Stearic Acid

Portions of this monograph that are national *USP* text, and are not part of the harmonized text, are marked with symbols (✦✦) to specify this fact.

Octadecanoic acid;
Stearic acid [57-11-4].

DEFINITION

Change to read:

▲Mixture consisting of stearic (octadecanoic) acid ($C_{18}H_{36}O_2$; M_r 284.5) and palmitic (hexadecanoic) acid ($C_{16}H_{32}O_2$; M_r 256.4) obtained from fats or oils of vegetable or animal origin.
Content:

Stearic acid 50	Stearic acid: 40.0%–60.0%. Sum of the contents of stearic acid and palmitic acids: NLT 90.0%.
Stearic acid 70	Stearic acid: 60.0%–80.0%. Sum of the contents of stearic and palmitic acids: NLT 90.0%.
Stearic acid 95	Stearic acid: NLT 90.0%. Sum of the contents of stearic acid and palmitic acids: NLT 96.0%

▲*NF30*
[NOTE—Stearic Acid labeled solely for external use is exempt from the requirement that it be prepared from edible sources.]

IDENTIFICATION

Add the following:

▲• **A.** It meets the requirements of the test for *Freezing Point*.▲*NF30*

Add the following:

▲• **B. ACID VALUE**
Light petroleum: Use a sample that has the following properties: a clear, colorless, liquid without fluorescence; practically insoluble in water; miscible with alcohol; density at 20° about 0.720; distillation range 100°– 120°; water content NMT 0.03%.[1]
Sample solution: Dissolve 0.5 g of Stearic Acid in 50 mL of a mixture of equal volumes of alcohol and *Light petroleum* previously neutralized with 0.1 N potassium hydroxide or 0.1 N sodium hydroxide, using 0.5 mL of phenolphthalein TS as indicator. If necessary, heat to about 90° to dissolve the substance to be examined.
Analysis: Titrate the *Sample solution* with 0.1 N potassium hydroxide or 0.1 N sodium hydroxide until the pink color persists for at least 15 s. When heating has been applied to aid dissolution, maintain the temperature at about 90° during the titration.
Calculate the acid value of the portion of Stearic Acid taken:

$$\text{Result} = I_A = n/m \times N \times 56.10$$

n = amount of titrant used (mL)
m = amount of Stearic Acid taken to prepare the *Sample solution* (g)
N = normality of the potassium hydroxide solution
56.10 = formula weight of potassium hydroxide
Acceptance criteria: 194–212▲*NF30*

Add the following:

▲• **C.** The retention times of the major peaks from the *Sample solution* correspond to those from the *Standard solution*, as obtained in the *Assay*.▲*NF30*

ASSAY

Delete the following:

▲• **PROCEDURE**
Standard: Place 50 mg of USP Stearic Acid RS and 50 mg of USP Palmitic Acid RS in a small conical flask fitted with a suitable reflux attachment.
Sample: Place 100 mg of Stearic Acid in a small conical flask fitted with a suitable reflux attachment.
Analysis: Treat each flask as follows. Add 5.0 mL of a solution prepared by dissolving 14 g of boron trifluoride in methanol to make 100 mL, swirl and reflux for 15 min or until the solid is dissolved. Cool, transfer the reaction mixture with the aid of 10 mL of chromatographic solvent hexane to a 60-mL separator, and add 10 mL of water and 10 mL of saturated sodium chloride solution. Shake, allow to separate, then drain and discard the lower, aqueous layer. Pass the hexane layer through 6 g of anhydrous sodium sulfate (previously washed with chromatographic solvent hexane) into a suitable flask. Using a syringe fitted with a suitable needle, introduce a 1-µL to 2-µL portion of the *Sample solution* (which contains the Stearic Acid).
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Mode: GC
Detector: Flame ionization
Column: 1.5-m × 3-mm, preferably glass; packed with 15% G4 on S1A
Carrier gas: Helium, passed through a bed of molecular sieve for drying, if necessary
Temperature
Column: 165°
Detector: 210°
Injector: 210°
System suitability
Samples: *Standard solution* and *Sample solution*
Suitability requirements
Resolution: NLT 2.0 between the methyl palmitate and methyl stearate peaks
[NOTE—Locate these peaks by comparison with the chromatogram of the *Standard solution*.]
Relative standard deviation: NMT 1.5% for methyl stearate and methyl palmitate peaks (from five replicate injections).
Analysis: Determine the percentage of $C_{18}H_{36}O_2$ in the portion of Stearic Acid taken:

$$\text{Result} = 100\ (A_S/A_T)$$

A_S = area due to the methyl stearate peak
A_T = sum of the areas of all of the fatty acid ester peaks in the chromatogram

