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Sodium Stearyl Fumarate

DEFINITION

Sodium Stearyl Fumarate contains NLT 99.0% and NMT 101.5% of sodium stearyl fumarate ($C_{22}H_{39}NaO_4$), calculated on the anhydrous basis.

IDENTIFICATION

• **Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197K**

Analysis: Perform test on an undried specimen (1 in 300).

Acceptance criteria: Meets the requirements

ASSAY

• **Procedure**

Sample: 250 mg

Blank: 10 mL of chloroform

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.1 N perchloric acid VS

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a 50-mL conical flask, mix with 10 mL of chloroform, and add 20 mL of glacial acetic acid to dissolve. Add quinaldine red TS, and titrate with 0.1 N perchloric acid VS.

Calculate the percentage of sodium stearyl fumarate ($C_{22}H_{39}NaO_4$) in the *Sample* taken:

$$\text{Result} = \{[(V_S - V_B) \times N \times F]/W\} \times 100$$

V_S = volume of *Titrant* consumed by the *Sample* (mL)

V_B = volume of *Titrant* consumed by the *Blank* (mL)

N = actual normality of the *Titrant* (mEq/mL)

F = equivalency factor, 390.5 mg/mEq

W = weight of the *Sample* (mg)

Acceptance criteria: 99.0%–101.5% on the anhydrous basis

OTHER COMPONENTS

• **Limit of Sodium Stearyl Maleate and Stearyl Alcohol**

Solvent: Chloroform and glacial acetic acid (4:1)

Standard monostearyl maleate stock solution: 1 mg/mL of USP Monostearyl Maleate RS in *Solvent*

Standard monostearyl maleate solution: Pipet 5.0 mL of *Standard monostearyl maleate stock solution*, and dilute with chloroform to 50 mL.

Standard stearyl alcohol stock solution: 1 mg/mL of USP Stearyl Alcohol RS in *Solvent*

Standard stearyl alcohol solution: Pipet 5.0 mL of *Standard stearyl alcohol stock solution*, and dilute with chloroform to 50 mL.

Sample solution: Transfer 200 mg of Sodium Stearyl Fumarate to a small, glass-stoppered conical flask. Add 10.0 mL of *Solvent*. Dissolve by placing the flask in an ultrasonic bath for 10 min.

Chromatographic system

(See *Chromatography* ⟨621⟩, *Thin-Layer Chromatography*.)

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel (TLC plates)

Spray reagent: Sulfuric acid and alcohol (1:9). [Note—Add cautiously and with stirring.]

Developing solvent system: Hexanes, toluene, and glacial acetic acid (5:5:1)

Analysis

Samples: *Standard monostearyl maleate solution*, *Standard stearyl alcohol solution*, and *Sample solution*

Apply 5 µL of *Standard monostearyl maleate solution* and 10 µL each of *Standard stearyl alcohol solution* and *Sample solution* to the plate. Immerse the plate in a tank containing a layer of 10 mm of chloroform on the bottom. Allow the solvent front to reach the upper edge of the spots. Withdraw the plate, and dry in a current of cold air. Repeat the immersion, development, and drying. This results in spots having a linear shape. Develop the chromatograph in a saturated chamber containing the *Developing solvent system* until the solvent front has moved 15 cm, and remove the plate from the chamber. Allow to dry for 10 min, and heat in an oven at 90° for 2 min. Allow to cool to room temperature. Replace the plate in the chamber for another 15-cm development, remove the plate, and allow to dry at room temperature for 15 min. Spray the plate with *Spray reagent*. Dry the plate in an oven at 150° for 15 min. Dark spots appear on a light background. Allow to cool. Faint spots at an R_F value of 0.9 may result from traces of distearyl maleate and distearyl fumarate.

Acceptance criteria: The intensity of any spot from the *Sample solution* is not greater than that from the corresponding spot from the *Standard monostearyl maleate solution* and *Standard stearyl alcohol solution* (NMT 0.25% sodium stearyl maleate, NMT 0.5% stearyl alcohol).

IMPURITIES

Change to read:

• ▲Lead ⟨251⟩, *Procedures, Procedure 1*▲ (CN 1-Jun-2023) : NMT 10 ppm

SPECIFIC TESTS

• **Water Determination,** *Method I* ⟨921⟩**:** NMT 5.0%

• **Fats and fixed Oils,** *Saponification Value* ⟨401⟩

Sample: 450 mg

Ethanolic potassium hydroxide: Dissolve 5.5 g of potassium hydroxide in absolute alcohol, heating if necessary to dissolve, and dilute with absolute alcohol to about 1000 mL. Prepare fresh daily, and filter if necessary to remove carbonate.

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Residual titration

Titrant: 0.1 N hydrochloric acid VS

Blank: 50.0 mL of *Ethanolic potassium hydroxide*

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a 300-mL conical flask, and add 50.0 mL of *Ethanolic potassium hydroxide*, rinsing down the inside of the flask during the addition. Gently reflux the mixture on a steam bath for NLT 2 h, occasionally swirl gently, but avoid splashing the mixture up into the condenser. Rinse the condenser with 10 mL of 70% alcohol, followed by three 10-mL portions of water, collecting the rinsings in the flask. Cool, rinse the sides of the flask with two 10-mL portions of 70% alcohol, add phenolphthalein TS, and titrate with 0.1 N hydrochloric acid VS to the disappearance of any pink color. Perform a blank determination.

Calculate the *Saponification Value* for Sodium Stearyl Fumarate in the *Sample* taken:

$$\text{Result} = [(V_B - V_S) \times N \times F]/W$$

V_B = volume of the *Titrant* consumed by the *Blank* (mL)

V_S = volume of the *Titrant* consumed by the *Sample* (mL)

N = actual normality of the *Titrant* (mEq/mL)

F = molecular weight of potassium hydroxide, 56.11

W = weight of the *Sample* (g)

Acceptance criteria: 142.2–146.0, calculated on the anhydrous basis

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

• **USP Reference Standards** ⟨11⟩

USP Monostearyl Maleate RS

USP Sodium Stearyl Fumarate RS

USP Stearyl Alcohol RS

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

Topic/Question	Contact	Expert Committee
SODIUM STEARYL FUMARATE	Documentary Standards Support	CE2020 Complex Excipients

Topic/Question	Contact	Expert Committee
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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