Designation: D 1342 – 92 (Reapproved 2002)

# Standard Test Method for Paraffin-Type Hydrocarbons in Carnauba Wax[1]

This standard is issued under the fixed designation D 1342; 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, by chromatography, of paraffin-type hydrocarbons in carnauba wax.

1.2 *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:*

D 94 Test Method for Saponification Number of Petroleum Products[2]

## 3. Significance and Use

3.1 This test method is used to determine the natural or added hydrocarbon component of carnauba. It is used for quality control or to assess adulteration.

## 4. Apparatus

4.1 *Cylindrical Separatory Funnel*, 250-mL capacity, having an open top.

4.2 *Analytical Balance*, having a sensitivity of 0.1 mg.

4.3 *Electric Hot Plate*.

4.4 *Electric Oven*, capable of maintaining a temperature of 100 ± 5°C.

## 5. Reagents

5.1 *Alumina*, having an activity of II[3] according to the Brockmann classification.[4] Since alumina of an activity of I according to the classification also has been found to be satisfactory for the separation, any alumina falling in the activity range from I to II is suitable for use in this test method. The alumina must be kept free from moisture to retain its adsorptive properties.

5.2 *Heptane*, technical grade.

## 6. Procedure

6.1 Place a wad of glass wool in the bottom of the separatory funnel and pour 220 to 225 g of alumina on top of this plug. Tap the side of the funnel sharply several times with the palm of the hand in order to pack the alumina evenly, and then cover the adsorbent with a thin layer of glass wool. Immediately warm the column by pouring 400 mL of heptane at its boiling point through the adsorbent with the stopcock adjusted to permit maximum flow of the solvent. Tap the column to remove entrained air. Close the stopcock after the last of the heptane has been added and while a layer about 10 mm deep still remains above the surface of the alumina.

6.2 Dissolve 4.0 to 4.5 g of the sample, weighed to the nearest 0.01 g, in 700 mL of boiling heptane in a 1-L, tall-form beaker. Pour the solution quantitatively onto the warm column of alumina. Collect the percolate in a beaker placed under the funnel with the stopcock in a position to permit maximum flow through the column. Maintain the temperature of the solution being added and the column at approximately the boiling point of heptane throughout the course of the addition. The temperature of the percolate issuing from the funnel stem shall be at 50 to 55°C. After the last of the solution has been added, rinse the tall-form beaker with two successive 50-mL portions of fresh, boiling heptane and add the washings to the column. At this stage a yellow band, which should not extend more than about 40 mm down from the top of the alumina, will have developed. A wider band than this probably indicates that the column was not packed properly. In such a case it may be difficult to effect a clean separation of the hydrocarbons in one pass through the alumina.

6.3 Elute the column with additional heptane at within 5°C of its boiling point and, when about 1 L of percolate has been obtained, begin to test subsequent portions of the percolate for complete elution of the hydrocarbons. To do this, collect 50-mL portions of the percolate in separate 100-mL beakers, evaporate the solvent on the hot plate, and allow the beakers to cool to room temperature. Elution of the paraffin-type hydrocarbons is complete when no solids or only a trace of greasy residue is obtained in one of these beakers.

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[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] *Annual Book of ASTM Standards*, Vol 05.01.

[3] The F-20 grade furnished by the Aluminum Ore Co., East St. Louis, Ill., conforms to this requirement.

[4] H. Brockmann and Hella Schodder, "Aluminiumoxyd mit Abgestuftem Adsorptions-vermögen zur Chromatographischen Adsorption," *Berichte der Deutschen Chemischen Gesellschaft*, Vol 74, pp. 73–78.

6.4 With the aid of warm heptane, transfer all residues quantitatively from the small beakers to the main percolate, evaporate the solvent until the volume is 30 to 50 mL, and with the aid of warm heptane, transfer the solution quantitatively to a beaker that has been weighed to the nearest 0.2 mg. Heat the beaker on the hot plate until the solvent is removed almost completely, and then transfer the beaker to the 100°C oven for 2 h. Cool and weigh the beaker and paraffin-type hydrocarbons.

6.5 Determine the saponification number of the percolated solids in accordance with Test Method D 94, but because of the small available sample and its low saponification number, use 0.1 *N* alcoholic KOH solution and 0.1 *N* HCl in place of the 0.5 *N* solutions used in Test Method D 94. The saponification number should not be higher than 2, if a clean cut separation of paraffin-type hydrocarbons had been effected. If this saponification value is exceeded, repeat the procedure with a fresh sample and a fresh adsorbent.

## 7. Calculations

7.1 Calculate the percentage of paraffin-type hydrocarbons in the sample as follows:

$$\text{Paraffin}-\text{type hydrocarbons, \%} = (A/B) \times 100$$

where:

$A$ = grams of solids recovered from the heptane percolate, and

$B$ = grams of sample used.

## 8. Precision and Bias

8.1 Duplicate results should agree within 0.3 %.

8.2 Results obtained with samples containing known hydrocarbon content will agree within 0.5 % between laboratories.

8.3 The procedure in this test method has no bias because the values for the percent paraffin-type hydrocarbons is defined only in terms of this method.