Calculate the content, in ppm, of sulfur dioxide in the *Sample* taken:

$$\text{Result} = 1000\ (32.03)\ VN/W$$

32.03 = milliequivalent weight of sulfur dioxide
V = volume of titrant consumed (mL)
N = normality of the titrant
W = weight of the *Sample* (g)

Acceptance criteria: NMT 50 ppm

- **LIMIT OF OXIDIZING SUBSTANCES**

Sample solution: Transfer 4.0 g to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 min. Transfer to a glass-stoppered, 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered, 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5 g to 1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25–30 min in the dark. Add 1 mL of starch TS.

Analysis: Titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch–iodine color. Perform a blank determination, and make any necessary correction. Each mL of 0.002 N sodium thiosulfate is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide.

Acceptance criteria: NMT 1.4 mL of 0.002 N sodium thiosulfate is required (20 ppm, calculated as H_2O_2).

SPECIFIC TESTS

- **✦MICROBIAL ENUMERATION TESTS** ⟨**61**⟩ and **TESTS FOR SPECIFIED MICROORGANISMS** ⟨**62**⟩**:** The total aerobic microbial count does not exceed 1000 cfu/g; the total combined molds and yeasts count does not exceed 100 cfu/g; and it meets the requirements of the test for the absence of *Escherichia coli*.✦
- **LOSS ON DRYING** ⟨**731**⟩**:** Dry 1 g at 130° for 90 min: it loses NMT 15.0% of its weight.
- **pH** ⟨**791**⟩**:** 4.5–7.0

Sample solution: Prepare a slurry by weighing 5.0 g of Wheat Starch, transferring to a suitable nonmetallic container, and adding 25.0 mL of freshly boiled and cooled water.

Analysis: Agitate continuously at a moderate rate for 1 min. Stop the agitation, and allow to stand for 15 min. Determine the pH to the nearest 0.1 unit.

- **TOTAL PROTEIN**

Analysis: Weigh 6.0 g of sample containing 2 mg of nitrogen; transfer to a combustion flask; add 4 g of a powdered mixture consisting of 100 g of potassium sulfate, 5 g of cupric sulfate, and 2.5 g of selenium; and add three glass beads. Wash any adhering particles from the neck into the flask with 5 mL of sulfuric acid, allowing it to run down the sides of the flask, and mix the contents by rotation. Close the mouth of the flask loosely, for example by means of a glass bulb with a short stem, to avoid excessive loss of sulfuric acid. Heat gradually at first, then increase the temperature until there is vigorous boiling with condensation of sulfuric acid in the neck of the flask; precautions should be taken to prevent the upper part of the flask from becoming overheated. Continue the heating for 30 min, unless otherwise prescribed. Cool, dissolve the solid material by cautiously adding to the mixture 25 mL of water, cool again, and place in a steam distillation apparatus. Add 30 mL of sodium hydroxide solution (42 in 100), and distill immediately by passing steam through the mixture. Collect 40 mL of distillate in 20.0 mL of 0.01 N hydrochloric acid and enough water to cover the tip of the condenser. Toward the end of the distillation, lower the receiver so that the tip of the condenser is above the surface of the acid. Take precautions to prevent any water on the outer surface of the condenser from reaching the contents of the receiver. Titrate the distillate with 0.01 N sodium hydroxide, using methyl purple TS as the indicator (n_1 mL of 0.01 N sodium hydroxide).

Repeat the test using 50 mg of glucose in place of the substance to be examined (n_2 mL of 0.01 N sodium hydroxide).

$$\text{Content of nitrogen} = [0.01401\ (n_2 - n_1)]/m$$

m = amount of test substance weighed (g)

Acceptance criteria: NMT 0.3% (corresponding to 0.048% N_2, conversion factor: 6.25)

ADDITIONAL REQUIREMENTS

- **✦PACKAGING AND STORAGE:** Preserve in well-closed containers. No storage requirements specified.✦

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.

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

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

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 in the *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 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 the methyl palmitate and methyl stearate peaks determined on 6 injections
Relative standard deviation: NMT 3.0% for the 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 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

Purified Stearic Acid

» Purified Stearic Acid is manufactured from fats and oils derived from edible sources and is a mixture of Stearic Acid ($C_{18}H_{36}O_2$) and palmitic acid ($C_{16}H_{32}O_2$), which together constitute not less than 96.0 percent of the total content. The content of $C_{18}H_{36}O_2$ is not less than 90.0 percent of the total.

NOTE—Purified Stearic Acid labeled solely for external use is exempt from the requirement that it be prepared from edible sources.

Packaging and storage—Preserve in well-closed containers.

Labeling—If it is for external use only, the labeling so indicates.

USP Reference standards ⟨11⟩**—**
USP Stearic Acid RS
USP Palmitic Acid RS

Congealing temperature ⟨651⟩: between 66° and 69°.

