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# Cascara Sagrada Extract

## DEFINITION

Cascara Sagrada Extract contains, in each 100 g, NLT 10.0 g and NMT 12.0 g of total hydroxyanthracene derivatives, of which NLT 50% consists of cascarosides, both calculated as cascaroside A.

Mix 900 g of Cascara Sagrada, in coarse powder, with 4000 mL of boiling water, and macerate the mixture for 3 h. Then transfer it to a percolator, allow it to drain, exhaust it by percolation, using boiling water as the menstruum, and collect 5000 mL of percolate. Evaporate the percolate to dryness, reduce the Extract to a fine powder, and, after assaying, add sufficient starch, dried at 100°, or other inert, nontoxic diluents to make the product contain, in each 100 g, 11 g of hydroxyanthracene derivatives. Mix the powders, and pass the Extract through a number 60 sieve.

## ASSAY

### • CONTENT OF TOTAL HYDROXYANTHRACENE DERIVATIVES

Perform all extractions by shaking vigorously, and allow all phases to separate completely before transferring. Entrapment 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.

Throughout this assay, use 1 N sodium hydroxide that is prepared without added barium ions as directed in [Reagents, Indicators, and Solutions –Volumetric Solutions](#).

**Ferric chloride solution:** 1 g/mL of ferric chloride in water

**Sample stock solution:** Transfer 1 g of Extract to a 100-mL volumetric flask. Add 60 mL of 70% alcohol, swirl or sonicate for 15–20 min several times, and allow to stand overnight. Sonicate or swirl for 10–15 min, dilute with 70% alcohol to volume, mix, and filter through suitable filter paper.

**Sample solution:** Pipet 10 mL of *Sample stock 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, and dilute with water to volume, and mix.

### Instrumental conditions

(See [Ultraviolet-Visible Spectroscopy \(857\)](#).)

- Mode:** Visible
- Analytical wavelength:** 515 nm
- Cell:** 1 cm
- Blank:** Methanol

### Analysis

**Sample:** *Sample solution*

**Analysis:** Pipet 10 mL of *Sample solution* 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 h 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 min, and discard the water washings. Transfer the washed methylene chloride extract to a 100-mL volumetric flask, and dilute with methylene chloride to volume. Evaporate a 20.0-mL portion carefully on a water bath to dryness, and dissolve the residue in 10.0 mL of a 5 mg/mL solution of magnesium acetate in methanol.

Calculate the quantity, in mg, of total hydroxyanthracene derivatives ( $T_{HD}$ ) in the portion of Cascara Sagrada Extract taken:

$$T_{HD} = A_U \times F$$

$A_U$  = absorbance of the *Sample solution*

$F$  = conversion factor, 155.2. [NOTE—This conversion factor considers an absorptivity of 16.1 for cascaroside A and the dilutions to prepare the solution for analysis.]

Calculate the percentage of total hydroxyanthracene derivatives in the portion of Cascara Sagrada Extract taken:

$$\text{Result} = (T_{HD}/W) \times 100$$

$W$  = weight of Cascara Sagrada Extract taken to prepare the *Sample stock solution* (mg)

**Acceptance criteria:** 10.0%–12.0% of total hydroxyanthracene derivatives, calculated as cascarioside A

• **CONTENT OF CASCARIOSIDES**

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.

Throughout this assay, use 1 N sodium hydroxide that is prepared without added barium ions as directed in [Reagents, Indicators, and Solutions –Volumetric Solutions](#).

**Ferric chloride solution** and **Sample stock solution:** Prepare as directed in the *Assay for Content of Total Hydroxyanthracene Derivatives*.

**Sample solution:** Pipet 10 mL of *Sample stock 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. Dilute with water to volume, and mix.

**Instrumental conditions**

(See [Ultraviolet-Visible Spectroscopy \(857\)](#).)

**Mode:** Visible

**Analytical wavelength:** 515 nm

**Cell:** 1 cm

**Blank:** Methanol

**Analysis**

**Sample:** *Sample solution*

Pipet 25 mL of the *Sample solution* 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 h 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 min, 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 5-mg/mL solution of magnesium acetate in methanol.

Determine the absorbance and calculate the percentage of cacarosides with respect to the content of total hydroxyanthracene derivatives in the portion of Cascara Sagrada Extract taken:

$$\text{Result} = (A_U/T_{HD}) \times F \times 100$$

$A_U$  = absorbance of the *Sample solution*

$T_{HD}$  = weight of total hydroxyanthracene derivatives (mg)

$F$  = conversion factor, 62.06. [NOTE—This conversion factor considers an absorptivity of 16.1 for cascarioside A and the dilutions to prepare the solution for analysis.]

**Acceptance criteria:** NLT 50% of total hydroxyanthracene derivatives calculated as cascarioside A

**ADDITIONAL REQUIREMENTS**

• **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers at a temperature not exceeding 30°.

Topic/Question	Contact	Expert Committee
CASCARA SAGRADA EXTRACT	<a href="#">Nam-Cheol Kim</a> Scientific Liaison	BDSHM2020 Botanical Dietary Supplements and Herbal Medicines

Chromatographic Database Information: [Chromatographic Database](#)

Most Recently Appeared In:

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