



Designation: D1386 – 15 (Reapproved 2022)

Standard Test Method for Acid Number (Empirical) of Synthetic and Natural Waxes¹

This standard is issued under the fixed designation D1386; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of the acid number of synthetic waxes and natural waxes. The number is obtained by direct titration of the material and indicates the amount of free acid present.

1.2 This test method, using an ethanol-xylene mixture, is applicable to all natural waxes, including carnauba. The test method is also applicable to oxidized microcrystalline waxes, oxidized Fischer-Tropsch, oxidized polyethylene, and montan esters.

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

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Document

2.1 *ASTM Standards:*²

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

3. Terminology

3.1 *Definitions:*

¹ This test method is under the jurisdiction of ASTM Committee D21 on Polishes and is the direct responsibility of Subcommittee D21.02 on Raw Materials.

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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.

3.1.1 *acid number or acid value*—the number of milligrams of potassium hydroxide necessary to neutralize 1 g of the sample.

4. Significance and Use

4.1 This test method is used to determine the property of acid functionality. Acid functionality determines the utility of the wax as well as being a significant Quality Control test.

5. Apparatus

5.1 *Analytical Balance.*

5.2 *Buret, 50 mL, with 0.1 mL graduations.*

5.3 *Flasks, acid value, 250 mL.*

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent-grade chemicals or equivalent, as specified in Practice E200, shall be used in all tests.

6.2 *Ethanolic Potassium Hydroxide, Standard Solution*—Dissolve approximately 5.6 g of potassium hydroxide in 5.6 g of distilled water. Dilute with USSD3A denatured ethanol or 95 % ethanol to 1000 mL. Standardize with 0.1 N hydrochloric acid.

6.3 *Phenolphthalein Indicator Solution (10 g/litre)*—Dissolve 1 g of phenolphthalein in 100 mL of USSD3A, denatured ethanol or 95 % ethanol.

6.4 *Xylene.*

7. Procedure

7.1 Transfer 1 g to 2 g of the sample, weighed to the nearest 0.001 g, to a 250 mL acid-value flask. Add 40 mL of xylene. Heat on a hot plate or water bath to put the sample into solution. Occasional swirling may be necessary.

7.2 Add 3 to 5 drops of phenolphthalein indicator solution and titrate the hot solution to the first persistent pink color. The end point is taken when the pink color remains for at least 10 s. Swirl the flask vigorously during the titration. If precipitation of waxes occurs during titration, reheat the sample. The titration should be carried out as quickly as possible. Record

the number of millilitres of standard alkali solution used.
Warning—To avoid saponification, do not reheat the solution during this operation.

7.3 Determine the blank titration value by repeating the procedure of 7.1 and 7.2 without the addition of sample material.

7.4 Run a total of three titrations of sample being tested and one blank.

8. Calculation

8.1 Calculate the acid number as follows:

$$\text{Acid number} = (A - B) \times N \times (56.1)/C$$

A = millilitres of alkali solution required for titration of the sample,

B = millilitres of alkali solution required for titration of the blank sample,

N = normality of the alkali solution, and

C = grams of sample used.

9. Precision and Bias

9.1 *Precision*—Duplicate results by the same operator shall not be considered suspect unless the results are greater than a standard deviation of 0.8.

9.2 *Bias*—This test has no bias because the values produced are defined only in terms of this test method.

10. Keywords

10.1 acid number; free acid; natural waxes; polish; synthetic waxes; titration; waxes

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