Acid value ⟨401⟩: between 195 and 200, about 1 g, accurately weighed, being used.

Iodine value ⟨401⟩: not more than 1.5. Proceed as directed in *Method I* except to use 35 mL of chloroform.

Other requirements—It meets the requirements for *Residue on ignition*, *Heavy metals*, *Mineral acid*, *Neutral fat or paraffin*, and *Assay* under *Stearic Acid*.

Stearoyl Polyoxylglycerides

Former Title: Stearoyl Macrogolglycerides

» Stearoyl Polyoxylglycerides is a mixture of monoesters, diesters, and triesters of glycerol and monoesters and diesters of polyethylene glycols. The polyethylene glycols used have a mean molecular weight between 300 and 4000. It is produced by partial alcoholysis of saturated oils, mainly containing triglycerides of stearic acid, with polyethylene glycol, by esterification of glycerol and polyethylene glycol with fatty acids, or as a mixture of glycerol esters and ethylene oxide condensate with the fatty acids of the hydrogenated oils. The Hydroxyl Value is not less than 85 percent and not more than 115 percent of the labeled nominal value, and the Saponification Value is not less than 90 percent and not more than 110 percent of the labeled nominal value. Stearoyl Polyoxylglycerides may contain free polyethylene glycols.

Packaging and storage—Preserve in tight containers, protected from light and moisture. Store at controlled room temperature.

Labeling—Label it to indicate the type and the average nominal molecular weight of polyethylene glycols used as part of the official title. The label also indicates the Hydroxyl Value and the Saponification Value.

USP Reference standards ⟨11⟩**—**
USP Stearoyl Polyoxylglycerides RS

Identification—

A: *Infrared Absorption* ⟨197K⟩.

B: *Thin-Layer Chromatographic Identification Test* ⟨201⟩—

Test solution: 50 mg per mL, in methylene chloride.

Standard solution: 50 mg of USP Stearoyl Polyoxylglycerides RS per mL, in methylene chloride.

Application volume: 10 µL.

Developing solvent system: a mixture of ether and hexanes (7:3).

Spray reagent—Prepare a 0.1 mg per mL solution of rhodamine B in alcohol.

Procedure—Proceed as directed in the chapter. Then spray the plate with *Spray reagent*, and locate the spots on the plate by examination under UV light at a wavelength of 365 nm: the R_F values of the principle spots obtained from the *Test solution* correspond to those obtained from the *Standard solution*.

C: It meets the requirements of the test for *Fatty acid composition*.

Acid value ⟨401⟩**:** not more than 2.0, determined on a 2.0-g specimen.

Hydroxyl value ⟨401⟩**—**The Hydroxyl Value, between 25 and 56, is not less than 85% and not more than 115% of the labeled nominal value, determined on a 1.0-g specimen, accurately weighed.

Iodine value ⟨401⟩**:** not more than 2.0.

Peroxide value ⟨401⟩**:** not more than 6.0, determined on a 2.0-g specimen.

Saponification value ⟨401⟩**—**The Saponification Value, between 67 and 112, is not less than 90% and not more than 110% of the labeled nominal value, determined on a 2.0-g specimen.

Fatty acid composition ⟨401⟩**:** Stearoyl Polyoxylglycerides exhibits the following composition profile of fatty acids, as determined in *Fatty Acid Composition* under *Fats and Fixed Oils* ⟨401⟩ (see *Table 1*).

Table 1

Carbon-Chain Length	Number of Double Bonds	Percentage (%)
12	0	≤5.0
14	0	≤5.0
16	0	40.0–50.0
18	0	48.0–58.0

Water, *Method I* ⟨921⟩**:** not more than 1.0%, determined on a 1.0-g specimen. Use as the solvent anhydrous pyridine or a mixture of methylene chloride and anhydrous methanol (7 : 3, v/v).

Total ash ⟨561⟩**:** not more than 0.2%.

Heavy metals, *Method II* ⟨231⟩**:** not more than 0.001%.

Alkaline impurities—Weigh 5.0 g of Stearoyl Polyoxylglycerides, heat slightly until the test substance melts, add 10 mL of alcohol and 0.05 mL of bromophenol blue TS, and mix well. While the solution is still warm, titrate with 0.01 N hydrochloric acid VS to change the color to yellow: not more than 1.0 mL of 0.01 N hydrochloric acid is required.

Limit of free ethylene oxide and dioxane—Proceed as directed in the test for *Limit of free ethylene oxide and dioxane* under *Caprylocaproyl Polyoxylglycerides:* not more than 1 µg of ethylene oxide per g is found; and not more than 10 µg of dioxane per g is found.

Limit of free glycerol—Proceed as directed in the test for *Limit of free glycerol* under *Caprylocaproyl Polyoxylglycerides:* not more than 5.0% is found.