Standard solutions: Prepare four solutions by dissolving glycerin in tetrahydrofuran, and diluting each with tetrahydrofuran as necessary, to obtain solutions having known concentrations of 0.4, 1.0, 2.0, and 4.0 mg/mL.
Standard curve
Samples: *Standard solutions*
Plot: Record the chromatograms, and measure the responses for the glycerin peaks. Plot the glycerin peak responses obtained versus the concentration, in mg/mL, of glycerin in the *Standard solutions*.
Analysis
Sample: *Sample solution*
From the *Standard curve*, determine the glycerin concentration, in mg/mL, in the *Sample solution*.
Calculate the percentage of free glycerin in the portion of Glyceryl Monooleate taken:

$$\text{Result} = (C_S/C_U) \times 100$$

C_S = concentration of glycerin in the *Sample solution* from the *Standard curve* (mg/mL)
C_U = concentration of Glyceryl Monooleate in the *Sample solution* (mg/mL)
Acceptance criteria: NMT 6.0%

SPECIFIC TESTS

- **FATS AND FIXED OILS,** *Acid Value* ⟨**401**⟩**:** NMT 6.0, determined on 1.0 g
- **FATS AND FIXED OILS,** *Iodine Value* ⟨**401**⟩**:** 65.0–95.0
- **FATS AND FIXED OILS,** *Peroxide Value* ⟨**401**⟩**:** NMT 12.0, determined on 2.0 g
- **FATS AND FIXED OILS,** *Saponification Value* ⟨**401**⟩**:** 150–175, determined on 2.0 g
- **FATS AND FIXED OILS,** *Fatty Acid Composition* ⟨**401**⟩**:** Glyceryl Monooleate exhibits the following composition profile of fatty acids (see *Table 1*), determined as directed in the chapter.

Table 1

Carbon-Chain Length	Number of Double Bonds	Percentage, NMT (%)
16	0	12.0
18	0	6.0
18	1	60.0
18	2	35.0
18	3	2.0
20	0	2.0
20	1	2.0

- **WATER DETERMINATION,** *Method I* ⟨**921**⟩**:** NMT 1.0%, using a mixture of methanol and methylene chloride (1:1) in place of methanol in the titration vessel
- **ARTICLES OF BOTANICAL ORIGIN,** *Total Ash* ⟨**561**⟩**:** NMT 0.1%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers. No storage requirements specified.
- **LABELING:** The labeling indicates the nominal content of monoglycerides and the name and the concentration of any added antioxidant.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Glyceryl Monooleate 40% RS
  USP Glyceryl Monooleate 90% RS

Glyceryl Monostearate

Octadecanoic acid, monoester with 1,2,3-propanetriol; Monostearin [31566-31-1].

DEFINITION

Glyceryl Monostearate contains NLT 90.0% of monoglycerides of saturated fatty acids, chiefly glyceryl monostearate ($C_{21}H_{42}O_4$) and glyceryl monopalmitate ($C_{19}H_{38}O_4$). It may contain a suitable antioxidant.

ASSAY

- **PROCEDURE**
Mobile phase: Tetrahydrofuran
Sample solution: 8 mg/mL of Glyceryl Monostearate in tetrahydrofuran
Chromatographic system
(See *Chromatography* ⟨621⟩*, System Suitability*.)
Mode: LC
Detector: Refractive index
Column: 7.5-mm × 60-cm; 5-µm 100-Å packing L21
Temperature: Column and detector temperatures are maintained at 40°.
[NOTE—Two or three 7.5-mm × 30-cm L21 columns may be used in place of the one 60-cm column, provided that system suitability requirements are met. The column temperature may be lowered to ambient temperature, although working at 40° provides stable separation conditions and ensures better sample solubility.]
Flow rate: 1 mL/min
Injection size: 40 µL
System suitability
Sample: *Sample solution*
[NOTE—The relative retention times for triglycerides, diglycerides, monoglycerides, and glycerin are 0.77, 0.81, 0.86, and 1.0, respectively.]
Suitability requirements
Relative standard deviation: NMT 2.0%, determined from the monoglycerides peak
Analysis
Sample: *Sample solution*
Calculate the percentage of monoglycerides in the portion of Glyceryl Monostearate taken:

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

r_U = peak response of the monoglycerides
r_T = sum of all the glyceride peak responses
Acceptance criteria: NLT 90.0% of monoglycerides of saturated fatty acids, chiefly $C_{21}H_{42}O_4$ and $C_{19}H_{38}O_4$

IMPURITIES

Inorganic Impurities
- **RESIDUE ON IGNITION** ⟨**281**⟩**:** NMT 0.5%
- **HEAVY METALS,** *Method II* ⟨**231**⟩**:** NMT 10 ppm

Organic Impurities
- **PROCEDURE: LIMIT OF FREE GLYCERIN**
Propionating reagent: Pyridine and propionic anhydride (1:2)
Internal standard solution: 0.2 mg/mL of tributyrin in chloroform
Standard solution: Transfer 15 mg of glycerin and 50 mg of tributyrin to a glass-stoppered, 25-mL conical flask. Add 3 mL of *Propionating reagent*, and heat at 75° for 30 min. Volatilize the reagents with the aid of a stream of nitrogen at room temperature, and add 12 mL of chloroform. Dilute 1 mL of this mixture with chloroform to 20 mL.
Sample solution: Transfer 50 mg of Glyceryl Monostearate to a glass-stoppered, 25-mL conical flask. Add 5 mL of *Internal standard solution* by pipet, and mix to dissolve. Immerse the flask in a water bath, maintained at a temperature between 45° and 50°, and volatilize the chloroform with the aid of a stream of nitrogen. Add 3 mL of *Propionating reagent*, and heat at 75° for 30 min. Volatilize the reagents with the aid of a stream of nitrogen at room temperature, and add 5 mL of chloroform.
Chromatographic system
(See *Chromatography* ⟨621⟩*, System Suitability*.)

