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Powdered Valerian

DEFINITION

Powdered Valerian is Valerian reduced to a fine or a very fine powder. It contains no calcium oxalate crystals and no foreign starch granules. It contains NLT 0.3% of volatile oil, NLT 0.04% of valerenic acid ($C_{15}H_{22}O_2$), and NLT 0.1% of total valerenic acids, calculated as the sum of hydroxyvalerenic acid, acetoxyvalerenic acid, and valerenic acid, on the dried basis.

IDENTIFICATION

- **A.** Meets the requirements for *Specific Tests, Botanic Characteristics*
- **B.**

Sample solution: 0.2 g of Powdered Valerian in 5 mL of methylene chloride. Shake several times, and allow to stand for 5 min. Filter, wash the filter with 2 mL of methylene chloride, combine the filtrate and washings, and evaporate to dryness. Dissolve the residue in 0.2 mL of methylene chloride.

Analysis: To 0.1 mL of the *Sample solution* add 3 mL of a mixture of equal volumes of glacial acetic acid and 25% hydrochloric acid, and shake several times.

Acceptance criteria: A blue color develops within 15 min.

- **C. Thin-Layer Chromatography**

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: About 0.5 g of Powdered Valerian in 5 mL of methanol. Sonicate for 10 min, centrifuge, and use the supernatant.

Chromatographic system

Adsorbent: Chromatographic silica gel mixture with an average particle size of 2–10 µm (HPTLC plates)

Application volume: 5 µL, as 8-mm bands

Developing solvent system: A mixture of cyclohexane, ethyl acetate, and glacial acetic acid (60:38:2)

Derivatization reagent A: A mixture of 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 to a suitable high-performance thin-layer chromatographic plate. Use a saturated chamber, and condition the plate to a relative humidity of about 33% using a suitable device. Develop the chromatograms over a distance of 6 cm. Remove the plate from the chamber, dry, derivatize with *Derivatization reagent A*, heat at 120° for 5 min, and examine under white light. Derivatize with *Derivatization reagent B*, heat at 100° for 3 min, and examine under white light.

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 the bands of *Standard solution B*. These bands include a minor band in the lower third of the chromatogram due to hydroxyvalerenic acid, a major band at about the middle the chromatogram due to acetoxyvalerenic acid [distinction from Scouler's valerian (*Valeriana wallichii*)], and a major band at an R_F corresponding to the valerenic acid band of *Standard solution A* and *Standard solution B*. Other minor bands may be observed in the *Sample solution* and in *Standard solution B*.

- **D. 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 the valerenic acid peak of *Standard solution A*. The *Sample solution* shows additional peaks corresponding to hydroxyvalerenic acid and acetoxyvalerenic acid.

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COMPOSITION

• **Content of Valerenic Acids**

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

Solvent: A mixture of methanol and a solution of 0.1% phosphoric acid in water (3:1)

Standard solution A: 0.02 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 *Solvent* to obtain a solution having a concentration of about 20 mg/mL. Before injection, pass through a membrane filter of 0.45-µm or finer pore size, discarding the first few mL of the filtrate.

Sample solution: To a 50-mL volumetric flask, transfer about 1.0 g of Powdered Valerian, accurately weighed, add 10.0 mL of water, and shake for 2 min while heating in a water bath maintained at about 50°. Sonicate for 15 min, add 35 mL of methanol, and sonicate for 15 min. Cool, dilute with methanol to volume, and mix. Before injection, pass through a membrane filter of 0.45-µm or finer pore size, discarding the first few mL 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% for the valerenic acid peak in repeated injections, *Standard solution A*

Analysis

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

Identify the valerenic acids in the *Sample solution* chromatogram by comparison with the chromatograms of *Standard solution A*, *Standard solution B*, and the reference chromatogram provided with the lot of USP Powdered Valerian Extract RS being used.

Calculate the percentages of hydroxyvalerenic acid, acetoxyvalerenic acid, and valerenic acid in the portion of Powdered Valerian taken:

$$\text{Result} = (r_U/r_S) \times C_S \times (V/W) \times F \times 100$$

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 valerenic acid in *Standard solution A* (mg/mL)

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

W = weight of Powdered Valerian taken to prepare the *Sample solution* (mg)

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