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Cinnamomum cassia Twig Powder

DEFINITION

Cinnamomum cassia Twig Powder consists of the dried twigs of *Cinnamomum cassia* (L.) J.Presl [syn. *Cinnamomum aromaticum* Nees] (Fam. Lauraceae) collected in spring or summer, reduced to a fine or very fine powder. It contains NLT 0.8% of total phenylpropanoids, calculated as the sum of cinnamyl alcohol, cinnamic acid, 2-methoxycinnamic acid, cinnamaldehyde, and 2-methoxycinnamaldehyde on the anhydrous basis.

IDENTIFICATION

• **A. [HPTLC FOR ARTICLES OF BOTANICAL ORIGIN \(203\)](#)**

Standard solution A: 0.5 mg/mL each of [USP Cinnamaldehyde RS](#) and coumarin in methanol

Standard solution B: [USP Cinnamomum cassia Twig Powder RS](#) in methanol (1 in 5). Sonicate for 10 min, centrifuge or filter, and evaporate the solvent. Suspend the residue in a volume of toluene equivalent to one-fifth the volume of methanol used for the extraction. Sonicate for about 2 min, centrifuge or filter, and use the supernatant or the filtrate.

Sample solution: 2 g of Powder in 10 mL of methanol. Sonicate for 10 min, centrifuge or filter, and transfer the extract to a round-bottom flask. Evaporate the solution under reduced pressure to dryness. Dissolve the residue in 2 mL of toluene, sonicate for about 2 min, centrifuge or filter, and use the supernatant or the filtrate.

Chromatographic system

Adsorbent: Chromatographic silica gel F₂₅₄ mixture with an average particle size of about 5 µm

Application volume: 6 µL, as 8-mm bands

Relative humidity: Condition the plate to a relative humidity of about 33% using a suitable device.

Temperature: About 25°

Developing solvent system: Toluene and ethyl acetate (19:1)

Developing distance: 6 cm

Derivatization reagent: Methanol, acetic acid, sulfuric acid, and *p*-anisaldehyde (170:20:10:1). [NOTE—Prepare fresh. Slowly add sulfuric acid to ice-cold methanol, followed by acetic acid and *p*-anisaldehyde.]

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 (20 min with filter paper), remove the plate from the chamber, and dry in air. Examine under UV light at 254 nm. Then treat the plate with the *Derivatization reagent*, heat at 100° for 4 min, and examine under UV light at 366 nm.

System suitability

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

Suitability requirements: Prior to derivatization, under UV light at 254 nm, *Standard solution A* exhibits a quenching band due to cinnamaldehyde in the middle third of the chromatogram, and a quenching band due to coumarin in the upper part of the lower third. Under UV light at 254 nm, *Standard solution B* exhibits, in the middle-third section, an intense quenching band corresponding in *R_F* to the cinnamaldehyde band in *Standard solution A*. In the upper part of the lower-third section, *Standard solution B* exhibits a weak quenching band due to coumarin, a quenching band close to the starting position due to the co-elution of cinnamic acid and 2-methoxycinnamic acid, and a quenching band between coumarin and cinnamic acid due to cinnamyl alcohol. After derivatization, under UV light at 366 nm, *Standard solution B* exhibits a band corresponding in *R_F* and color to the cinnamaldehyde band in *Standard solution A*, and a yellow band immediately below the cinnamaldehyde band.

Acceptance criteria: Under UV light at 254 nm, the *Sample solution* exhibits the most intense quenching band in the middle-third section, corresponding in *R_F* to the cinnamaldehyde band in *Standard solution A*. The *Sample solution* exhibits additional bands corresponding to similar bands in *Standard solution B*; these include a weak quenching band in the upper part of the lower-third section due to coumarin, a quenching band close to the starting position due to the co-elution of cinnamic acid and 2-methoxycinnamic acid, and a quenching band between the coumarin and cinnamic acid bands. After derivatization, under UV light at 366 nm, the *Sample solution* exhibits a band

corresponding in R_f and color to the cinnamaldehyde band in *Standard solution A* and *Standard solution B*, and a yellow band immediately below the cinnamaldehyde band. There is no red band immediately above the cinnamaldehyde band (a distinction from *Cinnamomum verum*).

• **B. HPLC**

Analysis: Proceed as directed in the test for *Content of Total Phenylpropanoids and Coumarin*.

Acceptance criteria: The *Sample solution* exhibits the most intense peak at a retention time corresponding to cinnamaldehyde in *Standard solution B*; a peak with a retention time corresponding to cinnamic acid in *Standard solution A*; and peaks due to coumarin, cinnamyl alcohol, 2-methoxycinnamic acid, and 2-methoxycinnamaldehyde at retention times corresponding to the same constituents in *Standard solution B*. The content ratios of these constituents relative to cinnamic acid are within the typical ratio ranges listed in [Table 2](#). The content of cinnamaldehyde is NLT 65% of the content of total phenylpropanoids.

COMPOSITION

• **CONTENT OF TOTAL PHENYLPROPANOIDS AND COUMARIN**

[NOTE—Protect from light and proceed under low actinic light. *Standard solution A*, *Standard solution B*, and the *Sample solution* are stable for 24 h at room temperature.]

Solution A: 0.05% phosphoric acid in water

Solution B: Acetonitrile

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

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	75	25
1	75	25
21	62	38
30	60	40
35	60	40

Solvent: Methanol and water (7:3)

Standard solution A: 0.05 mg/mL of [USP Cinnamic Acid RS](#) in methanol

Standard solution B: Transfer about 250 mg of [USP Cinnamomum cassia Twig Powder RS](#) into a 50-mL round-bottom centrifuge tube, and add 25 mL of *Solvent*. Sonicate for 30 min, cool, and centrifuge. Before injection, pass through a membrane filter of 0.45-µm or finer pore size.

Sample solution: Accurately transfer about 500 mg of Powder into a 50-mL round-bottom centrifuge tube, and add 25 mL of *Solvent*. Sonicate for 30 min (250 W, 33 kHz), cool, centrifuge, and transfer the supernatant to a 50-mL volumetric flask. Repeat the extraction one more time by adding 15 mL of *Solvent*, and transfer the supernatant to the same 50-mL volumetric flask, dilute with *Solvent* to volume, and mix. Before injection, pass through a membrane filter of 0.45-µm or finer pore size and discard the first portion of the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 265 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Column temperature: 25°

Flow rate: 1.0 mL/min

Injection volume: 10 µL

System suitability

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

Suitability requirements

Resolution: NLT 1.5 between the cinnamyl alcohol and cinnamic acid peaks and between the coumarin and cinnamyl alcohol peaks, *Standard solution B*

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