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▲Dong Quai Root Powder

DEFINITION

Dong Quai Root Powder (also known as *Danggui*) consists of the dried root of *Angelica sinensis* (Oliv.) Diels (Fam. Apiaceae) reduced to a fine or very fine powder. It contains NLT 0.60% of total butylidenephthalides calculated as the sum of *E*-ligustilide ($C_{12}H_{14}O_2$), *E*-butylidenephthalide ($C_{12}H_{12}O_2$), *Z*-ligustilide ($C_{12}H_{14}O_2$), and *Z*-butylidenephthalide ($C_{12}H_{12}O_2$); and NLT 0.15% of total phenylpropanoids calculated as the sum of ferulic acid ($C_{10}H_{10}O_4$) and coniferyl ferulate ($C_{20}H_{20}O_6$), both on the anhydrous basis.

IDENTIFICATION

• **A. HPTLC for Articles of Botanical Origin ⟨203⟩**

Standard solution A: 0.5 mg/mL each of USP *Z*–Ligustilide RS, USP Senkyunolide A RS, and USP Ferulic Acid RS in methanol

Standard solution B: 200 mg/mL of USP *Angelica sinensis* Root Powder RS in methanol. Sonicate for 20 min, centrifuge, and use the supernatant.

Sample solution: 200 mg/mL of Dong Quai Root Powder in methanol. Sonicate for 20 min, centrifuge, and use the supernatant.

Chromatographic system

(See standard parameters as defined in *HPTLC for Article of Botanical Origin ⟨203⟩*, *Table 1*.)

Application volume: 4 µL

Developing solvent system: Toluene, ethyl acetate, and glacial acetic acid (9: 1: 0.1)

Derivatization reagent: 10% sulfuric acid in alcohol. Slowly add sulfuric acid to ice-cold alcohol. Prepare on the day of the test; it can be used for up to 1 week if preserved in a tightly closed container and stored in a cool place.

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, remove the plate from the chamber, and dry in air. Examine under UV light at 254 nm. Then treat the plate with *Derivatization reagent*, heat at 105° for 5 min, and examine under white light.

System suitability

Under UV light at 254 nm: *Standard solution A* exhibits a quenching band due to ferulic acid in the lower-third section, a quenching band due to senkyunolide A in the middle-third section, and a blue band due to *Z*-ligustilide above senkyunolide A. *Standard solution B* exhibits a quenching band and a blue band corresponding in R_F to ferulic acid and *Z*-ligustilide in *Standard solution A*, respectively, as well as a quenching band right above ferulic acid. *Standard solution B* does not exhibit a band corresponding in R_F to senkyunolide A in *Standard solution A*.

Under white light: *Standard solution A* exhibits a purple band due to ferulic acid in lower-third section, a brown band due to senkyunolide A in the middle-third section, and a brown band due to *Z*-ligustilide above senkyunolide A. In the lower-third section, *Standard solution B* exhibits four bands in the following order with increasing R_F: a purple band corresponding to ferulic acid in *Standard solution A*, a brown band, a red-brown band, and a brown band. In the upper-half section, *Standard solution B* exhibits a band corresponding in R_F and color to *Z*-ligustilide in *Standard solution A* and a red brown band above *Z*-ligustilide. *Standard solution B* does not exhibit a band corresponding in R_F to senkyunolide A in *Standard solution A*.

Acceptance criteria

Under UV light at 254 nm: The *Sample solution* exhibits a quenching band and a blue band corresponding in R_F to ferulic acid and *Z*-ligustilide, respectively, in both *Standard solution A* and *Standard solution B*; a quenching band right above ferulic acid; and additional faint quenching bands corresponding in R_F to the same bands in *Standard solution B*. The *Sample solution* does not exhibit a band corresponding in R_F to senkyunolide A in *Standard solution A* (a distinction from *Ligusticum chuanxiong* rhizome).

Under white light: In the lower-third section, the *Sample solution* exhibits four bands in the following order with increasing R_F: a purple band corresponding to ferulic acid in *Standard solution A* and *Standard solution B*, and a brown band, a red brown band, and a brown band.

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corresponding to the same bands in *Standard solution B*. In the upper-half section, the *Sample solution* exhibits a band corresponding in R_F and color to *Z*-ligustilide in *Standard solution A* and a red brown band above *Z*-ligustilide corresponding to the same band in *Standard solution B*. The *Sample solution* does not exhibit a band corresponding in R_F to senkyunolide A in *Standard solution A* (a distinction from *L. chuanxiong* rhizome).

• **B. HPLC**

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

Acceptance criteria: The *Sample solution* exhibits the most intense peak corresponding to *Z*-ligustilide in *Standard solution A* (a distinction from *Angelica gigas*, which does not exhibit a *Z*-ligustilide peak), a peak corresponding to ferulic acid in *Standard solution B*, and peaks due to senkyunolide I, senkyunolide H, coniferyl ferulate (a distinction from *A. gigas* and *Angelica acutiloba*, which do not exhibit a significant coniferyl ferulate peak), *E*-ligustilide, *E*-butylidenephthalide, and *Z*-butylidenephthalide corresponding to the same constituents in *Standard solution C*. The peak intensity of senkyunolide I is much higher than senkyunolide H. Beside the above mentioned peaks, no peak between ferulic acid and coniferyl ferulate is more intense than senkyunolide H, and no peak between coniferyl ferulate and *E*-butylidenephthalide is more intense than *E*-butylidenephthalide. The content ratios of *E*-ligustilide, *E*-butylidenephthalide, and *Z*-butylidenephthalide to *Z*-ligustilide are within the ranges listed in *Table 2*.

COMPOSITION

• **Content of Butylidenephthalides and Phenylpropanoids**

[Note—Protect solutions from light and proceed under low-actinic light.]

Solution A: 0.1% formic acid in water

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	92	8
3	92	8
15	53	47
30	53	47
30.1	0	100
35	0	100
35.1	92	8
40	92	8

Solvent: Methanol and formic acid (95:5)

Standard solution A: Remove USP *Z*–Ligustilide RS from the freezer. When it reaches to room temperature, open the vial and immediately prepare a solution of 0.2 mg/mL of *Z*-ligustilide in methanol under low-actinic light.

Standard solution B: 0.10 mg/mL each of USP Ferulic Acid RS and USP Oxybenzone RS in *Solvent*

Standard solution C: 20 mg/mL of USP *Angelica sinensis* Root Powder RS in *Solvent*. Sonicate for 30 min, centrifuge, and pass through a suitable membrane filter of 0.45-µm or finer pore size.

Sample solution: Transfer about 200 mg of Dong Quai Root Powder, accurately weighed, to a suitable flask, add 10.0 mL of *Solvent*, and close tightly. Weigh the filled flask accurately and sonicate for 30 min. Cool to room temperature and, if necessary, adjust with *Solvent* to the initial weight. Before injection, pass through a suitable membrane filter of 0.45-µm or finer pore size and discard the initial portion of the filtrate.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 274 nm

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