Palmitic Acid

$C_{16}H_{32}O_2$ 256.43
Hexadecanoic acid [57-10-3].

DEFINITION

Palmitic Acid is a mixture of solid organic acids obtained from fats or oils of animal or vegetable origin. It contains NLT 92.0% of palmitic acid ($C_{16}H_{32}O_2$) and NMT 6.0% of stearic acid ($C_{18}H_{36}O_2$).

IDENTIFICATION

- **A.** The retention time of the major peak for palmitic acid of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

- **PROCEDURE**
  Sample solution: Proceed as directed for *Test solution* in *Fats and Fixed Oils* ⟨401⟩, *Fatty Acid Composition.*
  Standard solution: Prepare the *Standard solution* in the same manner as the *Sample solution*, using a mixture of 50 mg of USP Palmitic Acid RS and 50 mg of USP Stearic Acid RS instead of the substance to be examined.
  Chromatographic system: Prepare as directed in *Fats and Fixed Oils* ⟨401⟩, *Fatty Acid Composition.*
  Injection size: 1 µL
  System suitability
  Sample: *Standard solution*
  [NOTE—The relative retention times for methyl palmitate and methyl stearate are 0.9 and 1.0, respectively.]
  Suitability requirements
  Resolution: NLT 3.0 between methyl stearate and methyl palmitate
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Calculate the percentage of $C_{16}H_{32}O_2$ in the portion of Palmitic Acid taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response for methyl palmitate from the *Sample solution*
r_T = sum of the responses of all the peaks in the chromatogram except the solvent peak

Similarly, calculate the percentage of $C_{18}H_{36}O_2$ in the portion of Palmitic Acid taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response for methyl stearate from the *Sample solution*
r_T = sum of the responses of all the peaks in the chromatogram except the solvent peak

Acceptance criteria: NLT 92.0% of palmitic acid ($C_{16}H_{32}O_2$) and NMT 6.0% of stearic acid ($C_{18}H_{36}O_2$)

IMPURITIES

- **HEAVY METALS,** *Method II* **⟨231⟩:** NMT 10 ppm

SPECIFIC TESTS

- **COLOR:** Heat a sample of Palmitic Acid to 75°. The resulting liquid is not more intensely colored than a solution prepared by mixing 1.2 mL of ferric chloride CS and 0.3 mL of cobaltous chloride CS with 0.3 N hydrochloric acid to make 10 mL, and diluting 5 mL of this solution with 0.3 N hydrochloric acid to make 100 mL. Make the comparison by viewing the solutions downward in matched color-comparison tubes against a white surface (see *Color and Achromicity* ⟨631⟩).
- **CONGEALING TEMPERATURE ⟨651⟩:** 60°–66°
- **FATS AND FIXED OILS,** *Acid Value* **⟨401⟩:** 216–220, using 1 g
- **FATS AND FIXED OILS,** *Iodine Value* **⟨401⟩:** NMT 1. Proceed as directed in *Method I*, except use 35 mL of chloroform.
- **MINERAL ACID**
  Analysis: Shake 5 g of melted Palmitic Acid with an equal volume of hot water for 2 min. Cool, and filter.
  Acceptance criteria: The filtrate is not reddened by the addition of 1 drop of methyl orange TS.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers, and store at room temperature.
- **LABELING:** Label it to indicate whether it is derived from animal or vegetable sources.
- **USP REFERENCE STANDARDS ⟨11⟩**
  USP Palmitic Acid RS
  USP Stearic Acid RS

Paraffin

[8002-74-2].

» Paraffin is a purified mixture of solid saturated hydrocarbons obtained from petroleum. It may contain suitable antioxidants.

Packaging and storage—Preserve in light-resistant, well-closed containers, and avoid exposure to excessive heat.

Labeling—Label it to indicate the name and quantity of any antioxidants.

USP Reference standards ⟨11⟩—
USP Naphthalene RS
USP Paraffin RS

Identification—

A: *Infrared Absorption* ⟨197⟩, using a thin film of melted test specimen. [NOTE—Ensure complete melting to avoid doublet peaks that may be observed at wavenumbers at about 1460 cm^{-1} and 730 cm^{-1}.]

