Designation: ~~D464 – 05 (Reapproved 2010)~~ D464 – 15

Standard Test Methods for Saponification Number of ~~Naval Store~~Pine Chemical Products Including Tall Oil and Other Related Products[1]

This standard is issued under the fixed designation D464; 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 saponification number of tall oil and products obtained by the fractionation of tall oil such as rosin, fatty acids and distilled tall oil as defined in Terminology D804. These test methods are also applicable to gum and wood rosin. Two test methods are covered as follows:

1.1.1 Test method using a potentiometric method, and

1.1.2 Test method using an internal indicator method.

1.2 The potentiometric method is suitable for use with both light- and dark-colored test samples. It should be considered the referee method. The internal indicator method is suitable for use only with light- and medium-colored test samples. It should be considered the alternate method.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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]

D803 Test Methods for Testing Tall Oil

D804 Terminology Relating to Pine Chemicals, Including Tall Oil and Related Products

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Significance and Use

3.1 These test methods are designed to broaden the scope of the earlier editions of the test method by the inclusion of tall oil and tall oil derived products as test materials and is referenced in Test Methods D803.

3.2 The saponification number is an important property of tall oil and the products obtained by the fractionation of tall oil. It is the test method widely used to determine the total acid content, both free and combined, 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. Changes are very pronounced on ground rosin that has a large surface area exposed to air. Existing rosin dust and powdered rosin must not be used.

4.2 If the sample is a nonhomogeneous 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

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[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~~standard's Document Summary page on the ASTM website.

water bath to avoid overheating. When dealing with crystallized rosin, a higher temperature of approximately 160°C may be needed. Remove samples for testing only when the entire sample is homogeneous and has been well stirred.

5. Purity of Reagents and Water

5.1 Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society,[3] where such specifications are available. References to water shall be understood to mean distilled or deionized water.

POTENTIOMETRIC TEST METHOD (Referee Method)

6. Apparatus

6.1 *Erlenmeyer Flask,* 250-mL, of chemically resistant glass[4] with standard-taper glass joint.

6.2 *Hot Plates.*

6.3 *Water-Cooled Reflux Condenser,* with standard-taper joint to fit the Erlenmeyer flask.

6.4 *Buret,* with 0.1 mL divisions.

6.5 *Stirrer,* variable-speed, with a polytetrafluoroethylene (PTFE) coated magnetic stir bar.

6.6 *Delivery or Volumetric Pipet,* 50-mL constant.

6.7 *PTFE Boiling Stones,* or glass beads.

6.8 *Beaker,* 400-mL tall-form.

6.9 *Glass Electrode pH Meter,* conforming to the requirements of Test Method E70. Use either standard or alkali-resistant electrodes for this test. Alternatively, an automatic potentiometric titrator may be used.

7. Reagents

7.1 *Ethyl Alcohol*—~~Ethyl Alcohol~~(95 %) denatured by Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue.[5]

7.2 *Isopropyl Alcohol,* Reagent grade.

7.3 *Toluene,* Reagent grade.

7.4 *Alkali Solution, Standard Alcoholic (0.5 N)*—Dissolve 33 g of potassium hydroxide (KOH), preferably in pellet form, in ethyl alcohol conforming to 7.1 and dilute to 1 L with 3A ethyl alcohol. Standardize to ±0.001 *N* by dissolving potassium acid phthalate (C_6H_4 COOKCOOH) in 60 mL of water followed by the addition of 40 mL of isopropyl alcohol. Once the potassium acid phthalate has dissolved, 2.553 g of potassium acid phthalate will be neutralized by 25.0 mL of 0.5 *N* KOH solution. Protect the standardized solution against evaporation and absorption of carbon dioxide (CO_2) from the air. The solution should be standardized either potentiometrically or colorimetrically using either phenolphthalein or thymol blue as the indicator. The standardization should use the same equipment and techniques as used in the actual saponification number determination.

7.5 *Acid, Standard (0.5 N)*—Standardize a 0.5 *N* solution of HCl to ±0.001 *N* by any accepted procedure.

7.6 *Borax Buffer, Standard Solution (0.01 M, pH 9.18 at 25°C)*—Dissolve 3.81 ± 0.01 g of disodium tetraborate ($Na_2B_4O7{\cdot}10$ H_2O) in water and dilute to 1 L in a volumetric flask. Use the special grade[6] of borax prepared specifically for use as a pH standard. As an alternative, commercially available buffer with a pH between 9 and 11 may be used.

8. Procedure

8.1 Transfer 2.95 to 3.05 g of the sample, weighed to the nearest 0.001 g, to the Erlenmeyer flask. If necessary, 10 mL of isopropyl alcohol-toluene solution (1:1) can be added to the flask to ~~predissolve~~pre-dissolve the sample. Using a constant delivery pipet or volumetric pipet add 50.0 mL of the alkali solution. Add several PTFE boiling stones or glass beads and connect the flask to the condenser.

8.2 Place the flask on a hot plate and maintain the solution at reflux for 1 h. At the end of the reflux time, while the sample is still warm, transfer the contents of the Erlenmeyer flask into a 400-mL tall-form beaker rinsing with 100 mL of isopropyl alcohol

[3] *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.

[4] Alkali-resistant glasses, or equivalent, are suitable for this purpose. Borosilicate flasks may be used, but they should either be new or be cleaned by rinsing with a hot solution of HF (2 or 3 %). This removes from the flasks the adhering partially disintegrated silicates that would interfere with the determination.

[5] Available from the U.S. Bureau of Alcohol, Tobacco, and Firearms, Distilled Spirits and Tobacco Branch, 1200 Pennsylvania Ave., NW, Washington DC 20226, http://www.atf.gov.

[6] The National Institute of Standards and Technology standard sample of borax No. 187 is satisfactory for this purpose.