

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

# Standard Test Method for Determination of Iodine Value of Tall Oil Fatty Acids<sup>1</sup>

This standard is issued under the fixed designation D 5768; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

#### 1. Scope

- 1.1 This test method covers the Wijs procedure for determination of unsaturation (iodine value) of tall oil fatty acids.
- 1.2 Iodine value is a measure of the unsaturation of oils and fatty acids and is expressed in terms of the number of centigrams of iodine per gram of sample (weight percent of absorbed iodine).
- 1.3 When this test method is used to determine the iodine value of fatty acids having conjugated systems, the result is not a measure of total unsaturated, but rather is an empirical value that affords a comparison of unsaturation. Total unsaturation of conjugated systems may be measured in accordance with Test Method D 1541.
- 1.4 The test method described here is not reliable for tall oil fatty acids containing an appreciable quantity of rosin.
- 1.5 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 1193 Specification for Reagent Water<sup>2</sup>
- D 1541 Test Method for Total Iodine Value of Drying Oils and Their Derivatives<sup>3</sup>
- D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids<sup>3</sup>

## 3. Significance and Use

- 3.1 The iodine value of a fatty acid product is a measure of the unsaturated fatty acid content of that product and consequently a measure of the ease of oxidation or drying capacity of that fatty acid product.
- 3.2 This test method measures the unsaturation as iodine value by addition of an iodine/chlorine reagent. The amount of reagent absorbed is determined by back titrating the excess reagent and comparing it to a blank determination.
  - 3.3 In samples containing conjugated double bonds, the

iodine value obtained is empirical since the reagent does not react stoichiometrically with conjugated unsaturation. Where no conjugation is present, the iodine value obtained is a measure of the total unsaturation. By using proper specimen weights, the empirical values obtained are useful for comparative purposes.

- 3.4 This test method was developed in order to replace the hazardous solvent, carbon tetrachloride, used in Test Method D 1959 with the less hazardous and more available solvent, isooctane. Other solvents, such as cyclohexane, may also be satisfactory replacements for carbon tetrachloride. As data on the satisfactory use of other solvents becomes available, this test method will be amended to include those solvents.
- 3.5 This test method should have applicability to fatty acids and oils other than tall oil fatty acid but that possibility has not been investigated.

## 4. Apparatus

- 4.1 *Bottles*—Glass-stoppered bottles or Erlenmeyer flasks of 250-mL capacity.
  - 4.2 Pipets—20 and 25-mL capacity.

#### 5. Reagents

- 5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests unless otherwise specified. 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.<sup>4</sup> 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.
- 5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I of Specification D 1193.
- 5.3 Acetic Acid (Glacial) 17.4 M—Verify the absence of substances reducing permanganate as follows: Dilute 2 mL of the acid with 10 mL of water and add 0.1 mL of 0.1 N potassium permanganate (KMnO<sub>4</sub>) solution. The pink color

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Naval Stores.

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 06.03.

<sup>&</sup>lt;sup>4</sup> 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.