ETHIOPIAN STANDARD

ES 7246:2025

First edition
xx-xx-2025

Carnauba Wax — Specification

ICS: 67.220.10
Published by Institute of Ethiopian Standards

Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for spice and condiments (TC 14) and published by the Institute of Ethiopian Standards (IES).

The standard has been developed to address observed needs and to support the local industry in order to make progress through uprising competitiveness and maintain comparative market advantage both domestically and internationally.

In preparing this standard reference has been made to the following documents:

Carnauba Wax — Specification

1. Scope

This Ethiopian Standard specifies the requirements and methods of sampling and testing for Carnauba wax derived from the leaves of the Carnauba palm (*Copernicia prunifera*).

2. Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ASTM D1386, *Standard Test Method for Acid Number (Empirical) of Synthetic and Natural Waxes*

ASTM D1387, *Standard Test Method for Saponification Number (Empirical) of Synthetic and Natural Waxes*

ASTM D1342, *Standard Test Method for Paraffin-Type Hydrocarbons in Carnauba Wax.*

Pharmacopeia standards (e.g., Ph. Eur., USP) for testing methods where applicable.

3. Terms and Definitions

For the purpose of this standard the following definitions shall apply.

3.1
carnauba wax
is a natural hard, yellowish to brown vegetable wax derived from the leaves of the *Copernicia prunifera* botanical, more commonly known as the Carnauba tree.

3.2
crude carnauba wax
the raw, unrefined wax obtained after the initial extraction from the palm leaves.

3.3
refined carnauba wax
the wax obtained after subjecting crude Carnauba wax to purification processes such as filtration, centrifugation, and bleaching to achieve a desired purity and color.

4. Types of Carnauba Wax

The commercial Carnauba wax shall be of the following types, graded based on purity, color and physical property.

4.1 Type 1 (T1) *: The highest purity grade, typically light yellow in color, obtained from young, unopened leaves. It is used in food, pharmaceutical, and cosmetic applications.

4.2 Type 3 (T3): A darker, brownish-yellow wax used in general cosmetic, polish, and coating applications.

4.3 Type 4 (T4): The darkest grade, often brownish-black, suitable for industrial applications like polishes where color is not a primary concern.

5. Requirements

5.1 General Requirements

5.1.1 The Carnauba wax shall be in the form of hard, brittle flakes, powder, or blocks, as agreed upon between the purchaser and the supplier.

5.1.2 The color shall range from light yellow to yellowish-brown or dark brown. It shall have a characteristic, pleasant aroma when melted.

*In most international trade and technical literature, carnauba wax is standardized mainly into Type 1, 3, and 4 (T1, T3, and T4). A Type 2 (T2) is sometimes mentioned, but it is not widely recognized in the global standard classifications

5.1.3 Carnauba wax shall be practically insoluble in water and ethanol but becomes soluble upon heating in organic solvents such as ethyl acetate and xylene.

5.1.4 Carnauba wax shall be free from leaf debris, sand, or any other extraneous matter.

5.1.5 Carnauba wax shall be pure and unadulterated. It shall not be blended or contain any other waxes such as paraffin, microcrystalline, or synthetic waxes, nor shall it be mixed with any oil, fat, or any other contaminant.

5.2 Specific Requirements

Carnauba wax shall comply with the requirements given in Table 1 below.

