



# Standard Test Methods for Acid Number of Naval Stores Products Including Tall Oil and Other Related Products<sup>1</sup>

This standard is issued under the fixed designation D 465; 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 are intended for determining the acid number of naval store products as defined in Terminology D 804<sup>2</sup> including tall oil products, wood and gum rosin, and other related materials. These test methods may not be applicable to all modified rosin products. Two test methods are covered, as follows:

1.1.1 Potentiometric method (referee), and

1.1.2 Internal indicator method (alternate).

1.2 The potentiometric method is suitable for use with both light- and dark-colored products. It should be considered the referee method. The internal indicator method is suitable for use only with light- and medium-colored products with a Gardner color of less than 12. It should be considered the alternate method.

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

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 803 Test Methods of Testing Tall Oil<sup>2</sup>

D 804 Terminology Relating to Naval Stores, Including Tall Oil and Other Related Products<sup>2</sup>

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode<sup>3</sup>

E 177 Practice for the Use of the Terms Precision and Bias in ASTM Test Methods<sup>4</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

## 3. Significance and Use

3.1 These test methods are designed to broaden the scope of the previous edition of the test method by the inclusion of tall

oil and tall oil derived products as test materials. Test Methods D 803 currently includes a method for the determination of acid number. The details of that procedure will be deleted when Test Methods D 803 are revised.

3.2 The acid number is an important property of naval stores products, tall oil, and the products obtained by the fractionation of tall oil. It is the test method widely used to determine the total free acid content of these products.

3.3 The potentiometric test method should be used when the most reproducible results are required.

## 4. Preparation of Sample

4.1 If the sample for analysis is rosin, it shall consist of small pieces of rosin chipped from a freshly exposed part of a lump or lumps, and thereafter crushed to facilitate weighing and dissolution. Prepare the sample the same day on which the test is begun, in order to avoid changes in properties due to surface oxidation. This is very pronounced on ground rosin having a large surface area exposed to the air. Existing rosin dust and powdered rosin must not be used.

4.2 If the sample is a nonhomogenous liquid, heat the entire sample in a closed container fitted with a capillary vent or the equivalent. Some kind of agitation, even if done occasionally by hand, saves much time. Heat by immersion in open steam or hot water bath to avoid overheating. When dealing with crystallized rosin a temperature of approximately 160°C may be needed. Sampling should take place only when the entire sample is homogeneous and has been well stirred.

4.3 For other products no special preparation is necessary except storage in a closed container prior to testing.

4.4 Guideline for sample size, solvent, and titrant:

Sample	Sample Size, g	Solvent I, mL	Solvent II, mL	Titrant, KOH
Fatty acids DTO <sup>A</sup> Rosin soap	4.0 ± 0.05	Isopropyl alcohol 100	None	0.5N
FA esters	20 ± 0.1	Isopropyl alcohol 100	None	0.1N
Rosin CTO <sup>A</sup> Pitch	4.0 ± 0.05	Toluene 25	Isopropyl alcohol 75	0.5N
Rosin esters 10 ± 0.1 g	10 ± 0.1	Toluene 25	Isopropyl alcohol 75	0.1N

<sup>A</sup> DTO represents distilled tall oil, while CTO represents crude tall oil.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications, and are the direct responsibility of Subcommittee D01.34 on Naval Stores.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.