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Glyceryl Distearate

Change to read:

▲▲ (CN 1-Aug-2024)

DEFINITION

Glyceryl Distearate is a mixture of diglycerides, mainly glyceryl distearate, together with variable quantities of monoglycerides and triglycerides. It contains NLT 8.0% and NMT 22.0% of monoglycerides, NLT 40.0% and NMT 60.0% of diglycerides, and NLT 25.0% and NMT 35.0% of triglycerides. It is obtained by partial glycerolysis of vegetable oil that consists mainly of triglycerides of palmitic or stearic acid or by esterification of glycerol with stearic acid. The fatty acids may be of vegetable or animal origin.

IDENTIFICATION

- **A.** It meets the requirements in *Specific Tests* for *Melting Range or Temperature*, *Class II*⟨741⟩.
- **B.** **Thin-Layer Chromatographic Identification Test** ⟨201⟩

Standard solution: 50 mg/mL of USP Glyceryl Distearate RS in methylene chloride
Sample solution: 50 mg/mL in methylene chloride
Chromatographic system
Developing solvent system: Ether and hexane (70:30)
Spray reagent: 0.1 mg/mL of rhodamine B in alcohol
Analysis: Proceed as directed in the chapter. Spray with the *Spray reagent*, and locate the spots on the plate by examination under UV light at a wavelength of 365 nm.
Acceptance criteria: The principal spot of the *Sample solution* corresponds in color, size, and R_F value to that of the *Standard solution*.

ASSAY

- **Procedure**

Mobile phase: Tetrahydrofuran
Sample solution: 40 mg/mL of Glyceryl Distearate 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.
[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.]
Temperatures
Detector: 40°
Column: 40°
Flow rate: 1 mL/min
Injection volume: 40 µL
System suitability
Sample: *Sample solution*
[Note—The relative retention times for triglycerides, diglycerides, monoglycerides, and glycerin are 0.75, 0.78, 0.84, and 1.0, respectively.]
Suitability requirements
Resolution: NLT 1.0 between the diglycerides and monoglycerides peaks
Relative standard deviation: NMT 2.0%, determined from the monoglycerides peak

Analysis

Sample: *Sample solution*

Calculate the percentage of monoglycerides, diglycerides, and triglycerides in the portion of Glyceryl Distearate taken:

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

r_U = individual peak response for the monoglycerides, diglycerides, and triglycerides, as appropriate

r_T = sum of the responses for all of the glyceride peaks

Acceptance criteria

Monoglycerides: 8.0%–22.0%

Diglycerides: 40.0%–60.0%

Triglycerides: 25.0%–35.0%

IMPURITIES

• **Limit of Nickel**

[**Caution—**When using closed high-pressure digestion vessels and microwave laboratory equipment, be familiar with the safety and operating instructions given by the manufacturer.]

Magnesium nitrate solution: 5 mg/mL of magnesium nitrate

Standard stock solution: Transfer 5.0 mL of nickel standard solution TS to a 10-mL volumetric flask. Add 0.5 mL of nitric acid and 1.0 mL of 30% hydrogen peroxide, and dilute with water to volume.

Standard solutions: Into four identical 25-mL volumetric flasks, each containing 6 mL of nitric acid, transfer 20, 50, 100, and 150 µL, respectively, of the *Standard stock solution*, and dilute with water to volume. These solutions contain 4, 10, 20, and 30 ng/mL of nickel, respectively.

Sample solution: Transfer 0.1 g of Glyceryl Distearate into a suitable high-pressure resistant digestion vessel (fluoropolymer or quartz), and add 6.0 mL of nitric acid and 2.0 mL of 30% hydrogen peroxide. Place the closed vessel in a laboratory microwave oven, and digest using an appropriate program (for example, 250 W for 10 min; 600 W for 5 min; 400 W for 5 min; and 250 W for 7 min). Allow the digestion vessel to cool before opening. Quantitatively transfer the contents to a 25-mL volumetric flask, and dilute with water to volume.

Blank: Add 6.0 mL of nitric acid and 2.0 mL of 30% hydrogen peroxide to a high-pressure-resistant digestion vessel, and proceed as directed for the *Sample solution*.

Zero solution: Transfer 6.0 mL of nitric acid into a 25-mL volumetric flask. Dilute with water to volume.

Instrumental conditions

(See *Atomic Absorption Spectroscopy ⟨852⟩*.)

Mode: Atomic absorption spectrophotometry equipped with a pyrolytically coated tube with a platform

Analytical wavelength: 232 nm

Lamp: Nickel hollow-cathode

Graphite furnace parameters: Maintain the drying temperature of the furnace at 100° for 10 s after a 10-s ramp, the ashing temperature at 1400° for 10 s after a 20-s ramp, and the atomization temperature at 2500° for 5 s. Use the *Zero solution* to set the instrument to zero.

Analysis

Samples: Into seven separate 25-mL flasks, each containing 5.0 mL of *Magnesium nitrate solution*, transfer respectively 10.0 mL of each of the following: the *Sample solution*, the *Blank*, the four *Standard solutions*, and the *Zero solution*.

Concomitantly determine the absorbances of the *Samples* at least three times each. Record the average of the steady readings for each of the solutions. [Note—If necessary, dilute the *Sample solution* with the *Zero solution* to obtain a reading within the calibrated absorbance range.]

Acceptance criteria: NMT 1 µg/g

• **Limit of Free Glycerin**

Mobile phase, Sample solution, Chromatographic system, and **System suitability:** Proceed as directed in the *Assay*.

Standard solutions: 0.2, 0.4, 1.0, and 2.0 mg/mL of glycerin in tetrahydrofuran

Analysis

Samples: *Standard solutions* and *Sample solution*

Plot the glycerin peak responses versus the concentration of glycerin in the *Standard solutions*. From the standard curve so obtained, determine the glycerin concentration in the *Sample solution*.

Calculate the percentage of free glycerin in the portion of Glyceryl Distearate taken:

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

C = concentration of glycerin in the *Sample solution* from the standard curve (mg/mL)

C_U = concentration of Glyceryl Distearate in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 1.0%

SPECIFIC TESTS

- **Melting Range or Temperature, *Class II*⟨741⟩:** 50°–70°
- **Fats and Fixed Oils, *Acid Value*⟨401⟩**
  Sample: 1.0 g
  Analysis: Use a mixture of alcohol and toluene (1:1, v/v) as the solvent and with gentle heating.
  Acceptance criteria: NMT 6.0
- **Fats and Fixed Oils, *Fatty Acid Composition*⟨401⟩:** The fatty acid fraction of it contains NLT 40.0% of stearic acid, and the sum of the contents of palmitic and stearic acids is NLT 90.0%.
- **Fats and Fixed Oils, *Iodine Value*⟨401⟩:** NMT 3.0
- **Fats and Fixed Oils, *Saponification Value*⟨401⟩**
  Sample: 2.0 g
  Analysis: Carry out the titration with heating.
  Acceptance criteria: 165–195
- **Water Determination, *Method I*⟨921⟩**
  Analysis: Use pyridine in place of methanol in the titration vessel.
  Acceptance criteria: NMT 1.0%
- **Articles of Botanical Origin, *Total Ash*⟨561⟩:** NMT 0.1%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight containers. No storage requirements are specified.
- **USP Reference Standards ⟨11⟩**
  USP Glyceryl Distearate RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
GLYCERYL DISTEARATE	Documentary Standards Support	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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