Designation: D7389 – 07 (Reapproved 2012)

Standard Test Method for Acid Number (Empirical) of Maleic Anhydride (MAH) Grafted Waxes[1]

This standard is issued under the fixed designation D7389; 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 maleic anhydride (MAH) grafted waxes. The number is obtained by direct titration of the material and indicates the amount of free acid present.

1.2 This test method is applicable to MAH-grafted waxes because it uses a special sample preparation step (7.1) that is not required for other waxes. The special sample preparation reverses the hydrolysis of acid anhydride that can occur during storage of the wax.

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]

D1386 Test Method for Acid Number (Empirical) of Synthetic and Natural Waxes

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

3. Terminology

3.1 *Definitions:*

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 free acid content of MAH-grafted waxes. The potential hydrolysis of the anhydride functionality of this wax in storage makes them unsuitable for determining free acid content by Test Method D1386. Free acid content is a significant quality control test, and is a determinant of the utility of the wax.

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 (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. Sample Preparation

7.1 Melt 20 g of the sample wax in a 250-mL flask or beaker and heat to 180–190°C. Apply a slight vacuum as the wax melts and hold at temperature for 10 to 15 min, or as long as bubbles appear in the melt. If a vacuum is not available, hold the sample at 180–190°C for 4 h. Determination of the acid number is made immediately after dehydration. Over heating or holding the sample for more than 4 h will cause excess darkening of the wax and making the end point difficult to see (8.2).

8. Procedure

8.1 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.

[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.

Heat the mixture on a hot plate or water bath to dissolve the sample. Occasional swirling may be necessary.

8.2 Add 3 to 5 drops of phenolphthalein indicator solution and titrate the hot solution to the first persistent pink color. The end point it 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. To avoid saponification, do not reheat the solution during this operation.

9. Calculation

9.1 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.

10. Precision and Bias

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

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

11. Keywords

11.1 acid anhydride function; acid number; free acid; MAH grafted waxes; polish; synthetic waxes; titration; waxes