Table 1 Specific requirements of Carnauba Wax

S.No	Characteristics	Requirements Type 1 (T1)	Requirements Type 3 (T3)	Requirements Type 4 (T4)	Test methods	References
1	Specific gravity at 20 °C	About 0.997	About 0.997	About 0.997	Annex A	(EFSA, 2012)
2	Melting point, °C	80–86	80–86	80–86	Annex B	(The United States Pharmacopeial Convention, 2012); (Dhariwal Corp Limited, n.d.)
3	Refractive index at 90 °C	1.450–1.460	1.450–1.460	1.450–1.460	Annex C	(JECFA, 1998)
4	Ash, % by mass, max.	< 0.25	< 0.50	< 1.0	Annex D	(The United States Pharmacopeial Convention, 2012); (OMRI, 2014)
5	Total volatile matter, % by mass, max.	< 0.5	< 1.0	< 1.0	Annex E	(OMRI, 2014)
6	Acid value, mg KOH/g	2–7	4–10	4–10	Annex F	(JECFA, 1998; USP, 2012); (Dhariwal Corp Limited, n.d.); (OMRI, 2014)
7	Saponification value, mg KOH/g	78–95	78–95	80–90	Annex G	(JECFA, 1998; USP, 2012); (Dhariwal Corp Limited, n.d.); (OMRI, 2014)
8	Ester value, mg KOH/g	71–93	68–91	70–86	Annex H	(JECFA, 1998); Calculated
9	Paraffin and other waxes	To pass the test	To pass the test	To pass the test	Annex J	(ASTM D1342)
10	Lead (Pb), max	2 mg/kg	2 mg/kg	2 mg/kg	Annex K	(EFSA, 2012)
11	Arsenic (As), max	3 mg/kg	3 mg/kg	3 mg/kg	Annex L	(EFSA, 2012)
12	Sulphated ash, % by mass, max	< 0.25	< 0.25	< 0.25	Annex M	(EFSA, 2012)

6. Packing and Labeling

6.1 Packing

6.1.1 Carnauba wax shall be packed in greaseproof paper or any suitable material like polythene, jute, sisal bags or with agreement between the purchaser and the supplier. A number of such blocks shall be packed together in a suitable and clean packaging material.

6.1.2 The packaging material shall be clean and free from any chemical contamination.

6.1.3 The packaging material shall be moth proof.

6.1.4 The packaging material shall be kept dry and free from moisture.

6.2 Labeling

The labeling shall comply with the requirements of CES 73, and shall be legibly and indelibly marked with the following:

a) name of the product as" Carnauba wax";

b) name and address of the manufacturer or packer;

c) lot or batch number;

d) net weight in SI unit;

e) producing country;

f) date of manufacturing (dd/mm/yy);

g) best before date (dd/mm/yy);

h) trade name or brand name, if any;

i) storage recommendation; and

j) instruction for use.

7. Sampling

7.1 General Precautions

7.1.1 Samples shall be taken in a protected place not exposed to damp air, dust, or soot.

7.1.2 The sampling instrument (e.g., trier, auger) shall be clean and dry when used.

7.1.3 Precautions shall be taken to protect the samples, the sampling instrument, and the containers from adventitious contamination.

7.1.4 The sample shall be placed in clean and dry containers.

7.1.5 Each container shall be marked with full details of sampling, batch or lot number, name of the supplier, and other important particulars of the consignment.

7.1.6 Samples shall be stored in such a manner that the temperature of the material does not vary from the normal temperature.

7.1.7 The storage of Carnauba wax shall be placed in a manner that allows accessibility for sampling each part of the lot.

7.2 Scale of sampling

7.2.1 Lot: All containers of Carnauba wax in a single consignment of the same type and from a single batch shall constitute a lot.

7.2.2 The number of containers to be selected from the lot shall be in accordance with Table 2. The containers shall be selected at random.

Table 2: Lot size and number of containers to be selected

Number of containers in a Lot	Number of containers to be selected
Up to 25	3
26 to 100	4
101 to 500	5
501 to 1000	7
1001 and above	9

7.3 Preparation of Samples

7.3.1 Take equal quantities of sample with a suitable instrument from different parts of the selected containers to obtain a total of approximately 500g.

7.3.2 Divide the sample into three equal parts. Each part shall constitute an individual sample and shall be transferred immediately to thoroughly clean and dry containers, which are then sealed and marked with full particulars.

7.3.3 One set of individual samples shall be marked for the supplier, another for the laboratory, and the third for the referee.

7.3.4 From the remaining material, a composite sample of about 150g shall be prepared by mixing equal quantities.

Annex A
(Normative)
Determination of specific gravity

A.1Apparatus

A.1.1 Water-Bath - maintained at 20 ± 1 ^{0}C.

A.1.2 Specific Gravity Bottle-25ml capacity.

A.2 Reagent

A.2.1Alcohol

A.3 Procedure

A.3.1 Melt approximately 2g of the material in a porcelain crucible at a temperature of about 100^{0}C.

A.3.2 Allow to cool to room temperature. Remove the solidified bees wax from the crucible, warming slightly if necessary.

A.3.3 Attach a tarred silk or similar item thread that will suspend the beeswax during weighing.

