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## Saw Palmetto Capsules

### DEFINITION

Saw Palmetto Capsules contain Saw Palmetto Extract. Capsules contain NLT 22.0% and NMT 34.0% of lauric acid in the labeled amount of Saw Palmetto Extract. The ratio of the concentrations of lauric acid to caprylic acid is NLT 8.5 and NMT 17.5. The ratio of the concentrations of lauric acid to myristic acid is NLT 2.2 and NMT 2.8.

### IDENTIFICATION

• **A.** The retention times of the peaks for methyl caprate, methyl caproate, methyl caprylate, methyl laurate, methyl linoleate, methyl linolenate, methyl myristate, methyl oleate, methyl palmitate, methyl palmitoleate, and methyl stearate of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the test for *Content of Lauric Acid and the Ratios of the Concentrations of Lauric Acid to Caprylic Acid and Lauric Acid to Myristic Acid*.

**Change to read:**

• **B. PRESENCE OF STEROLS**

**Derivatizing stock solution:** *N,O*-[bis\(trimethylsilyl\)-acetamide](#), [trimethylsilylimidazole](#), and [trimethylchlorosilane](#) (3:3:2)

**Derivatizing solution:** *Derivatizing stock solution*, [bis\(trimethylsilyl\)trifluoroacetamide](#), and [pyridine](#) (1:1:1)

**Internal standard solution:** 10 mg/mL of [eicosanol](#) and 5 mg/mL of [cholesterol](#) in [chloroform](#)

**System suitability stock solution A:** 2 mg/mL each of tetracosanol, octacosanol, [USP Hexacosanol RS](#), and triacontanol in [chloroform](#)

**System suitability solution A:** Mix 5.0 mL of *System suitability stock solution A* with 1.0 mL of *Internal standard solution*. Evaporate 0.75 mL of this solution to dryness using a stream of nitrogen. Dissolve the residue in 1.0 mL of *Derivatizing solution*, and allow to stand for NLT 15 min at room temperature.

**System suitability stock solution B:** 0.37 mg/mL of stigmasterol and 2 mg/mL each of campesterol, stigmasterol, and [USP  \$\beta\$ -Sitosterol RS](#) in [chloroform](#)

**System suitability solution B:** Mix 5.0 mL of *System suitability stock solution B* with 1.0 mL of *Internal standard solution*. Evaporate 0.75 mL of this solution to dryness using a stream of nitrogen. Dissolve the residue in 1.0 mL of *Derivatizing solution*, and allow to stand for NLT 15 min at room temperature.

**$\beta$ -Cholestanol solution:**  [\$\beta\$ -Cholestanol](#) in [chloroform](#) (1 in 100)

**Standard stock solution:** 0.75 mg/mL of [USP Hexacosanol RS](#) and 1.4 mg/mL of [USP  \$\beta\$ -Sitosterol RS](#) in [chloroform](#)

**Standard solution:** Mix 5.0 mL of *Standard stock solution* with 1.0 mL of the *Internal standard solution*. Evaporate 0.75 mL of this solution to dryness using a stream of nitrogen. Dissolve the residue in 1.0 mL of *Derivatizing solution*, and allow to stand for NLT 15 min at room temperature.

**Sample solution:** Open a number of Capsules, equivalent to 10 g of Saw Palmetto Extract, and transfer the shells and contents to a suitable container. Transfer 5 g of Saw Palmetto Extract to a 250-mL round-bottom flask, and evaporate under vacuum at a temperature of NMT 50°. Add 50 mL of a solution prepared by dissolving 130 mg/mL of [potassium hydroxide](#) in [methanol](#) and [water](#) (4:1). Attach a condenser, and reflux in a bath at 100° until a clear solution is obtained. Reflux for an additional 10 min, and cool by adding 50 mL of water through the condenser. Transfer to a separation funnel, rinsing the flask with a total of 50 mL of [water](#) in small portions. Extract with 80 mL of [ether](#), shaking for 30 s, and repeat twice. [NOTE—If an emulsion forms, it can be eliminated by adding small quantities of [methanol](#).] Transfer the combined ether layers to a separation funnel, and wash with successive portions of 50 mL of [water](#) until a neutral washing is obtained. [NOTE—If an emulsion forms, it can be eliminated by adding small quantities of [methanol](#).] Pass the ether extract through filter paper containing [anhydrous sodium sulfate](#), wash the filter with 30 mL of [ether](#), and evaporate to dryness under vacuum. Dissolve the residue in 2.0 mL of [chloroform](#). Extract the sterols using the following *Chromatographic extraction system*.

