Designation: D1585 – 15 (Reapproved 2020)

Standard Test Methods for Fatty Acids Content of Pine Chemicals, Including Rosin, Tall Oil, and Related Products[1]

This standard is issued under the fixed designation D1585; 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 These test methods cover the determination of the fatty acids of pine chemicals, including rosin, tall oil, and related products.

1.2 These test methods may not be applicable to adducts or derivatives of rosin or other pine chemical products.

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

1.4 *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]

D465 Test Methods for Acid Number of Pine Chemical Products Including Tall Oil and Other Related Products

D803 Test Methods for Testing Tall Oil

D890 Test Method for Water in Liquid Pine Chemicals

D1065 Test Method for Unsaponifiable Matter in Pine Chemicals, Including Rosin, Tall Oil, and Related Products

D1240 Test Methods for Rosin Acids Content of Pine Chemicals, Including Rosin, Tall Oil, and Related Products

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. Summary of Test Method

3.1 The rosin acids content is determined using either the modified potentiometric Wolfe Method or the modified indicator Wolfe Method described in Test Methods D1240. Rosin acids are calculated as abietic acid.

3.2 The acid number is determined by either the potentiometric or the indicator method in accordance with Test Methods D465.

3.3 The unsaponifiable matter is determined in accordance with the methods described in Test Method D1065.

3.4 The fatty acids are calculated by two methods.

3.4.1 For materials with a fatty acid content less than 5 %, fatty acid content is calculated from the rosin acids content and the acid number.

3.4.2 For materials with a fatty acid content greater than 5 %, fatty acid content is calculated from the rosin acids and unsaponifiables content.

3.5 The same method for end point detection, either potentiometric or indicator, should be used for acid number, unsaponifiables, and rosin acids content determination, in order to avoid slight variables that might occur.

3.6 Since the fatty acids remaining in tall oil rosin, tall oil, and other pine chemical products consist of oleic acid with varying amounts of other saturated and unsaturated acids, it has become customary to calculate and report the fatty acid content as oleic acid.

4. Significance and Use

4.1 These test methods are designed to broaden the scope of the previous edition of these test methods by the inclusion of tall oil as a test material. Test Methods D803 currently includes methods for the determination of the rosin acid and fatty acid content of crude tall oil. Test Methods D803 references Test Method D1585.

4.2 Rosin and tall oil are composed primarily of rosin acids and fatty acids, and the measurement of these components is important in establishing the composition of these materials.

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

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[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.

5. Purity of Reagents

5.1 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.[3] 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 Unless otherwise indicated, references to water shall be understood to mean deionized or distilled water.

6. Preparation of Sample

6.1 Homogeneous liquid materials may be used without further preparation.

6.2 Heat nonhomogeneous liquid materials until they are homogeneous, then take a portion for analysis.

6.3 Solid samples are subject to surface oxidation which may affect the results. Prepare the sample for analysis by chipping small pieces from a freshly exposed surface of a lump or lumps and crush to a coarse powder to facilitate weighing and solution. Prepare fresh on the same day, prior to weighing, in order to avoid changes due to surface oxidation of crushed rosin on exposure to the air.

PERCENT ROSIN ACID

7. Procedure

7.1 Determine the percent rosin acid of the sample by the potentiometric or the indicator modified Wolfe Method in accordance with Test Methods D1240.

ACID NUMBER

8. Procedure

8.1 Determine the acid number by either the potentiometric or the indicator modification in accordance with Test Methods D465.

UNSAPONIFIABLE MATTER

9. Procedure

9.1 Determine the percentage of unsaponifiable matter by either the potentiometric or the indicator modification in accordance with Test Method D1065.

WATER

10. Procedure

10.1 Determine the percent water in the sample by the Karl-Fischer Method in accordance with Test Method D890.

FATTY ACIDS

11. Calculation

11.1 For materials containing less than 5 % fatty acid, calculate the percentage of fatty acids from the acid number and the percentage of rosin acids as follows:

$$\text{Fatty acids as oleic acid, \%} = [A - (R \times 1.855)]/1.986 \quad (1)$$

where:

A = acid number of original sample,
R = rosin acids, %,
1.855 = (56.1 × 1000)/(302.4 × 100) (factor to convert the percentage of rosin acids to acid number), and
1.986 = (56.1 × 1000)/(282.4 × 100) (factor to convert acid number to percentage of oleic acid).

11.2 For materials containing equal to or greater than 5 % fatty acid, calculate the percent fatty acid from the percent rosin acids and unsaponifiables as follows:

$$\text{Fatty Acids, \%} = 100 - R - U - W \quad (2)$$

where:

R = rosin acids, %,
U = unsaponifiable matter, %,
W = water, %.

11.3 Report the percentage of fatty acids determined in 11.1 or 11.2 to one decimal place.

12. Precision and Bias[4]

12.1 *Interlaboratory Test Program*—An interlaboratory study of the fatty acid content of three substances, tall oil fatty acids, distilled tall oil, and rosin, was run in 1994. Each of 14 laboratories tested each of the three materials. The design of the experiment, similar to that of Practice E691 and a within-between analysis of the data are given in ASTM Research Report RR:D01-1088.

12.2 *Test Result*—The precision information given below for the fatty acid content of pine chemical products is for the comparison of two test results, each of which is the average of three test determinations as follows:

12.2.1 *Repeatability Limit,* 95 % (within laboratory) = 0.6 %.

12.2.2 *Reproducibility Limit,* 95 % (between laboratories) = 1.9 %.

12.3 These terms (repeatability limit and reproducibility limit) are used as specified in Practice E177. The respective standard deviations among test results, related to the above numbers by the factor of 2.8, are as follows:

12.3.1 Repeatability standard deviation = 0.2 %.

12.3.2 Reproducibility standard deviation = 0.7 %.

12.4 *Bias*—These test methods have no bias because fatty acid content is defined only in terms of these test methods.

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1088. Contact ASTM Customer Service at service@astm.org.