

Status: Currently Official on 17-Feb-2025
 Official Date: Official as of 01-Aug-2018
 Document Type: Dietary Supplement Monographs
 DocId: GUID-44EC2B17-2A69-4A25-828F-991FD914C2AB_4_en-US
 DOI: https://doi.org/10.31003/USPNF_M87575_04_01
 DOI Ref: 13za0

© 2025 USPC
 Do not distribute

Valerian Root Dry Extract Capsules

DEFINITION

Valerian Root Dry Extract Capsules contain dry extract derived from the subterranean parts of *Valeriana officinalis* L. (Fam. Caprifoliaceae, formerly Valerianaceae) including rhizome, root, and stolon. They contain NLT 90% and NMT 120% of the labeled amount of valerenic acids calculated as the sum of hydroxyvalerenic acid ($C_{15}H_{22}O_3$), acetoxyvalerenic acid ($C_{17}H_{24}O_4$), and valerenic acid ($C_{15}H_{22}O_2$).

IDENTIFICATION

• A. HPTLC FOR ARTICLES OF BOTANICAL ORIGIN (203)

Standard solution A: 0.25 mg/mL of [USP Valerenic Acid RS](#) in [methanol](#)

Standard solution B: 40 mg/mL of [USP Powdered Valerian Extract RS](#) in [methanol](#). Sonicate for 10 min, centrifuge, and use the supernatant.

Sample solution: Transfer a portion of the Capsule contents, equivalent to 400 mg of valerian root dry extract, to a conical flask, add 10 mL of [methanol](#), mix and sonicate for 20 min, centrifuge, and use the supernatant.

Chromatographic system

Adsorbent: Chromatographic silica gel mixture with an average particle size of 5 µm (HPTLC plates)

Application volume: 5 µL, as 8-mm bands

Relative humidity: Condition the plate to a relative humidity of 33%.

Temperature: Ambient, not to exceed 30°

Developing solvent system: [Cyclohexane](#), [ethyl acetate](#), and [glacial acetic acid](#) (60:38:2)

Developing distance: 6 cm

Derivatization reagent A: [Glacial acetic acid](#) and [hydrochloric acid](#) (1:4)

Derivatization reagent B: 0.5 mL of [p-anisaldehyde](#), 10 mL of [glacial acetic acid](#), and 5 mL of [sulfuric acid](#). Add to 85 mL of ice-cold [methanol](#), and mix.

Analysis

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

Apply the *Samples* as bands, and dry in air. Develop in a saturated chamber, remove the plate from the chamber, and dry in air. Treat the plate with *Derivatization reagent A*, heat at 120° for 5 min, and examine under white light. Then treat with *Derivatization reagent B*, heat at 100° for 3 min, and examine under white light.

System suitability: After treatment with *Derivatization reagent A* and heating, *Standard solution A* and *Standard solution B* do not exhibit an intense blue band at about the middle of the chromatogram nor any other significant bands. After treatment with *Derivatization reagent B* and heating, *Standard solution A* exhibits a violet band due to valerenic acid, while *Standard solution B* exhibits a band corresponding in R_f and color to that of valerenic acid in *Standard solution A* as well as a violet band below valerenic acid at about middle of the chromatogram due to acetoxyvalerenic acid and a minor violet band in the lower third of the chromatogram due to hydroxyvalerenic acid.

Acceptance criteria: After treatment with *Derivatization reagent A* and heating, the *Sample solution* does not exhibit an intense blue band at about the middle of the chromatogram nor any other significant bands [distinction from Mexican valerian (*Valeriana edulis*)], though minor bands may be observed. After treatment with *Derivatization reagent B* and heating, the *Sample solution* exhibits three violet bands in positions and colors similar to those in *Standard solution B*. These bands include a band corresponding in R_f to valerenic acid in *Standard solution A* and *Standard solution B*, a band below valerenic acid at about the middle of the chromatogram due to acetoxyvalerenic acid [distinction from Scouler's valerian (*Valeriana wallichii*)], and a minor violet band in the lower third of the chromatogram due to hydroxyvalerenic acid. Other faint bands may be observed.

• B. HPLC

Analysis: Proceed as directed in the test for *Content of Valerenic Acids*.

Acceptance criteria: The *Sample solution* exhibits a peak at a retention time corresponding to valerenic acid in *Standard solution A*. The *Sample solution* shows additional peaks corresponding to hydroxyvalerenic acid and acetoxyvalerenic acid.

STRENGTH**• CONTENT OF VALERENIC ACIDS**

Extraction solvent: [Methanol](#) and a solution of 0.1% [phosphoric acid](#) in [water](#) (3:1)

Solution A: Mix 6 mL of 85% [phosphoric acid](#) with 900 mL of [water](#), dilute with [water](#) to 1000 mL, mix, filter, and degas.

Solution B: Mix 6 mL of 85% [phosphoric acid](#) with 900 mL of [methanol](#), dilute with [methanol](#) to 1000 mL, mix, filter, and degas.

Mobile phase: See [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	40	60
15	5	95
25	5	95
30	40	60

Standard solution A: 0.05 mg/mL of [USP Valerenic Acid RS](#) in [methanol](#). Sonicate if necessary.

Standard solution B: Sonicate a portion of [USP Powdered Valerian Extract RS](#) in *Extraction solvent* to obtain a solution with a concentration of about 20 mg/mL. Before injection, pass through a membrane filter of 0.45-μm or finer pore size.

Sample solution: Determine the total weight of 20 Capsules. Empty the Capsules and combine their contents in an appropriate container.

Weigh the empty Capsule shells and calculate the average fill weight per Capsule. Transfer a portion of the Capsule contents, equivalent to about 3.0 mg of valerenic acids, to a 25-mL volumetric flask. Add 20 mL of *Extraction solvent* and sonicate for 30 min with occasional shaking. Cool to room temperature, dilute with *Extraction solvent* to volume, mix well, and centrifuge. Pass through a membrane filter of 0.45-μm or finer pore size, discarding the first few milliliters of the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 225 nm

Column: 4.6-mm × 25-cm; end-capped, 5-μm, 100-Å packing L1

Column temperature: 40°

Flow rate: 1.0 mL/min

Injection volume: 25 μL

System suitability

Samples: *Standard solution A* and *Standard solution B*

Suitability requirements

Chromatogram similarity: The chromatogram of *Standard solution B* is similar to the reference chromatogram provided with the lot of [USP Powdered Valerian Extract RS](#) being used.

Tailing factor: NMT 2.0 for the valerenic acid peak, *Standard solution A*

Relative standard deviation: NMT 2.0%, *Standard solution A*

Analysis

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

Using the chromatograms of *Standard solution B* and the reference chromatogram provided with the lot of [USP Powdered Valerian Extract RS](#) being used, identify and measure the areas of the peaks corresponding to hydroxyvalerenic acid, acetoxyvalerenic acid, and valerenic acid in the *Sample solution*.

Calculate the quantity, in mg, of hydroxyvalerenic acid, acetoxyvalerenic acid, and valerenic acid in each Capsule:

$$\text{Result} = (r_U/r_S) \times C_S \times (V \times W_{AV}/W) \times F$$

r_U = peak area of the relevant analyte from the *Sample solution*

r_S = peak area of valerenic acid from *Standard solution A*

C_S = concentration of [USP Valerenic Acid RS](#) in *Standard solution A* (mg/mL)