Designation: D 1386 – 98

Standard Test Method for Acid Number (Empirical) of Synthetic and Natural Waxes[1]

This standard is issued under the fixed designation D 1386; 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 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 *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 Document

2.1 *ASTM Standards:*

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[2]

3. Terminology

3.1 *Definition:*

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 E 200, shall be used in all tests.

6.2 *Ethanolic Potassium Hydroxide, Standard Solution (0.1 N)*—Dissolve 6.6 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 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.

NOTE 1—**Caution:** To avoid saponification, do not reheat the solution during this operation.

8. Calculation

8.1 Calculate the acid number as follows:

$$\text{Acid number} = (AN \times 56.1)/B$$

where:

A = millilitres of alkali solution required for titration of the sample,
N = normality of the alkali solution, and
B = 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.

[1] This test method is under the jurisdiction of ASTM Committee D-21 on Polishes and is the direct responsibility of Subcommittee D21.02 on Raw Materials.

Current edition approved Sept. 10, 1998. Published December 1998. Originally published as D 1386 – 55 T. Last previous edition D 1386 – 83 (1993)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 15.05.