



Designation: ~~D803-02~~ Designation: D 803 – 03 (Reapproved 2008)

## Standard Test Methods for Testing Tall Oil<sup>1</sup>

This standard is issued under the fixed designation D 803; 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 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, are referenced.

1.2 The procedures appear in the following order:

Physical Tests	Sections
Viscosity:	
Brookfield Method (Preferred Method)	7
Bubble Time Method	8
<del>Pour Point</del>	<del>9</del>
Flash Point	9
<del>Flash Point</del>	<del>9</del>
Color	Color
Moisture:	Moisture:
<del>Insoluble Matter</del>	<del>Insoluble Matter</del>
Ash <sup>14</sup>	13
Ash	13
	Chemical Analysis
Acid-Number <sup>17</sup>	16
Acid Number	16
<del>Saponification Number<sup>18</sup></del>	<del>17</del>
Saponification Number	17
<del>Resin Acids<sup>19</sup></del>	<del>18</del>
Rosin Acids	18
<del>Unsaponifiable Matter<sup>20</sup></del>	<del>19</del>
Unsaponifiable Matter	19
<del>Fatty Acids<sup>21</sup></del>	<del>20</del>
Fatty Acids	20

### 1.3

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:<sup>2</sup>

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

~~D 97 Test Method for Pour Point of Petroleum Products<sup>2</sup>~~

D 269 Test Method for Insoluble Matter in Rosin and Rosin Derivatives

<sup>1</sup> 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 Naval Stores.

Current edition approved May 10, 2002. Published July 2002. Originally published as D803-44T. Last previous edition D803-97.

Current edition approved June 1, 2008. Published June 2008. Originally approved in 1944. Last previous edition approved in 2003 as D 803 – 03.

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

- D 464 Test Methods for Saponification Number of Naval Store Products Including Tall Oil and Other Related Products
- D 465 Test Methods for Acid Number of Naval Stores Products Including Tall Oil and Other Related Products
- D 890 Test Method for Water in Liquid Naval Stores
- D 1065 Test Method for Unsaponifiable Matter in Naval Stores, Including Rosin, Tall Oil, and Related Products
- D 1240 Test Methods for Rosin Acids Content of Naval Stores, Including Rosin, Tall Oil, and Related Products
- D 1466 Test Method for Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method
- D 1585 Test Methods for Fatty Acids Content of Naval Stores, Including Rosin, Tall Oil, and Related Products
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer
- D 3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus
- D 5974 Test Methods for Fatty and Rosin Acids in Tall Oil Fractionation Products By Capillary Gas Chromatography
- D 6166 Test Method for Color of Naval Stores and Related Products (Instrumental Determination of Gardner Color)
- E 300 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.<sup>3</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.

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 D 1466 and Practice E 300 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.

#### VISCOSITY BY THE BROOKFIELD METHOD

*(Preferred Method)*

### 7. Procedure

7.1 Determine the viscosity in centipoise in accordance with Test Methods D 2196. 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 D 1545. The testing temperature shall be 210°F (99°C) unless a lower temperature is specified.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 06.03: 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.