Designation: D803 – 15

Standard Test Methods for Testing Tall Oil[1]

This standard is issued under the fixed designation D803; 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 test procedures to be applied to whole tall oils or refined tall oils. Previous editions of these test methods have described test procedures that are used to test tall oil fatty acid, rosin, and other tall oil-derived products as well as test crude and refined tall oil. Consequently, these test methods are widely cited in reference books and industry literature for the testing of tall oil-derived products.

1.1.1 In this current revision, procedural details of some of the often-cited test methods have been removed and the test methods consolidated with other existing test methods. In such cases the consolidated methods, applicable to all tall oil-derived products.

1.2 The procedures appear in the following order:

Physical Tests	Sections
Viscosity:	
Rotational Viscometer Method (Preferred Method)	7
Bubble Time Method	8
Flash Point	9
Color	10
Moisture:	11
Insoluble Matter	12
Ash	13
Chemical Analysis	
Acid Number	16
Saponification Number	17
Rosin Acids	18
Unsaponifiable Matter	19
Fatty Acids	20

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *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:*[2]

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D269 Test Method for Insoluble Matter in Rosin and Rosin Derivatives

D464 Test Methods for Saponification Number of Naval Store Products Including Tall Oil and Other Related Products

D465 Test Methods for Acid Number of Naval Stores Products Including Tall Oil and Other Related Products

D890 Test Method for Water in Liquid Pine Chemicals

D1065 Test Method for Unsaponifiable Matter in Naval Stores, Including Rosin, Tall Oil, and Related Products

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

D1466 Test Method for Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials (Withdrawn 2003)[3]

D1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)

D1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method

D1585 Test Methods for Fatty Acids Content of Naval Stores, Including Rosin, Tall Oil, and Related Products

D2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer

D3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus

D5974 Test Methods for Fatty and Rosin Acids in Tall Oil Fractionation Products by Capillary Gas Chromatography

D6166 Test Method for Color of Pine Chemicals and Related Products (Instrumental Determination of Gardner Color)

[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.

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

E300 Practice for Sampling Industrial Chemicals

3. Significance and Use

3.1 Tall oil, both crude and refined, is an important by-product of the alkaline (kraft) pulping of pine wood. It consists primarily of fatty acids, resin acids, and neutral materials; the levels of these various components depend on factors such as the species of pine tree, geographic location, climate, etc.

3.1.1 Tall oil is used primarily as a commercial source of fatty acids and rosin, and, therefore, reliable methods for the analysis of these components is necessary.

4. Purity of Reagents

4.1 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.[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.

4.2 Unless otherwise indicated, references to water shall be understood to mean distilled water or deionized water.

5. Sampling

5.1 As with many industrial chemicals, the procedure for obtaining a representative sample of tall oil depends on the container to be sampled and the uniformity of the material to be sampled. No specific standard procedure for sampling tall oil has been developed but Test Method D1466 and Practice E300 describe applicable methods when the tall oil contains no separated solids or separated water. If rosin has separated, the rosin can be redissolved by heating and agitating the material to be sampled until it is homogeneous. If separated water is present, special procedures should be followed to ensure that a representative sample be obtained. The selection of an appropriate procedure will depend on the water content and the type of container being sampled.

6. Conditioning

6.1 Heat the entire sample in a closed container fitted with a capillary vent or its equivalent. Some kind of agitation, even if done occasionally by hand, saves much time. Heat in a forced-air oven in order to dissolve any crystalline material. A temperature of 100°C is usually sufficient to dissolve crystals present in crude tall oil, but a temperature as high as 160°C may be required to remove rosin crystals from distilled tall oil. Withdraw the specimens only when all crystalline matter has dissolved and when the entire sample becomes a homogeneous fluid after thorough stirring.

[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.

VISCOSITY BY THE ROTATIONAL VISCOMETER METHOD

(*Preferred Method*)

7. Procedure

7.1 Determine the viscosity in centipoise in accordance with Test Methods D2196. The testing temperature shall be 210°F (99°C) unless a lower temperature is specified.

VISCOSITY BY THE BUBBLE TIME METHOD

(*Alternative Method*)

8. Procedure

8.1 Determine the viscosity in accordance with Test Method D1545. The testing temperature shall be 210°F (99°C) unless a lower temperature is specified.

FLASH POINT

9. Procedure

9.1 Determine the flash point in accordance with any referenced method (see Test Methods D92, D93, and D3278).

COLOR

10. Procedure

10.1 Determine the color in accordance with Test Methods D1544 or D6166.

MOISTURE CONTENT

11. Procedure

11.1 Determine the moisture content in accordance with the methods described in Test Method D890. The Karl Fischer method is the preferred method.

INSOLUBLE MATTER

12. Procedure

12.1 Determine the insoluble matter in accordance with Test Method D269.

ASH

13. Apparatus

13.1 *Platinum Dish*—A platinum dish of 50 to 100-mL capacity.

NOTE 1—A porcelain or silica dish may be used in place of platinum, if the ash is not to be analyzed.

14. Procedure

14.1 Heat the platinum dish to redness and, after cooling in a desiccator, weigh to the nearest 0.0001 g. Place an approximately 20-g sample of tall oil in the dish and weigh to the nearest 0.1 g (Note 2). Heat the dish gently by means of a bunsen burner until the oil can be ignited at the surface (Note 3). Remove the burner and allow the oil to burn completely. Burn all free carbon on the sides of the dish and heat the

residue with a strong flame, or in a muffle furnace, until all carbonaceous matter disappears. After cooling in a desiccator weigh the dish to the nearest 0.0001 g. Repeat the heating until a constant weight is obtained.

NOTE 2—This size is suitable for ash contents in the range from 0.02 to 0.2 %, but in the case of lower or higher ash contents, a larger or smaller sample may be used. In such cases the result shall be specified or reported as follows: "Ash (...g sample) ... percent."

NOTE 3—To avoid foaming and loss of sample in the case of samples containing moisture, it is advisable to add 1 to 2 mL of absolute alcohol before heating.

15. Calculation

15.1 Calculate the percentage of ash as follows:

$$\text{Ash. } \% = (R/W) \times 100 \quad (1)$$

where:

R = residue, g, and
W = sample, dry basis, g.

15.2 Report the percentage of ash to the third decimal place.

CHEMICAL ANALYSIS

16. Acid Number

16.1 Determine the acid number in accordance with Test Methods D465.

17. Saponification Number

17.1 Determine the saponification number in accordance with Test Methods D464.

18. Rosin Acid Content

18.1 Determine the rosin acid content in accordance with Test Methods D1240.

19. Unsaponifiable Matter

19.1 Determine the unsaponifiable matter in accordance with Test Method D1065.

20. Fatty Acid Content

20.1 Determine the fatty acid content in accordance with Test Methods D1585.

21. Gas Chromatographic Analysis

21.1 Analyze for rosin acids and fatty acids in accordance with Test Methods D5974.

22. Precision and Bias (General Statement)

22.1 Where the test method listed in this standard references another ASTM method, that method should be studied to determine the precision and bias. Other methods listed are well established, and determination of precision and bias is not practical.

23. Keywords

23.1 acid number; fatty acids; rosin acids; saponification number; tall oil