A.3.4 Store the sample for 2 hours at a temperature of 20 ±1^{0}C. Determine the mass of the sample, first in air and then in rectified spirit maintained at 20± 1^{0}C. Determine the specific gravity at 20^{0}C /20^{0}C of the rectified spirit by means of the specific gravity bottle.

A. 4.Calculation

Specific gravity at 20^{0}C/20^{0}C = $\frac{M1xd}{M1-M2}$

Where

M1-mass in g of the material in air,

d- specific gravity of rectified spirit, and

M2- mass in g of the material in alcohol.

Annex B
(normative)
Determination of melting point

B.1Apparatus

B.1.1 Thermometer-of a suitable type, with an accuracy of 0.1^0C and graduated at every 0.1^0C.

B.1.2 Test-Tube-with a centrally bored cork to take the thermometer. The cork shall have a slit so as to permit circulation of air.

B.1.3 Water-Bath-of a suitable type, with a thermometer.

B.2 Procedure

B.2.1 Melt the material by warming it in a water-bath at a temperature just sufficient to melt it.

B.2.2 Dip the thermometer and withdraw, so as to get the bulb thinly coated with the wax. Let it stand for 24 hours.

B.2.3 Insert this thermometer into the test-tube through the bored cork and then place the test-tube in the water-bath.

B.2.4 Raise the temperature gradually, at the rate of 1 0C in 3 minutes. Note the temperature, accurately to 0.1 0C, at which a transparent drop forms on the end of the thermometer bulb. Record this temperature as the melting point of the material.

Annex C
(Normative)
Determination of refractive index

C.1Apparatus

C.1.1Refractometer

C.1.2 Water bath thermostatically controlled to maintain refractometer prism at 75±1^{0}C

C.2 Procedure

C.2.1 The refractometer should be calibrated using reference material or distilled water.

C.2.2 The sample shall be melted and filtered through fast filter paper to remove any impurities and last traces of moisture.

C.2.3 The temperature of the refractometer shall be adjusted at 75±1^{0}C by circulating water from the water bath.

C.2.4 Place few drops of the sample on the lower prism, the prism shall be closed tightened firmly allowed to stand for one or two minutes.

C.2.5 Read and record the refractive index after the sample has attained the test temperature**.**

Annex D
(Normative)
Determination of ash

D.1Apparatus

D.1.1 Desiccators with desiccant.

D.1.2 Muffle furnace capable of operating at a temperature of 550^0C–650^0C.

D.1.3 Analytical balance Capable of weighing 1mg.

D.1.4 Platinum, porcelain or silica Dish-having a capacity of 100 ml.

D.2 Procedure

D.2.1 Heat the dish at 550^0C -650^0C for 1hour, cool to room temperature in a desiccator and weigh. Take about 50g of the material in a watch-glass and weigh accurately.

D.2.2 Transfer about three-quarters of this quantity to the platinum dish and heat on a Bunsen burner so that the material burns gently at the surface. When about half of the material is burnt away, stop heating, cool and add the remainder of the material.

D.2.3 Weigh the watch-glass again and find, by difference, the exact mass of sample transferred to the platinum dish. Heat again as before till the material is completely charred.

D.2.4 Incinerate in a muffle furnace at 550^0C to 650^0C for 1hour. Cool to room temperature in a desiccator and weigh. Repeat incineration, cooling and weighing until the difference between two successive weightings is less than one milligram**.**

D.3 Calculation

$$\text{Ash, percent by mass} = \frac{100M2}{M1}$$

Where,

M2-mass in g of the ash, and

M1- mass in g of the material taken for the test.

Annex E
(Normative)
Determination of Total volatile matter

E.1 Apparatus

E 1.1 Oven maintained at 105^0C.

E.1.2 Analytical balance Capable of weighing 1mg.

E.1.3 Metal or aluminum dish.

E.2Procedure

E.2.1 Dry the dish at 105^0C for 1hour, cool to room temperature in a desiccator and weigh.

E.2.2 Weigh accurately about 10g of the material in a suitable dish, previously dried and weighed, and place it in an oven maintained at 105± 2^0C for 6 hours.

E.2.3 Cool the dish in a desiccator and weigh with the lid on. Heat the dish again in the oven for 30 minutes**.**

E.2.4 Repeat the process until the loss in mass between two successive weightings in less than one milligram. Record the lowest mass obtained.

