

Powdered Valerian Extract

Powdered Valerian Extract is prepared from comminuted Valerian and with 70 percent alcohol or other suitable solvents. It contains not less than 0.3 percent of valerenic acid ($C_{15}H_{22}O_2$). The ratio of the starting crude plant material to the Extract is between 4:1 and 7:1.

Packaging and storage— Preserve in tight containers, store at controlled room temperature, and protect from moisture and light.

Labeling— The label states the official name of the article and states also the Latin binomial and the part of the plant from which the article was prepared. Label it to indicate the content of valerenic acid, the extracting solvent used for preparation, and the ratio of the starting crude plant material to the Extract.

Identification—

A: [Thin-Layer Chromatographic Identification Test](#) 201 —

Adsorbent: 0.5-mm layer of chromatographic silica gel mixture.

Test solution— Dissolve about 0.2 g of Extract, accurately weighed, in 2 mL of water, add 3 mL of a 10% aqueous solution of potassium hydroxide, and extract this mixture with two 5-mL portions of methylene chloride. Discard the organic phase, heat the aqueous phase on a water bath at 40°C or 10 minutes, cool, acidify with 7% hydrochloric acid, and extract this solution with two 5-mL portions of methylene chloride. Dry the organic phase over anhydrous sodium sulfate, and filter. Evaporate the filtrate to dryness, and dissolve the residue in 1.0 mL of methylene chloride. Apply 20 μ L to the plate.

Standard solution: 0.5 mg each of [USP Fluorescein RS](#) and [USP Valerenic Acid RS](#) per mL, prepared in methanol. Apply 10 μ L to the plate.

Developing solvent system: a mixture of solvent hexane, ethyl acetate, and glacial acetic acid (65:35:0.5).

Procedure— Spray the plate with anisaldehyde reagent solution, prepared by mixing 0.5 mL of anisaldehyde with 10 mL of glacial acetic acid, 85 mL of methanol, and 5 mL of sulfuric acid, added in the sequence specified. Heat the plate in an oven at 105 °C for about 10 minutes, and examine the plate under white light: the chromatogram of the Standard solution shows a violet zone due to valerenic acid at an R_F value of about 0.4, and a yellow zone due to fluorescein at an

R_F value of about 0.1; the chromatogram of the Test solution shows a violet zone due to valerenic acid at an R_F value of about 0.4, and a blue-violet zone due to hydroxyvalerenic acid at an R_F value of about 0.12, just above the yellow zone in the Standard solution; and the chromatogram of the Test solution may show other colored zones at R_F values lower than those of valerenic acid.

B: The retention time of the valerenic acid peak in the chromatogram of the Test preparation corresponds to that in the chromatogram of the Standard preparation, as obtained in the test for Content of valerenic acid.

[Microbial enumeration](#) [2021](#) — It meets the requirements of the tests for absence of Salmonella species, Escherichia coli, and Staphylococcus aureus. The total bacterial count does not exceed 10,000 cfu per g, the total combined molds and yeasts count does not exceed 1,000 cfu per g, the coliform count does not exceed 1,000 cfu per g, and the Enterobacteriaceae count does not exceed 1,000 cfu per g.

[Loss on drying](#) [731](#) — Dry about 1.0 g of the Extract, accurately weighed, at 105 °C for 2 hours: it loses not more than 9.0% of its weight.

[Total ash](#) [561](#) : not more than 7.0%.

[Pesticide residues](#) [561](#) : meets the requirements.

[Alcohol content, Method II](#) [611](#) (if present): not more than 2.0%.

Content of valerenic acid—

Mobile phase— Prepare a mixture of methanol and water (77:27), add 0.5 mL of phosphoric acid to each 100 mL of the mixture, filter, and degas. Make adjustments if necessary (see System

Suitability under [Chromatography](#) [621](#)).

Standard preparation— Dissolve an accurately weighed quantity of [USP Valerenic Acid RS](#) in methanol, and dilute quantitatively, and stepwise if necessary, with methanol to obtain a solution having a known concentration of about 24 µg per mL.

Test preparation— Transfer an accurately weighed quantity of the Extract, equivalent to about 0.6 mg of valerenic acid, to a 25-mL volumetric flask, and add 15 mL of methanol. Stir for 10 minutes, dilute with methanol to volume, mix, and filter.

Chromatographic system— The liquid chromatograph is equipped with a 225-nm detector and a 4.6-mm × 25-cm column that contains packing L1. The flow rate is about 1.5 mL per minute. The column temperature is maintained at 30 . Chromatograph the Standard preparation, and record the peak responses as directed for Procedure: the capacity factor, k' , determined from valerenic acid is not less than 5; the tailing factor for valerenic acid is not more than 2.0; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure— Separately inject equal volumes (about 20 μ L) of the Standard preparation and the Test preparation into the chromatograph, record the chromatograms, and measure the responses for valerenic acid. Calculate the percentage of valerenic acid (C₁₅H₂₂O₂) in the portion of the Extract taken by the formula:

$$2500(C / W)(r_U / r_S)$$

in which C is the concentration, in mg per mL, of [USP Valerenic Acid RS](#) in the Standard preparation; W is the weight, in mg, of the Extract taken to prepare the Test preparation; and r_U and r_S are the valerenic acid peak responses obtained from the Test preparation and the Standard preparation, respectively: not less than 0.3% is found.