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Casanthranol

» Casanthranol is obtained from Cascara Sagrada. It contains in each 100 g not less than 20.0 g of total hydroxyanthracene derivatives calculated on the dried basis, calculated as cascaroside A. Not less than 80 percent of the total hydroxyanthracene derivatives consists of cascarosides, calculated as cascaroside A.

Packaging and storage—Preserve in tight, light-resistant containers, at a temperature not exceeding 30°.

Loss on drying ⟨731⟩—Dry it in vacuum at 80° for 16 hours: it loses not more than 10.0% of its weight.

Residue on ignition ⟨281⟩: not more than 4.0%.

Assay for total hydroxyanthracene derivatives—[Note 1—Perform all extractions by shaking vigorously, and allow all phases to separate completely before transferring. Entrainment of aglycones into the aqueous phase, as indicated by a value of less than 2.6 for the ratio of the absorbance of the final solution at 515 nm to that at 440 nm, may lead to false results. Note 2—Throughout this assay, use 1 N sodium hydroxide that is prepared without added barium ions as directed for *Volumetric Solutions* in the section *Reagents, Indicators, and Solutions*.]

Ferric chloride solution—Dissolve 100 g of ferric chloride in water to make 100 mL.

Assay solution—Mix a portion of Casanthranol, and transfer an accurately weighed quantity of about 500 mg to a 100-mL volumetric flask. Add about 30 mL of 70 percent alcohol, swirl to dissolve, dilute with 70 percent alcohol to volume, and mix. Quickly filter through soft, rapid-flow filter paper, taking precautions to minimize loss by evaporation.

Assay preparation—Pipet 10 mL of *Assay solution* into a separatory funnel containing 5 mL of water and 2 drops of 1 N hydrochloric acid. Extract with 40 mL of methylene chloride, and transfer the lower layer to a second separatory funnel. Add 10 mL of water to the second separatory funnel, and shake. Allow to separate, discard the lower layer, and transfer the water layer to the first separatory funnel. Extract the combined water layers with 40 mL of methylene chloride, and transfer the lower layer to the second separatory funnel. Add 10 mL of water to the second separatory funnel, and shake. Allow to separate, and discard the lower layer. Transfer the combined water layers, with the aid of water, to a 50-mL volumetric flask, filtering through a small pledget of cotton, water-wet, dilute with water to volume, and mix.

Procedure—Pipet 10 mL of *Assay preparation* into a flask containing 2 mL of *Ferric chloride solution* and 12 mL of hydrochloric acid. Attach a condenser arranged for refluxing, and heat for 3 hours by keeping the flask immersed in boiling water or continuously exposed to steam heat. Cool, wash down the condenser, and transfer to a separatory funnel with the aid of 4 mL of 1 N sodium hydroxide and five 6-mL portions of water. Extract with 20 mL of methylene chloride, and transfer the lower layer to another separatory funnel. Repeat the extraction with three additional 20-mL portions of methylene chloride, wash the combined methylene chloride extracts with two 10-mL portions of water, shaking each time for 2 minutes, and discard the water washings. Transfer the washed methylene chloride extract to a 100-mL volumetric flask, dilute with methylene chloride to volume, and mix. Evaporate a 20.0-mL portion carefully on a water bath to dryness, and dissolve the residue in 10.0 mL of a 1 in 200 solution of magnesium acetate in methanol. Determine the absorbance against methanol as a reference, in 1-cm cells at the wavelength of maximum absorbance at about 515 nm. Calculate the quantity, in mg, of total hydroxyanthracene derivatives in the portion of Casanthranol taken by the formula:

$$155A_U$$

in which A_U is the absorbance of the solution from the *Assay preparation*.

Assay for cascarosides—[Note 1—Perform all extractions by shaking vigorously, and allow all phases to separate completely before transferring. Entrainment of aglycones into the aqueous phase, as indicated by a value of less than 2.7 for the ratio of the absorbance of the final solution at 515 nm to that at 440 nm, may lead to false results. Note 2—Throughout this assay, use 1 N sodium hydroxide that is prepared without added barium ions as directed for *Volumetric Solutions* in the section *Reagents, Indicators, and Solutions*.]

Ferric chloride solution and *Assay solution*—Prepare as directed in the *Assay for total hydroxyanthracene derivatives*.

Assay preparation—Pipet 10 mL of *Assay solution* into a separatory funnel containing 5 mL of water and 2 drops of 1 N hydrochloric acid. Extract with 40 mL of methylene chloride, and transfer the lower layer to a second separatory funnel. Add 10 mL of water to the second separatory funnel, and shake. Allow to separate, discard the lower layer, and transfer the water layer to the first separatory funnel. Extract the combined water layers with 40 mL of methylene chloride, and transfer the lower layer to the second separatory funnel. Add 10 mL of water to the second separatory funnel, and shake. Allow to separate, discard the lower layer, and transfer the water layer to the first separatory funnel. Extract the combined aqueous phase with 30 mL of clear, freshly prepared water-saturated ethyl acetate, and transfer the water layer to another separatory funnel. Repeat the extraction with two additional 30-mL portions of the freshly prepared water-saturated ethyl acetate. Add 5 mL of water to the combined ethyl acetate extracts, shake, allow the phases to separate, discard the ethyl acetate extracts, and add 30 mL of the freshly prepared water-saturated ethyl acetate to the water wash. Shake, allow the phases to separate, and discard the ethyl acetate phase. Transfer the combined aqueous phases, with the aid of water, to a 50-mL volumetric flask, filtering through a small pledget of cotton, water-wet, dilute with water to volume, and mix.

Procedure—Pipet 15 mL of *Assay preparation* into a flask containing 2 mL of *Ferric chloride solution* and 12 mL of hydrochloric acid. Attach a condenser arranged for refluxing, and heat for 3 hours by keeping the flask immersed in boiling water or continuously exposed to steam heat. Cool, wash down the condenser, and transfer to a separatory funnel with the aid of 4 mL of 1 N sodium hydroxide and five 6-mL portions of water. Extract with 20 mL of methylene chloride, and transfer the lower layer to another separatory funnel. Repeat the extraction with three additional 20-mL portions of methylene chloride, wash the combined methylene chloride extracts with two 10-mL portions of water, shaking each time for 2 minutes, and discard the water washings. Transfer the washed methylene chloride extract to a 100-mL volumetric flask, dilute with methylene chloride to volume, and mix. Evaporate a 20.0-mL portion carefully on a water bath to dryness, and dissolve the residue in 10.0 mL of a 1 in 200 solution of magnesium acetate in methanol. Determine the absorbance, against methanol as a reference, in 1-cm cells at the wavelength of maximum absorbance at about 515 nm. Calculate the quantity, in mg, of cascarosides in the portion of Casanthranol taken by the formula:

$$103.5A_U$$

in which A_U is the absorbance of the solution from the *Assay preparation*.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
CASANTHRANOL	Nam-Cheol Kim Scientific Liaison	BDSHM2020 Botanical Dietary Supplements and Herbal Medicines

Chromatographic Database Information: Chromatographic Database

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