E. 2.5 Calculation

Total volatile matter at 105^0C, percent by mass **=** $\frac{100(M1-M2)}{M1-M3}$

Where,

M_1 –mass in gram of the dish with the material before heating.

M_2 - mass in gram of the dish with the material after heating.

M_3 - mass in gram of the empty dish.

Annex F
(Normative)
Determination of acid value

F.1Principle

The acid value is determined by titrating the material in benzene-alcohol medium with potassium hydroxide solution.

F.2 Apparatus

F.2.1 Burette 10-25ml capacity graduated by 0.1ml

F.2.2 Conical Flasks-250ml capacity.

F.2.3 Reflux condenser - at least 65 cm long.

F.2.4 Analytical balance Capable of weighing 1mg.

F.2.5 Hot plate or water bath.

F.3Reagents

F.3.1 Benzene-neutral to phenolphthale inindicator.

F.3.2 Rectified Spirit-neutral to phenolphthale inindicator.

F.3.3 Standard Potassium Hydroxide Solution 0.5N

F.3.4 Phenolphthalein Indicator Solution Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit and dilute with water to 100 ml.

F.4Procedure

F.4.1 Mix the material thoroughly, making it entirely liquid before weighing.

F.4.2 Weigh accurately about 5 g of the material in a 250-m1 conical flask. Add 75 ml of a mixture of two parts of benzene and one part of rectified spirit. F2.2 Heat under reflux condenser until the sample is dissolved. Allow it to cool to room temperature add 3-5 drop of Phenolphthalein Indicator Solution and titrate with standard potassium hydroxide solution .and record the volume of potassium hydroxide solution

F.4.3 Calculation

$$\text{Acid value} = \frac{56.1VN}{M}$$

Where

V-volume in ml of standard potassium hydroxide solution used,

N-normality of standard potassium hydroxide. Solution, and

M- mass in g of the material taken for the test.

Annex G
(Normative)
Determination of saponification value

G.1 Principles

The material saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali consumed for saponification is determined by titrating the excess alkali with standard acid.

G.2 Apparatus

G.2.1 Conical Flasks-250 to 300ml capacity made of alkali-resistant glass.

G.2.2 Reflux condenser - at least 65 cm long. 2.3AnalyticalbalanceCapableofweighing 1mg

G.2.4 Hot plate or water bath

G.2.5 Burette10-25ml capacity graduated by 0.1ml

G.3 Reagents

G.3.1 Methyl Ethyl Ketone(2-Butanone)

G.3.2 Rectified Spirit**-**

G.3.3 Alcoholic Potassium Hydroxide. Solution -Dissolve 30 g of potassium hydroxide in rectified spirit and make up to 1 liter. Allow to settle overnight in a dark place, decant the clear liquid and keep in a bottle closed tight with cork or rubber stopper.

G.3.4 Phenolphthalein Indicator Solution Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit and dilute with water to 100 ml.

G.3.5 Standard Hydrochloric Acid 0.5 N`.

G.4 Procedure

G.4.1 Weigh accurately about 2.0 g of the material in a tarred conical flask .Add 25 ml of methyl ethyl ketone, followed by25 ml of alcoholic potassium hydroxide solution. Add a few pieces of pumice stone and connect the reflux condenser to the flask. Heat the flask on a water-bath or electric hot-plate for about 2 hours. Boil steadily but gently. After the flask and condenser have cooled, wash down the inside of the condenser with about 10 ml of rectified spirit. Add about 1 ml of phenolphthalein indicator solution and titrate with standard hydrochloric acid (G3.5). Carry out a blank determination at the same time.

G.4.2Calculation

$$\text{Saponification value} = \frac{56.1(B-S)N}{M}$$

Where,

B= volume in ml of standard hydrochloric acid required for the blank,

S= volume in ml of standard hydrochloric acid required for the material,

N= normality of standard hydrochloric acid, and

M= mass in g of the material taken for the test.

Annex H
(Normative)
Determination of ester value

H.1 Principle

This committee draft specifies the method of deterring the ester value in bees wax by calculation

H.2 Expression of result

The ester value shall be calculated as follow:

The acid value determined according to Annex F shall be subtracted from the saponification value determined according to Annex G to get the ester value.

