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Monoglyceride Citrate

Citric acid ester of glyceryl monooleate

CAS RN®: 36291-32-4.

DEFINITION

Monoglyceride Citrate is a mixture of glyceryl monooleate and its citric acid monoester, manufactured by the reaction of glyceryl monooleate with citric acid under controlled conditions. It contains NLT 14.0% and NMT 17.0% of total citric acid, calculated on the anhydrous basis.

IDENTIFICATION

- **A.**

Sample: 1 g

Analysis: Reflux the *Sample* with 15 mL of 0.5 N potassium hydroxide solution in dehydrated alcohol for 1 h. Add 15 mL of water, and acidify with diluted hydrochloric acid (about 6 mL). Dissolve any oil drops or solid produced in 5 mL of hexane. Remove the hexane layer, extract again with 5 mL of hexane, and again remove the hexane layer.

[Note—Keep the resulting aqueous layer for *Identification* tests *B* and *C*.]

Acceptance criteria: Oil drops or a white to yellowish-white solid are produced that are soluble in 5 mL of hexane.

- **B.** Identification Tests—General, *Citrate* ⟨191⟩

Sample: 1 mL of the aqueous layer resulting from *Identification* test *A*

Analysis: Evaporate the *Sample* in a porcelain dish.

Acceptance criteria: The residue meets the requirements.

- **C.**

Sample: 5 mL of the aqueous layer resulting from *Identification* test *A*

Analysis: Transfer the *Sample* to a test tube. Add excess calcium hydroxide as a powder, place in boiling water for 5 min, shaking several times, cool, and filter. Transfer one drop of the filtrate into a test tube, and add about 50 mg of potassium hydrogen sulfate. On top of the test tube, place a filter paper moistened with a reagent for acrolein consisting of a mixture of 5% nitroprusside solution in water and 20% piperidine solution in water (1:1). Heat the test tube.

Acceptance criteria: The filter paper turns blue (presence of glycerin). The color changes to light red after addition of sodium hydroxide TS.

ASSAY

- **Content of Citric Acid**

Standard solution: 0.23 mg/mL of USP Citric Acid RS

Sample solution: Transfer 150 mg of Monoglyceride Citrate into a saponification flask, add 50 mL of 4% potassium hydroxide solution in dehydrated alcohol, and reflux for 1 h. Acidify the reaction mixture with hydrochloric acid to a pH of 2.8–3.2, transfer into a 400-mL beaker, and evaporate to dryness on a steam bath. Quantitatively transfer the contents of the beaker into a separator, using NMT 50 mL of water, and extract with three 50-mL portions of petroleum ether, discarding the extracts. Transfer the water layer to a 100-mL volumetric flask, and dilute with water to volume.

Blank: Water

Instrumental conditions

Mode: UV-Vis

Analytical wavelength: 450 nm

Cell: 1 cm

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*

Pipet 2.0 mL each of the *Standard solution*, *Sample solution*, and *Blank* into separate 40-mL graduated centrifuge tubes. Add 2 mL of a 1 in 2 sulfuric acid solution and 11 mL of water to each tube. Boil for 3 min, cool, and add 5 mL of bromine TS to each tube. Dilute to 20 mL,

allow to stand for 10 min, and centrifuge. Transfer 4.0 mL of the supernatant from each tube into separate 19- × 110-mm test tubes, add 1 mL of water, 0.5 mL of a 1 in 2 sulfuric acid solution, and 0.3 mL of 1 M potassium bromide, and shake. Add 0.3 mL of 1.5 N potassium permanganate, shake, and allow to stand for 2 min. Add 1 mL of a saturated solution of ferrous sulfate, shake, allow to stand for 2 min, and then dilute with water to 10 mL. Add 10.0 mL of *n*-hexane (previously washed with sulfuric acid, followed by a water wash, and then dried over anhydrous sodium sulfate), shake vigorously for 2 min, and centrifuge at low speed for 1 min. Transfer 5.0 mL of the hexane extract into a 20- × 145-mm tube containing 10.0 mL of 4% sodium sulfide solution, and briefly shake vigorously (three oscillations only). Centrifuge the mixture at low speed for 1 min. Immediately determine the absorbance of each aqueous layer from the *Standard solution* and *Sample solution* against the aqueous layer from the *Blank*.

Calculate the percentage of citric acid in the portion of Monoglyceride Citrate taken:

$$\text{Result} = (A_U/A_S) \times (V \times C_S/W) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

V = volume of the *Sample solution* (mL)

C_S = concentration of USP Citric Acid RS in the *Standard solution* (mg/mL)

W = weight of Monoglyceride Citrate taken to prepare the *Sample solution* (mg)

Acceptance criteria: 14.0%–17.0% on the anhydrous basis

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.3%, determined on 1 g

SPECIFIC TESTS

• **Fats and Fixed Oils, *Acid Value*⟨401⟩:** 70–100

• **Fats and Fixed Oils, *Saponification Value*⟨401⟩:** 260–265

• **Water Determination, *Method I*⟨921⟩:** NMT 0.2%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers. No storage requirements specified.

• **USP Reference Standards ⟨11⟩**
USP Citric Acid RS

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

Topic/Question	Contact	Expert Committee
MONOGLYCERIDE CITRATE	Documentary Standards Support	SE2020 Simple Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

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