Mode: GC
Detector: Flame ionization (under typical conditions)
Column: 4-mm × 2.4-m borosilicate glass, packed with 2% liquid phase G16, on 80- to 100-mesh support S1A
Temperature
Injector: 300°
Detector: 310°
Column: The column is maintained isothermally at a temperature between 190° and 200°.
Carrier gas: Helium
Flow rate: 70 mL/min
System suitability
Sample: *Standard solution* (6–10 injections)
Suitability requirements
Resolution: NLT 4.0 between derivatized glycerin and tributyrin
Relative standard deviation: NMT 2.0% of the ratio of their peak areas
Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the response factor, F:

$$F = (A_D/A_S) \times (W_S/W_D)$$

A_D = peak area of tributyrin from the *Standard solution*
A_S = peak area of tripropionin from the *Standard solution*
W_S = weight of glycerin in the *Standard solution* (mg)
W_D = weight of tributyrin in the *Standard solution* (mg)

Calculate the percentage of glycerin in the portion of Glyceryl Monostearate taken:

$$\text{Result} = (A_U/A_S) \times (W_D/W_U) \times F \times 100$$

A_U = peak area of tripropionin in the *Sample solution*
A_S = peak area of tributyrin in the *Sample solution*
W_D = weight of tributyrin in 5 mL of *Internal standard solution* (mg)
W_U = weight of Glyceryl Monostearate in the *Sample solution* (mg)

Acceptance criteria: NMT 1.2%

SPECIFIC TESTS
- **Melting Range or Temperature,** *Class I* ⟨**741**⟩**:** Does not melt below 55°
- **Fats and Fixed Oils,** *Acid Value* ⟨**401**⟩**:** NMT 6
- **Fats and Fixed Oils,** *Hydroxyl Value* ⟨**401**⟩**:** 290–330
- **Fats and Fixed Oils,** *Iodine Value* ⟨**401**⟩**:** NMT 3
- **Fats and Fixed Oils,** *Saponification Value* ⟨**401**⟩**:** 150–165

ADDITIONAL REQUIREMENTS
- **Packaging and Storage:** Preserve in tight, light-resistant containers.
- **Labeling:** Label it to indicate the name and quantity of any added antioxidant.

Glycine—*see Glycine General Monographs*

Guar Gum

DEFINITION
Guar Gum is a gum obtained from the ground endosperms of *Cyamopsis tetragonolobus* (Linné) Taub. (Fam. Leguminosae). It consists chiefly of a high molecular weight hydrocolloidal polysaccharide, composed of galactan and mannan units combined through glycosidic linkages, which may be described chemically as a galactomannan.

IDENTIFICATION
- **A.**
Sample: 2 g
Analysis: Place the *Sample* in a 400-mL beaker, and moisten it with 4 mL of isopropyl alcohol. Add 200 mL of cold water with vigorous stirring, and continue stirring until the *Sample* is completely and uniformly dispersed.
Acceptance criteria: An opalescent, viscous solution results.
- **B.**
Analysis: Transfer 100 mL of the solution prepared in *Identification* test *A* to a 400-mL beaker, heat in a boiling water bath for about 10 min, and cool.
Acceptance criteria: No appreciable increase in viscosity is produced. (Distinction from locust bean gum: see *Reagents, Indicators, and Solutions—Reagents*).

ASSAY
- **Content of Galactomannans**
Analysis: Subtract from 100.0 the total percentages from the tests for *Articles of Botanical Origin, Total Ash; Acid-Insoluble Matter; Protein;* and *Loss on Drying*.
Acceptance criteria: NLT 66.0%

IMPURITIES
- **Arsenic,** *Method II* ⟨**211**⟩**:** NMT 3 ppm
- **Lead** ⟨**251**⟩
Analysis: Prepare a *Test Preparation* as directed in the chapter, and use 10 mL of *Diluted Standard Lead Solution* (10 µg of Pb) for the test.
Acceptance criteria: NMT 10 ppm
- **Heavy Metals,** *Method II* ⟨**231**⟩**:** NMT 20 ppm

SPECIFIC TESTS
- **Articles of Botanical Origin,** *Total Ash* ⟨**561**⟩**:** NMT 1.5%
- **Acid-Insoluble Matter**
Sample: 1.5 g
Analysis: Transfer the *Sample* to a 250-mL beaker containing 150 mL of water and 1.5 mL of sulfuric acid. Cover the beaker with a watch glass, and heat the mixture on a steam bath for 6 h, rubbing down the wall of the beaker frequently with a rubber-tipped stirring rod, and replacing any water lost by evaporation. At the end of the 6-h heating period, add 500 mg of a suitable filter aid, and pass through a suitable tared, ashless filter. Wash the residue several times with hot water, dry the filter and its contents at 105° for 3 h, cool in a desiccator, and weigh. Determine the amount of acid-insoluble matter by subtracting the weight of the filter aid from that of the residue.
Acceptance criteria: NMT 7.0%
- **Protein**
Sample: 1.0 g
Analysis: Transfer the *Sample* to a 500-mL Kjeldahl flask, and proceed as directed in *Nitrogen Determination* ⟨461⟩, *Method I*. Determine the percentage of nitrogen. Calculate the amount of protein by multiplying the percentage of nitrogen by 6.25.
Acceptance criteria: NMT 10.0%
- **Starch**
Analysis: To a solution (1 in 10) of Guar Gum add a few drops of iodine TS.
Acceptance criteria: No blue color is produced.
- **Loss on Drying** ⟨**731**⟩**:** Dry a sample at 105° for 5 h: it loses NMT 15.0% of its weight.