Ester value = Saponification value – Acid value

Annex I
(Normative)
Test for paraffin and other waxes

I.1 Principle

The material is treated with alcoholic potassium hydroxide solution and boiled under reflux and determination of paraffin and other waxes based on formation of cloudy at specific temperature.

I.2 Apparatus

I.2.1 Analytical balance Capable of weighing 1mg.

I.2.2 Conical flask 250ml.

I.2.3 Reflux condenser.

I.2.4 Water bath or hot plate.

I.3 Reagents

I.3.1 AlcoholicPotassium Hydroxide Solution approximately 0.5N, prepared by dissolving potassium hydroxide in 95 percent ethanol.

I.3.2 Ethanol95%.

I.4 Procedure

I.4.1 Weigh 1.0 g of the material and place it in a conical flask fitted with a water-cooled reflux condenser. Add 10 ml of alcoholic potassium hydroxide solution. Boil under reflux for one hour. Detach the flask from the condenser, insert suitably a thermometer into the liquid in the flask and allow to cool, stirring constantly.

I.4.1.1 The material shall be taken to have passed the test if the following conditions are satisfied:

a) The liquid does not become cloudy at a temperature higher than 61^0C but becomes cloudy at a temperature between 61^0C and 59^0C, and

b) Precipitation of large flocks occurs at not more than 2^0C below the temperature at which the liquid becomes cloudy**.**

Example: If the liquid becomes cloudy at 60°C, precipitation should take place at a 58^0C.

Annex J
(normative)
Determination of Sulphated ash

J.1 Principle

The material is treated with sulfuric acid and the sulphated ash content is determined by ignition on muffle furnace.

J.2 Apparatus

J.2.1 Desiccators with desiccant.

J.2.2 Muffle furnace capable of operating at a temperature of 550^0C–650^0C.

J.2.3 Analytical balance Capable of weighing 1mg.

J.2.3 Platinum, porcelain or silica Dish-having a capacity of 100ml.

J.2.4 Reagent

J2.5 Sulphuric Acid-10 percent.

J.3 Procedure

Accurately weigh about 5g of the prepared sample to a 9-cm diameter platinum or silica crucible. Add 5 ml of sulphuric acid (3.1) to the material in the dish. Gently heat the dish on a hot plate until the material is well carbonized, and then increase the heat until the evolution of sulphuric acid fumes ceases.

Ash the carbonized matter in a muffle furnace at 550 ± 25 ^{0}C. Cool the ash and moisten it with 2 - 3 ml of sulphuric acid (3.1). Heat strongly on a hotplate until sulphuric acid fumes cease to be evolved and finally ash in muffle furnace at 550 ± 25^0C for about 1 hour. Cool in a decicator and weigh. Heat again in the muffle Furnace for 30 minutes at 550 ± 25^0C..Cooling a desiccator and weigh. Repeat the process of heating in the muffle furnace for 30 minutes, cooling and weighing till the difference between two successive weightings is less than 1mg. Record the lowest mass.

J.4 Calculation

Sulphated Ash, percent by mass = (M1/M2)x100

Where

M1-mass in g of the ash, and

M2-mass in g of the prepared sample taken for the test

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INSTITUTE OF ETHIOPIAN STANDARDS

Organization and Objectives

The Institute of Ethiopian Standards (IES) is the national standards body of Ethiopia. IES is re-named by the proclamation number 1263/2021, from Ethiopian Standards Agency (ESA) to Institute of Ethiopian standards, with the mandate given by the regulation Number, 193/2010 and proclamation number, 1263/2021.

IES's objectives are:

- Develop Ethiopian standards and establish a system that enable to check whether goods and service are in compliance with the required standards,
- Facilitate the country's technology transfer through the use of standards,
- Develop national standards for local products and services so as to make them competitive in the international market.
- Conduct standards related research and provide training and technical support.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational and research institutes, governmental organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.

Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices). A catalogue of Ethiopian Standards is also available freely and can be accessed from our website.

 International Involvement IES, representing Ethiopia, is a member of the International Organization for Standardization (ISO), International Electro-technical Commission (IEC) and Codex Alimentarius Commission (CODEX). It also maintains close working relations with the American Society for Testing and Materials (ASTM). It is a founding member of the African Regional Organization for standardization (ARSO).

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