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Polyglyceryl 3 Diisostearate

$R–O–(CH_2–CH(OR)–CH_2–O)_3–R$ R = H, or $CO–C_{17}H_{35}$-iso
1,2,3-Propanetriol, homopolymer, diisooctadecanoate;
Triglyceryl diisostearate
CAS RN®: 63705-03-3.

DEFINITION

Polyglyceryl 3 Diisostearate is a mixture of polyglyceryl diesters of mainly isostearic acid, obtained by esterification of polyglycerin and isostearic acid. The polyglycerin consists mainly of triglycerin.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197F**▲ (CN 1-May-2020)

• **B.** It meets the requirements of the test for *Content of Fatty Acids*.

ASSAY

• **Content of Fatty Acids**

0.5 N methanolic sodium hydroxide solution: Dissolve 20 g of sodium hydroxide in 50 mL of water, and mix. Cool to room temperature, and add 950 mL of methanol.

Boron trifluoride–methanol solution: Dissolve 14 g of boron trifluoride in methanol to make 100 mL, and mix well.[1]

Saturated sodium chloride solution: Dissolve about 375 g of sodium chloride in water to make 1000 mL.

Standard solution: Prepare the calibration ester mixture by mixing up each individual ester component (see *Table 1*). Dissolve 500 mg of the calibration ester mixture in *n*-heptane, and dilute with *n*-heptane to 50 mL.

Table 1

Component in the Calibration Ester Mixture	Composition (%)[a]
USP Methyl Myristate RS (C14:0)	7
USP Methyl Palmitate RS (C16:0)	70
USP Methyl Stearate RS (C18:0)	23

[a] Composition is proposed according to the relative composition of these three fatty acid groups in Polyglyceryl 3 Diisostearate.

Sample solution: Introduce 100 mg of Polyglyceryl 3 Diisostearate into a 25-mL conical flask, fitted with a suitable water-cooled reflux condenser and a magnetic stir bar. Add 2 mL of *0.5 N methanolic sodium hydroxide solution*, mix, and reflux for about 30 min. Add 2 mL of *Boron trifluoride–methanol solution* through the condenser, and reflux for about 30 min. Add 4 mL of *n*-heptane through the condenser, and reflux for 5 min. Cool, remove the condenser, add about 10 mL of *Saturated sodium chloride solution*, shake, add a quantity of *Saturated sodium chloride solution* to bring the upper layer up to the neck of the flask, and allow the layers to separate. Collect 2 mL of *n*-heptane layer (upper layer), wash with three quantities, each of 2 mL of water, and dry the *n*-heptane phase over anhydrous sodium sulfate.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m fused-silica capillary column, 0.25-µm layer of phase G16

Temperature

Detector: 250°

Injection port: 240°

Column: See the temperature program table below.

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
150	6	250	6

Carrier gas: Nitrogen

Flow rate: 1.0–1.2 mL/min

Injection size: 1 µL

Injection type: Split injection. Split ratio is about 1:80.

System suitability

Sample: *Standard solution*

[NOTE—See the relative retention time table below.]

Name	Relative Retention Time
Methyl myristate	1.0
Methyl palmitate	1.4
Methyl stearate	1.8

Suitability requirements

Resolution: NLT 10 between the peaks due to methyl palmitate and methyl stearate

Relative standard deviation: NMT 6.0% for the peak responses for palmitate and stearate

Analysis

Samples: *Standard solution* and *Sample solution*

Identify the fatty acid ester peaks in the chromatogram of the *Sample solution* by comparing the retention times of these peaks with those obtained in the chromatogram of the *Standard solution*, and measure the peak areas for all of the fatty acid ester peaks in the chromatogram obtained from the *Sample solution*.

Calculate the percentage of each fatty acid ester component in the test specimen:

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

r_U = peak response for each individual fatty acid ester component

r_T = sum of the peak responses, excluding the solvent peak, in the chromatogram obtained from the *Sample solution*

Acceptance criteria

Sum of the contents of the fatty acids eluting between palmitic acid and stearic acid (excluding palmitic acid and stearic acid): NLT 60.0%

Sum of the contents of myristic acid, palmitic acid, and stearic acid: NMT 11.0%

IMPURITIES

Inorganic Impurities

- **Residue on Ignition**

Analysis: Heat a silica crucible to redness for 30 min, allow to cool in a desiccator, and weigh. Evenly distribute about 1.0 g of Polyglyceryl 3 Diisostearate in the crucible and weigh. Dry at 100°–105° for 1 h, and ignite in a muffle furnace at 600 ± 25°, until the test substance is thoroughly charred. Perform the test for *Residue on Ignition* ⟨281⟩ on the residue obtained, starting with "Moisten the sample with a small amount (usually 1 mL) of sulfuric acid".

Acceptance criteria: NMT 0.5%

SPECIFIC TESTS

• **Acid Value**

Analysis: Accurately weigh (to within 0.1 mg) 5–10 g of Polyglyceryl 3 Diisostearate, add 10 mL of alcohol and 3 drops of phenolphthalein TS, and titrate with 0.1 N potassium hydroxide VS or 0.1 N sodium hydroxide VS until the solution remains faintly pink after shaking for 30 s. Follow the procedures for *Fats and Fixed Oils, Acid Value* ⟨401⟩ to perform the calculation.

Acceptance criteria: NMT 3.0

• **Fats and Fixed Oils,** *Hydroxyl Value*⟨401⟩**:** 180–230, determined on a 0.25-g specimen

• **Iodine Value**

Analysis: Accurately weigh 3 g of Polyglyceryl 3 Diisostearate, transfer to a dry 250-mL flask with a ground-glass stopper, and add 25 mL of methylene chloride. Add 20 mL of the Wijs' solution.[2] Close the flask, and keep it in the dark for 1 h while shaking frequently. Perform the test in *Fats and Fixed Oils* ⟨401⟩ *Iodine Value*, starting with "Then add, in the order named, 30 mL of potassium iodide TS and 100 mL of water".

Acceptance criteria: NMT 3.0

• **Fats and Fixed Oils,** *Peroxide Value*⟨401⟩**:** NMT 6.0. Use 30 mL of a mixture of glacial acetic acid and methylene chloride (3:2) to replace the 30 mL of a mixture of glacial acetic acid and chloroform (3:2).

• **Fats and Fixed Oils,** *Saponification Value*⟨401⟩**:** 128–160

• **Water Determination,** *Method I*⟨921⟩**:** NMT 0.5%, determined on a 2.0-g specimen

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers, protected from heat and moisture.

• **USP Reference Standards** ⟨11⟩

USP Methyl Myristate RS
USP Methyl Palmitate RS
USP Methyl Stearate RS
USP Polyglyceryl 3 Diisostearate RS

[1] Boron trifluoride–methanol solution (14% in methanol) is also commercially available from Sigma, B-1252, or equivalent quality.

[2] Wijs' reagent RPE for analysis from Carlo Erba Reference 491902, Wijs' solution from www.sigmaaldrich.com, or equivalent quality.

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

Topic/Question	Contact	Expert Committee
POLYGLYCERYL 3 DIISOSTEARATE	Documentary Standards Support	CE2020 Complex Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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