Designation: ~~D5768 – 02 (Reapproved 2014)~~ D5768 – 02 (Reapproved 2018)

Standard Test Method for Determination of Iodine Value of Tall Oil Fatty Acids[1]

This standard is issued under the fixed designation D5768; 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 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 D1541.

1.4 The test method described here is not reliable for tall oil fatty acids containing an appreciable quantity of rosin.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *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. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1541 Test Method for Total Iodine Value of Drying Oils and Their Derivatives (Withdrawn 2006)[3]

D1959 Test Method for Iodine Value of Drying Oils and Fatty Acids (Withdrawn 2006)[3]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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 D1959 with the less hazardous and more available solvents, iso-octane and cyclohexane. As data on the satisfactory use of other solvents becomes available, this test method will be amended to include those solvents.

---

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Pine Chemicals and Hydrocarbon Resins.

Current edition approved ~~Dec. 1, 2014~~June 1, 2018. Published ~~December 2014~~June 2018. Originally approved in 1995. Last previous edition approved in ~~2010~~2014 as D5768 – 02 ~~(2010).~~(2014). DOI: ~~10.1520/D5768-02R14.~~10.1520/D5768-02R18.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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.

4.3 Analytical balance

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.[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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I of Specification D1193.

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 ($KMnO_4$) solution. The pink color should not be entirely discharged at the end of 2 h.[5]

5.4 *Iso-octane or cyclohexane.*

5.5 *Chlorine (99.8 % Cl)*—(~~**Warning:**~~**Warning** ~~Extremely~~ —Extremely hazardous. For specific hazard information and guidance, see supplier's Material Safety Data sheets.) Commercial grades of chlorine available in cylinders may be used, provided the gas is dried by passing through concentrated sulfuric acid (H_2SO_4, sp gr 1.84) before passing it into the iodine solution. Alternatively, the chlorine may be prepared by allowing concentrated hydrochloric acid (HCl, sp gr 1.19) to drop onto potassium permanganate ($KMnO_4$) or onto a mixture of $KMnO_4$ and manganese dioxide (MnO_2). Dry the gas thus generated by passing it through concentrated H_2SO_4.

5.6 *Potassium Iodide Solution (150 g/L)*—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 L.

5.7 *Sodium Thiosulfate, Standard Solution (0.1 N)*—Dissolve 24.8 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in water and dilute to 1 L. Standardize against potassium dichromate ($K_2Cr_2O_7$)[6] as follows: Weigh to 0.1 mg, by difference from a weighing bottle, 0.16 to 0.22 g of $K_2Cr_2O_7$ that has been finely ground and then dried to constant weight at 105 to 110°C prior to use. Place the $K_2Cr_2O_7$ in a 500-mL flask or bottle and dissolve in 25 mL of water. Add 5 mL of concentrated hydrochloric acid (11.6 *M*) and 20 mL of KI solution, and rotate to mix. Allow to stand for 5 min and then add 100 mL of water. Titrate with the $Na_2S_2O_3$ solution, while shaking constantly, until the yellow color has almost disappeared. Add 1 to 2 mL of starch indicator solution and continue the titration, adding the $Na_2S_2O_3$ solution slowly until the blue color has just disappeared. Calculate the normality, *N*, of the $Na_2S_2O_3$ as follows:

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] "Analytical Reagents, ACS Specifications," American Chemical Society, Washington, DC, 1960.

[6] National Institute of Standards and Technology Standard Reference Material No. 136 of potassium dichromate is recommended for this purpose and should be treated as directed in the certificate of analysis accompanying the standard sample. Available from NIST, Gaithersburg, MD.