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Pygeum Capsules

DEFINITION

Pygeum Capsules contain Pygeum Extract. Capsules contain NLT 90.0% and NMT 110.0% of the labeled amount of Extract, calculated as sterols and docosyl ferulate.

IDENTIFICATION

- **A.** The retention times of the peaks for campesterol, stigmasterol, and β -sitosterol, of the *Sample solution*, correspond to those of the *Standard solution*, as obtained in the test for *Content of Sterols*.
- **B.** The retention time of the peak for docosyl ferulate in the *Sample solution* corresponds to that in the *Standard solution*, as obtained in the test for *Content of Docosyl Ferulate*.

STRENGTH

• CONTENT OF STEROLS

Derivatizing solution: Bis(trimethylsilyl)acetamide and trimethylchlorosilane (9:1)

Internal standard solution: 2 mg/mL of 5 α -cholestane in chloroform

System suitability stock solution: 2 mg/mL each of campesterol, stigmasterol, and USP β -Sitosterol RS

System suitability solution: Mix 2.0 mL of the *System suitability stock solution* and 2.0 mL of the *Internal standard solution*, and dilute with chloroform to 10 mL. Evaporate 500 μ L of this solution to dryness using a stream of nitrogen. Dissolve the residue in 80 μ L of *Derivatizing solution* and 20 μ L of pyridine. Allow to stand for NLT 10 min at room temperature.

Standard stock solution: 2.0 mg/mL of USP β -Sitosterol RS in chloroform

Standard solution: Mix 2.0 mL of the *Standard stock solution* and 2.0 mL of the *Internal standard solution*, and dilute with chloroform to 10 mL. Evaporate 500 μ L of this solution to dryness using a stream of nitrogen. Dissolve the residue in 80 μ L of *Derivatizing solution* and 20 μ L of pyridine. Allow to stand for NLT 10 min at room temperature.

Sample solution: Transfer a quantity of Capsules, equivalent to 100 mg of the labeled amount of Extract, into a 100-mL round-bottomed flask. Add 2.0 mL of the *Internal standard solution* and 20 mL of diluted hydrochloric acid. Attach a condenser, and reflux in a bath at 100° for 30 min. Cool the solution to room temperature, and adjust by the addition of about 5 mL of 10 N sodium hydroxide to a pH of 8. Extract twice using 50 mL of ether each time, wash the collected organic phases with 50 mL of water, and evaporate the organic phase to dryness under vacuum. Dissolve the residue with 4 mL of chloroform, and transfer to a cartridge containing 500 mg of packing L8 that has been conditioned with a 2-column volume of *n*-hexane. [NOTE—A suitable cartridge is Chromabond NH2, manufactured by Macheray Nagel, or equivalent.] Collect the eluate. Elute twice with a 1-column volume of a mixture of chloroform and isopropanol (2:1). Combine the eluates, and evaporate to dryness. Dissolve the residue in 10 mL of chloroform. Evaporate 500 μ L of this solution to dryness under a stream of nitrogen. Dissolve the residue with 80 μ L of *Derivatizing solution* and 20 μ L of pyridine. Allow to stand for NLT 10 min at room temperature.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.32-mm $\times$ 30-m capillary column coated with a G27 phase of 0.25- μ m thickness

Temperature

Detector: 285°

Injector: 285°

Column: See the temperature program table below.

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
250	0	250	5
250	5	320	as needed

Carrier gas: Helium. [NOTE—The carrier gas flow rate should be adjusted to obtain a retention time of 19 min for β -sitosterol.]

Makeup gas: Helium

Injection size: 2 μ L

Injection type: Split injection system

Split ratio: 1:50

System suitability

Sample: *System suitability solution*

[NOTE—The relative retention times for 5α -cholestane, campesterol, stigmasterol, and β -sitosterol are about 0.66, 0.94, 0.96, and 1.00, respectively.]

Suitability requirements

Resolution: NLT 2 between campesterol and stigmasterol

Column efficiency: NLT 150,000 theoretical plates for the 5α -cholestane peak

Tailing factor: NMT 2.0 for each relevant peak

Analysis

Samples: *Standard solution* and *Sample solution*

Identify the signals corresponding to the relevant analytes by comparison with the chromatograms obtained with the *System suitability solution*.

Calculate the percentages of the labeled amount of Extract as sterols in the portion of the Capsules taken:

$$\text{Result} = (\Sigma R_U / R_S) \times (C_S \times V / W) \times (A_W \times 100 / L_E) \times 100 / L$$

ΣR_U = ratio of the sum of peak responses for campesterol, stigmasterol, and β -sitosterol relative to the internal standard from the *Sample solution*

R_S = ratio of the β -sitosterol peak relative to the internal standard from the *Standard solution*

C_S = concentration of β -sitosterol in the *Standard stock solution* (mg/mL)

V = volume of the *Standard stock solution* used to prepare the *Standard solution* (2.0 mL)

W = weight of the sample of Capsules taken to prepare the *Sample solution* (mg)

A_W = average weight of the Capsules contents (mg)

L_E = content of sterols in 100 mg of the Extract used to prepare the Capsules (mg)

L = labeled amount of Extract per Capsule (mg/Capsule)

Acceptance criteria: 90%–110% of the labeled amount of Extract, calculated as sterols

• CONTENT OF DOCOSYL FERULATE

Solution A: Methanol and water (95:5)

Solution B: Acetonitrile

Mobile phase: *Solution A* and *Solution B* (17:3)

Standard solution: Dissolve a quantity of [USP Docosyl Ferulate RS](#) in chloroform, and dilute stepwise if necessary, with acetonitrile to obtain a concentration of 0.01 mg/mL. Pass through a membrane filter of 0.45- μ m or finer pore size.

Sample solution: Weigh the contents of NLT 20 Capsules and transfer a quantity of the material, equivalent to 0.2 mg of docosyl ferulate, to a 50-mL beaker. Add 5 mL of chloroform and dissolve in an ultrasonic bath. Transfer to a 20-mL volumetric flask with the aid of NMT 2 mL of chloroform. Dilute with acetonitrile to volume and mix. Pass through a membrane filter of 0.45- μ m or finer pore size.

Chromatographic system

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