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Designation: D 1387 – 89 (Reapproved 2002)

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

This standard is issued under the fixed designation D 1387; 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 saponification number of synthetic waxes and natural waxes.

1.2 This test method is applicable to Fischer Tropsche, microcrystalline, polyethylene, and Montan Ester waxes.

1.3 Certain synthetic waxes, notably copolymers of ethylene, exhibit poor reproducibility when running saponification values. Reproducibility can be improved if cooking time in 7.2 is extended from 3 h to 18–20 h.

1.4 Some oxidized polyethylene and other waxes with a melt temperature above 100°C may give poor reproducibility.

1.5 Some dark-colored (Gardner Color 14) waxes may obscure the color change of the indicator, resulting in poor reproducibility.

1.6 *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:*

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 *saponification number*—the number of milligrams of potassium hydroxide required to hydrolyze 1 g of the sample and is a measure of the amount of saponifiable matter present.

4. Significance and Use

4.1 This test method is used to determine the property of ester functionality. Ester functionality determines the utility of the wax as well as being a significant quality control test.

5. Apparatus

5.1 *Analytical Balance*.

5.2 *Boiling Chips*, chemically resistant glass.

5.3 *Burets*, two 50-mL capacity with 0.1-mL graduations.

5.4 *Erlenmeyer Flasks*, 250-mL, alkali-resistant.

5.5 *Hot Plate*.

5.6 *Reflux Condenser*.

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 *Hydrochloric Acid Standard* (*0.5 N*).

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 *Potassium Hydroxide, Alcoholic Solution* (*6.6 g/litre*)—Dissolve 6.6 g of potassium hydroxide (KOH) in USSD3A denatured ethanol or 95 % ethanol. Dilute to 1 L with the ethanol.

6.5 *Xylene*.

7. Procedure

7.1 Transfer approximately 1 g of the sample, weighed to the nearest 0.001 g to a 250-mL Erlenmeyer flask.

7.2 Add 40 mL of xylene and a few boiling chips to the flask. Dissolve by heating on the hot plate to the boiling point of xylene. As soon as the wax dissolves, remove from the hot plate and add 50.0 mL of 0.1 *N* ethanolic KOH solution from the buret. Fit the flask with a reflux condenser and reflux for 3 h using the hot plate.

7.3 Remove the condenser from the flask, add 5 drops of the phenolphthalein solution and titrate the sample with 0.5 *N* HCl until the pink color disappears. Reheat the sample to the boiling point, and if it turns pink, resume titration until the color once again disappears. Repeat this procedure until the pink color does not reappear on heating. Saponified waxes usually require two repetitions of heating and additional titration until the pink color does not reappear.

[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.

Current edition approved May 26, 1989. Published July 1989. Originally published as D 1387–55T. Last previous edition D 1387–84.

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

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