[1] Petroleum ether; boiling range 100–140°; CAS 64742-49-0 from Fisher Scientific; catalog number AC23302-0025 is suitable.

Similarly, determine the percentage of $C_{16}H_{32}O_2$ in the portion of Stearic Acid taken:

$$\text{Result} = 100\ (A_P/A_T)$$

A_P = area due to the methyl palmitate peak
A_T = sum of the areas of all of the fatty acid ester peaks in the chromatogram

Acceptance criteria: NLT 40.0% of $C_{18}H_{36}O_2$, and the sum of the two is NLT 90.0%▲*NF30*

Add the following:

▲• **PROCEDURE**

Boron trifluoride-methanol solution: 140 g/L of boron trifluoride in methanol

Standard solution: Prepare as directed under *Sample solution* using 50 mg of USP Stearic Acid RS and 50 mg of USP Palmitic Acid RS.

Sample solution: Dissolve 100 mg of Stearic Acid in a small conical flask fitted with a suitable reflux attachment with 5 mL of *Boron trifluoride-methanol solution*. Boil under reflux for 10 min. Add 4.0 mL of heptane through the condenser and boil again under reflux for 10 min. Allow to cool. Add 20 mL of a saturated solution of sodium chloride. Shake, and allow the layers to separate. Remove about 2 mL of the organic layer and dry it over 0.2 g of anhydrous sodium sulfate. Dilute 1.0 mL of this solution with heptane to 10.0 mL.

Chromatographic system

(See *Chromatography* ⟨621⟩, *System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 30-m × 0.32-mm fused silica column coated with a 0.5-µm layer of stationary phase G16

Temperature

Injector: 220°

Detector: 260°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
70	—	70	2
70	5	240	5

Carrier gas: Helium, passed through a bed of molecular sieve for drying, if necessary

Flow rate: 2.4 mL/min

Injection size: 1 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Resolution: NLT 5.0 between methyl palmitate and methyl stearate peaks determined on 6 injections

Relative standard deviation: NMT 3.0% for methyl stearate and methyl palmitate peaks (from 6 replicate injections of *Sample solution*); NMT 1.0% for the ratio of the peak areas of methyl palmitate to the peak areas of methyl stearate, from 6 replicate injections

Analysis: Calculate the percentage of stearic acid ($C_{18}H_{36}O_2$) in the portion of sample taken:

$$\text{Result} = (A_S/A_T) \times 100$$

A_S = peak area due to methyl stearate
A_T = sum of the peak areas of all the fatty acid esters in the chromatogram

Similarly, calculate the percentage of palmitic acid ($C_{16}H_{32}O_2$) in the portion of sample taken:

$$\text{Result} = (A_P/A_T) \times 100$$

A_P = peak area due to methyl palmitate
A_T = sum of the peak areas of all the fatty acid esters in the chromatogram

Acceptance criteria

For Stearic Acid 50: 40.0–60.0% of $C_{18}H_{36}O_2$, and the sum of the stearic acid and palmitic acid is NLT 90.0%

For Stearic Acid 70: 60.0–80.0% of $C_{18}H_{36}O_2$, and the sum of the stearic acid and palmitic acid is NLT 90.0%.

For Stearic Acid 95: NLT 90.0% of $C_{18}H_{36}O_2$, and the sum of the stearic acid and palmitic acid is NLT 96.0%.▲*NF30*

IMPURITIES

• ✦**RESIDUE ON IGNITION** ⟨**281**⟩**:** NMT 4 mg, determined on a 4-g portion (0.1%)✦

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

SPECIFIC TESTS

Delete the following:

▲• **CONGEALING TEMPERATURE** ⟨**651**⟩**:** NLT 54°▲*NF30*

Change to read:

• **FATS AND FIXED OILS,** *Iodine Value* ⟨**401**⟩

▲**Sample:** 1 g

Analysis: Proceed as directed in *Method I* except to use 15 mL of chloroform.

