Designation: D 1065 – 96

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Unsaponifiable Matter in Naval Stores, Including Rosin, Tall Oil, and Related Products[1]

This standard is issued under the fixed designation D 1065; 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 determination of the percentage of material in Naval Stores products as defined in Terminology D 804[2] including rosin, tall oil and related products, other than insoluble dirt or similar visible foreign matter that does not yield a water-soluble soap when the sample is saponified with potassium hydroxide.

1.2 *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 509 Test Methods of Sampling and Grading Rosin[2]
D 803 Test Methods of Testing Tall Oil[2]
D 804 Terminology Relating to Naval Stores and Related Products[2]
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Significance and Use

3.1 This test method is designed to broaden the scope of the previous edition of the test method by the inclusion of tall oil and tall oil derived from products as test materials. Test Methods, D803 currently includes a method for the determination of unsaponifiable matter.

3.2 The amount of unsaponifiable matter in tall oil and other related products is important in characterizing such products as it indicates the level of nonacidic material, both free and combined, present in the test material. The unsaponifiable in naval stores products is primarily composed of higher molecular weight alcohols, sterols, and hydrocarbons.

4. Apparatus

4.1 *Erlenmeyer or Other Flat-Bottom Flask*, of 125-mL to 250-mL capacity, with standard-taper 24/40 joint.

4.2 *Erlenmeyer Flask*, of 250-mL to 300-mL capacity, with wide mouth.

4.3 *Separatory Funnels*, of 300-mL to 500-mL capacity, with glass or polytetrafluoroethylene (PFTE) stoppers.

4.4 *Graduated Cylinder*, one of 10 to 25-mL and one of 50 to 100-mL capacity.

4.5 *Beaker*, of up to 250-mL capacity.

5. Reagents

5.1 *Purity of Reagents*—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 use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled, or deionized water.

6. Preparation of Sample

6.1 Procurement and handling of samples will vary depending upon the physical state of the material. In all instances, the sampling should conform to accepted sampling techniques which ensure the sample is representative of the material being sampled.

6.2 Uniform liquid material should be mixed well and an aliquot removed for analysis. Titer in fatty acid samples should be resolubilized by gentle heating and agitation. Rosin crystallization in liquid samples, such as distilled tall oil (DTO), should be resolubilized by heating to 160°C with periodic agitation. Homogeneous representative samples are imperative.

6.3 Solids that melt at relatively low temperature (that is,

[1] 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.

Current edition approved Dec. 10, 1996. Published February 1997. Originally published as D 1065 – 49 T. Last previous edition D 1065 – 92.

[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[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.