### Chromatographic extraction system

**Mode:** TLC

**Adsorbent:** Chromatographic plate coated with a 0.25-mm layer of silica gel having an application zone that was previously dipped under 3 cm of a solution prepared by dissolving 13 mg/mL of potassium hydroxide in methanol and water (49:1)

**Developing solvent system:** Hexanes and ether (7:3)

**Application volumes:** 200  $\mu$ L of chloroform solution containing Capsule residue and 20  $\mu$ L of  $\beta$ -Cholestanol solution



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After the spots have been applied, allow the plate to dry, and heat it to 100° for 1 h before use. The plate can be stored in a desiccator containing [calcium chloride](#) until the time of use. Develop the plate until the solvent front has moved 17–19 cm. Keep the chamber temperature between 15° and 20°. Dry the plate in a current of warm air, then spray with an alkaline solution of [2,7-dichlorofluorescein](#) in [alcohol](#) (0.2 in 100). Observe the plate under 366-nm wavelength light, and identify the bands corresponding to the sterols by referring to the  $\beta$ -cholesterol spot. Scrape off these bands and transfer them to a test tube. Add 10 mL of warm [chloroform](#), and shake for 2 min with the aid of several glass beads. Filter the chloroform solution, wash the filter with [chloroform](#), and evaporate the combined filtrate and washings to dryness under vacuum. Dissolve the residue with some drops of [anhydrous acetone](#), and evaporate under vacuum. Dry the residue in an oven at 105° for 15 min. Dissolve the residue in 0.2 mL of *Derivatizing solution*. Use this resulting solution for GC analysis.

#### Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

**Mode:** GC

**Detector:** Flame ionization

**Column:** 0.2-mm × 25-m capillary; coated with a 0.33- $\mu$ m film of phase G1

#### Temperatures

**Injection port:** 325°

**Detector:** 325°

**Column:** See [Table 1](#).

Table 1

| Initial Temperature (°) | Temperature Ramp (°/min) | Final Temperature (°) | Hold Time at Final Temperature (min) |
|-------------------------|--------------------------|-----------------------|--------------------------------------|
| 200                     | 0                        | 200                   | 3                                    |
| 200                     | 10                       | 300                   | 35                                   |

**Carrier gas:** Helium

**Flow rate:** 0.5 mL/min

**Makeup gas flow:** 25 mL/min

**Injection volume:** 1  $\mu$ L

**Injection type:** Split; split ratio 1:40

#### System suitability

**Samples:** *System suitability solution A* and *System suitability solution B*

[NOTE—The relative retention times for tetracosanol,  $\blacktriangle$ hexacosanol, octacosanol,  $\blacktriangle$  (USP 1-Aug-2020) and triacontanol are 0.89, 1.00, 1.15, and 1.36, respectively, *System suitability solution A*; and the relative retention times for cholesterol, campesterol, stigmasterol,  $\beta$ -sitosterol, and stigmastanol are 0.85, 0.92, 0.95, 1.00, and 1.01, respectively, *System suitability solution B*.]

#### Suitability requirements

**Resolution:** NLT 2 between  $\beta$ -sitosterol and stigmastanol, *System suitability solution B*

**Column efficiency:** NLT 200,000 theoretical plates for the eicosanol peak, *System suitability solution A*; NLT 150,000 theoretical plates for the cholesterol peak, *System suitability solution B*

**Tailing factor:** NMT 2.0 for each relevant peak, *System suitability solution A*; NMT 2.0 for each relevant peak, *System suitability solution B*

#### Analysis

**Samples:** *System suitability solution A*, *System suitability solution B*, *Standard solution*, and *Sample solution*

Identify the signals corresponding to the relevant analytes by comparing the chromatograms of the *Standard solution* and the *Sample solution* with those of *System suitability solution A* and *System suitability solution B*.

**Acceptance criteria:** The *Sample solution* exhibits peaks for campesterol,  $\beta$ -sitosterol, and stigmasterol, identified by their retention times relative to the  $\beta$ -sitosterol peak from the *Standard solution*.

#### STRENGTH

• **CONTENT OF LAURIC ACID AND THE RATIOS OF THE CONCENTRATIONS OF LAURIC ACID TO CAPRYLIC ACID AND LAURIC ACID TO MYRISTIC ACID**

**Internal standard solution:** 12 mg/mL of [nonadecane](#) in [hexanes](#)

**Standard stock solution:** Dissolve quantities of [USP Methyl Laurate RS](#), [USP Methyl Oleate RS](#), [USP Methyl Myristate RS](#), [USP Methyl Palmitate RS](#), [USP Methyl Linoleate RS](#), [USP Methyl Caproate RS](#), [USP Methyl Caprylate RS](#), [USP Methyl Caprate RS](#), [USP Methyl](#)