Acceptance criteria: See *Table 2*.

Table 2

Type	Iodine Value
Stearic acid 50	NMT 4.0
Stearic acid 70	NMT 4.0
Stearic acid 95	NMT 1.5

▲*NF30*

Delete the following:

▲• **MINERAL ACID:** Shake 5 g of melted Stearic Acid with an equal volume of hot water for 2 min, cool, and filter: the filtrate is not reddened by the addition of 1 drop of methyl orange TS.▲*NF30*

Delete the following:

▲• **NEUTRAL FAT OR PARAFFIN**

Sample solution: 1 g of Stearic Acid in 30 mL anhydrous sodium carbonate solution (1 in 60)

Analysis: Boil the *Sample solution*.

Acceptance criteria: The resulting solution, while hot, shows NMT a faint opalescence.▲*NF30*

Add the following:

▲• **COLOR OF SOLUTION**

Standard stock solution Y (yellow): 2.4 mL of ferric chloride CS, 0.6 mL of cobaltous chloride CS, and 7.0 mL of hydrochloric acid solution (10 g/L)

Standard stock solution BY (brownish-yellow): 2.4 mL of ferric chloride CS, 1.0 mL of cobaltous chloride CS, 0.4 mL of cupric sulfate CS, and 6.2 mL of hydrochloric acid solution (10 g/L)

Standard solution Y: 2.5 mL of *Standard stock solution Y* and 97.5 mL of hydrochloric acid solution (10 g/L)

Standard solution BY: 2.5 mL of *Standard stock solution BY* and 97.5 mL of hydrochloric acid solution (10 g/L)

Analysis: Heat Stearic Acid to 75°.

Acceptance criteria: The resulting liquid is not more intensely colored than *Standard solution Y* or *Standard solution BY*.▲*NF30*

Add the following:

▲• **ACIDITY**

Analysis: Melt 5.0 g of Stearic Acid, shake for 2 min with 10 mL of hot carbon dioxide-free water, cool slowly, and filter. To the filtrate add 0.05 of methyl orange TS.

Acceptance criteria: No red color develops.▲*NF30*

Add the following:

▲• **FREEZING POINT**

Apparatus: Consists of a test tube about 25 mm in diameter and 150 mm long placed inside a test tube about 40 mm in diameter and 160 mm long. The inner tube is closed by a stopper which carries a thermometer about 175 mm long and graduated in 0.2°, fixed so that the bulb is about 15 mm above the bottom of the tube. The stopper has a hole allowing the passage of the stem of a stirrer made from a glass rod or other suitable material formed at one end into a loop of about 18 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported centrally in a 1-L beaker containing a suitable cooling liquid to within 20 mm of the top. A thermometer is supported in the cooling bath. Place in the inner tube sufficient quantity of the liquid or previously melted substance to be examined, to cover the thermometer bulb, and determine the approximate freezing point by cooling rapidly.

Analysis: Place the inner tube in a bath about 5° above the approximate freezing point until all but the last traces of crystals are melted. Fill the beaker with water or a saturated solution of sodium chloride, at a temperature about 5° lower than the expected freezing point, insert the inner tube into the outer tube, ensuring that some seed crystals are present, and stir thoroughly until solidification takes place. Note the highest temperature observed during solidification.

Acceptance criteria: See *Table 3*.

Table 3

Type	Freezing Point (°)
Stearic acid 50	53–59
Stearic acid 70	57–64
Stearic acid 95	64–69

▲*NF30*

ADDITIONAL REQUIREMENTS

- ✦**PACKAGING AND STORAGE:** Preserve in well-closed containers.✦

Change to read:

- **LABELING:** If it is for external use only, the labeling so indicates. ▲The label states the type of stearic acid (50, 70, or 95).▲*NF30*
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Palmitic Acid RS
  USP Stearic Acid RS