B: It meets the requirements of the test for *Congealing range.*

Congealing range ⟨651⟩: between 47° and 65°.

Acidity—Introduce 15 g into a suitable separator, add 30 mL of boiling water, and shake vigorously for about 1 minute. Allow to cool, and draw off the separated water. To 10 mL of the filtrated aqueous layer add 0.1 mL of phenolphthalein TS: the solution does not produce a pink color. Not more than 1.0 mL of 0.01 M sodium hydroxide is subsequently required to change the color of the indicator to pink.

Alkalinity—To an additional 10 mL of the filtrated aqueous layer obtained from the test for *Acidity* add 0.1 mL of methyl red TS 2: the solution produces a yellow color. Not more than 0.5 mL of 0.01 M hydrochloric acid is subsequently required to change the color of the indicator to red.

Readily carbonizable substances ⟨271⟩—Use a clean, dry, heat-resistant, glass-stoppered test tube, 140 ± 2 mm in length with an outside diameter between 14.5 and 15.0 mm and calibrated at the 5- and 10-mL liquid levels. The capacity of the tube with stopper inserted is between 13.6 and 15.6 mL.* Place in the test tube 5 mL of Paraffin at a temperature just above the melting point, add 5 mL of sulfuric acid containing 94.5% to 94.9% of H_2SO_4, and heat in a water bath at 70° for 10 minutes. When 5 minutes have elapsed, and at each successive minute thereafter, remove the tube from the bath, place a finger over the stopper, and give the tube three vigorous vertical shakes over an amplitude of about 12 cm, returning the tube to the bath within 3 seconds after the time when it was removed therefrom. At the end of 10 minutes from the time the tube was placed in the bath, the acid (lower layer) has no more color than a mixture of 3 mL of ferric chloride CS, 1.5 mL of cobaltous chloride CS, and 0.50 mL of cupric sulfate CS,

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*A suitable test tube is available from Kimble Kontes. Item number: 34-19426. Description: Nessler Tube. Contact: phone 800-682-6644, fax 856-692-6644, e-mail customglass@kimkon.com.

overlaid with 5 mL of mineral oil. If the sulfuric acid remains dispersed in the molten paraffin, the color of the emulsion is not darker than that of the standard mixture when shaken vigorously.

Limit of polycyclic aromatic hydrocarbons—

Dimethyl sulfoxide—Use spectrophotometric grade dimethyl sulfoxide.

Standard solution—Dissolve a quantity of USP Naphthalene RS, accurately weighed, in *Dimethyl sulfoxide,* and dilute quantitatively and stepwise with *Dimethyl sulfoxide* to obtain a solution having a concentration of 7.0 µg per mL. Determine the absorbance of this solution in a 1-cm cell at the wavelength of maximum absorbance at about 278 nm, using *Dimethyl sulfoxide* as the blank.

Procedure—Dissolve 0.50 g of Paraffin, accurately weighed, in 25 mL of *n*-heptane, place in a 125-mL separator with unlubricated ground-glass parts (stopper, stopcock), and mix. Add 5.0 mL of *Dimethyl sulfoxide,* and shake the mixture vigorously for 1 minute. Allow to stand until two clear layers are formed. Transfer the lower layer to another 125-mL separator, add 2 mL of *n*-heptane, and shake the mixture vigorously. Allow to stand until two clear layers are formed. Separate the lower layer, and determine its absorbance in a 1-cm cell, in the range of 265 nm to 350 nm, using as the blank *Dimethyl sulfoxide* that previously has been shaken vigorously for 1 minute with *n*-heptane in the ratio of 5 mL of *Dimethyl sulfoxide* to 25 mL of *n*-heptane. The absorbance at any wavelength in the specified range is not greater than one-third of the absorbance, at 278 nm, of the *Standard solution*.

