil. An excessive amount of unsaponifiable matter indicates contamination with nonglyceride matter, such as mineral oil, hydrocarbon resins, etc.

9.2 Determine the unsaponifiable matter in accordance with Test Method D 1965, which is the referee method. Since the exact amount of unsaponifiable matter obtained is governed by the partition coefficient of the matter between the soap solution and the solvent, different results may be expected if some other solvent such as ethyl ether is used. A rapid, qualitative test for excessive unsaponifiable matter consists in saponifying a small quantity of the oil and diluting with water. A milky emulsion indicates excess unsaponifiable matter.

10. Ash

10.1 Ash is determined by igniting the oil, under specified conditions, and weighing the noncombustible material. Most natural and processed oils contain small amounts of ash, but the amount is insignificant. Certain, synthetic drying oils may contain residual catalyst or other materials, thus giving larger amounts of ash. Although the ash and metal content of boiled oils may be specified, the trend with new and improved driers is toward the specification of drying time, allowing the manufacturer to obtain this in any way desired.

10.2 Determine the ash in accordance with Test Method D 1951. Determine the drier metal content of the ash in accordance with Test Methods D 564. Wet ashing or extraction methods may give more accurate results.

11. Foots

11.1 "Foots" is the term applied to nonoil material that will settle out of natural oils on storage. Since the material measured as foots is usually suspended rather than dissolved in the oil, extreme care in sampling is necessary to get a representative sample for measurement. This applies not only to the sampling of the bulk oil but also to the taking of specimens for running the test. Careful agitation to ensure thorough mixing before sampling is absolutely essential.

11.2 Some of the materials included in the foots hydrate readily in the presence of moisture, particularly at low temperatures, and these hydrated materials have a much larger volume than they had originally. Therefore, oils exposed to moisture for periods of time, particularly with chilling, may be expected to show a substantial increase in volumetric foots with time.

11.3 Determine volumetric foots in accordance with Test Method D 1954. Very careful control of all variables in the test is absolutely necessary, as the test is empirical. Even under the best conditions, the reproducibility is not good. The test is usually used for raw linseed oil. It has no meaning when applied to oils that have been processed to any marked degree.

11.4 Determine gravimetric foots in accordance with Test Method D 1966. The precision of this procedure is much better than the older volumetric method. Internationally the procedure is known as the P.A.T. test and its use is increasing in oil trading.