Limit of sulfur compounds—To 4.0 g of Paraffin add 2 mL of dehydrated alcohol, and then add 2 drops of a clear saturated solution of lead (II) oxide in sodium hydroxide solution (1 in 5). Heat the mixture at 70° for 10 minutes with frequent shaking, and cool. No dark brown color develops.

Synthetic Paraffin

DEFINITION

Synthetic Paraffin is synthesized by the Fischer-Tropsch process from carbon monoxide and hydrogen, which are catalytically converted to a mixture of paraffin hydrocarbons; the lower molecular weight fractions are removed by distillation, and the residue is hydrogenated and further treated by percolation through activated charcoal. This mixture may be fractionated into its components by a solvent separation method, using a suitable synthetic isoparaffinic petroleum hydrocarbon solvent. It may contain NMT 0.005% of a suitable antioxidant.

IDENTIFICATION

- **A. Infrared Absorption ⟨197⟩:** A thin film of it, cast from a melt onto a cesium bromide plate, exhibits a pair of very strong IR absorption peaks between 2840 cm^{-1} and 3000 cm^{-1}, a pair of moderately strong peaks between 1430 cm^{-1} and 1490 cm^{-1}, a pair of medium peaks between 720 cm^{-1} and 750 cm^{-1}, and only weak peaks at any other wavenumbers.

IMPURITIES

- **Heavy Metals,** *Method II* **⟨231⟩:** NMT 20 ppm
- **Limit of Oil Content**

  Analysis: Follow ASTM Method D721-68, "Standard Test Method for Oil Content of Petroleum Waxes" (Reapproved 1987).[1]

  Acceptance criteria: NMT 0.5%

SPECIFIC TESTS

- **Absorptivity**

  Sample solution: Transfer 50–100 mg to a 100-mL volumetric flask. Dissolve in decahydronaphthalene at 88°, dilute with the same solvent at this temperature to volume, and mix.

  Blank: Decahydronaphthalene

  Instrumental conditions

  (See *Spectrophotometry and Light-Scattering* ⟨851⟩.)

  Mode: UV

  Analytical wavelength: 290 nm

  Cell: 10 cm (jacketed cells maintained at 88°)

  Analysis

  Samples: *Sample solution* and *Blank*

  Determine the absorbance of the *Sample solution*, and calculate the absorptivity.

  Acceptance criteria: NMT 0.01

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.
- **Labeling:** The labeling indicates its congealing temperature, viscosity, and needle penetration range under the specified conditions.

Peanut Oil

[8002-03-7].

DEFINITION

Peanut Oil is the fully-refined (alkali-refined, bleached, and deodorized at 230°–260°) oil obtained from the seed kernels of one or more of the cultivated varieties of *Arachis hypogaea* Linné (Fam. Leguminosae). It may contain suitable antioxidants.

IDENTIFICATION

- It meets the requirements for *Fats and Fixed Oils, Fatty Acid Composition* ⟨401⟩.

IMPURITIES

Inorganic Impurities

- **Heavy Metals,** *Method II* **⟨231⟩:** NMT 10 ppm
- **Alkaline Impurities**

  Sample: 10 mL of Peanut Oil

  Analysis: Mix 10 mL of freshly opened acetone and 0.3 mL of water, and add 0.05 mL of bromophenol blue TS. Add the *Sample*, shake, and allow to stand. Titrate with 0.01 N hydrochloric acid VS to change the color of the upper layer to yellow.

  Acceptance criteria: NMT 0.1 mL of 0.01 N hydrochloric acid is required.

SPECIFIC TESTS

- **Fats and Fixed Oils,** *Fatty Acid Composition* **⟨401⟩:** Peanut Oil exhibits the following fatty acid composition profile.

Carbon-Chain Length	Number of Double Bonds	Percentage (%)
<14	0	≤0.1
14	0	≤0.2
16	0	7.0–16.0
16	1	≤1.0
18	0	1.3–6.5
18	1	35.0–72.0
18	2	13.0–43.0
18	3	≤0.6
20	0	0.5–3.0
20	1	0.5–2.1
22	0	1.0–5.0

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[1] Available from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.