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^Sichuan Lovage Rhizome Powder

DEFINITION

Sichuan Lovage Rhizome Powder (also known as *Chuanxiong*) consists of the dried rhizome of *Ligusticum striatum* DC. (syn. *Ligusticum chuanxiong* S.H.Qiu, Y.Q.Zeng, K.Y.Pan, Y.C.Tang & J.M.Xu) (Fam. Apiaceae) reduced to a fine or very fine powder. It contains NLT 1.5% of total butyridenepthalides calculated as the sum of senkyunolide A ($C_{12}H_{16}O_2$), *E*-ligustilide ($C_{12}H_{14}O_2$), *Z*-ligustilide ($C_{12}H_{14}O_2$), and *Z*-butyridenepthalide ($C_{12}H_{12}O_2$); and NLT 0.2% 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 *Ligusticum chuanxiong* Rhizome Powder RS](#) in [methanol](#). Sonicate for 20 min, centrifuge, and use the supernatant.

Sample solution: 200 mg/mL of Sichuan Lovage Rhizome Powder in [methanol](#). Sonicate for 20 min, centrifuge, and use the supernatant.

Chromatographic system

(See standard parameters as defined in [HPTLC for Articles of Botanical Origin \(203\)](#), [Table 1](#).)

Application volume: 4 μ L

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

Derivatization reagent: 10% [sulfuric acid](#) in [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. Slowly add [sulfuric acid](#) to ice-cold [alcohol](#).

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 two quenching bands and a blue band corresponding in R_f to ferulic acid, senkyunolide A, and *Z*-ligustilide in *Standard solution A*, respectively, as well as three weak quenching bands: one right above ferulic acid, one right below senkyunolide A, and one right above or overlapped with *Z*-ligustilide.

Under white light: *Standard solution A* exhibits a purple band due to ferulic acid in the 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 weak purple band, an intense red-brown band, and a red-brown band. In the middle-third section, *Standard solution B* exhibits two brown bands corresponding in R_f to senkyunolide A and *Z*-ligustilide in *Standard solution A* and a weak band right above senkyunolide A and below *Z*-ligustilide. In the upper-third section, *Standard solution B* exhibits an intense red-brown band.

Acceptance criteria

Under UV light at 254 nm: The *Sample solution* exhibits two quenching bands and a blue band corresponding in R_f to ferulic acid, senkyunolide A (a distinction from *Angelica sinensis* root, which does not exhibit a distinct band due to senkyunolide A), and *Z*-ligustilide, respectively, in both *Standard solution A* and *Standard solution B*. The *Sample solution* exhibits additional weak quenching bands corresponding in R_f to the same bands in *Standard solution B*.

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 weak purple band, an intense red-brown band, and a red-brown band.

red-brown band corresponding to the same bands in *Standard solution B*. In the middle-third section, the *Sample solution* exhibits a brown band corresponding in R_f to senkyunolide A (a distinction from *A. sinensis* root) in *Standard solution A*, a weak band right above senkyunolide A, and a brown band corresponding in R_f to Z-ligustilide in *Standard solution A*. In the upper-third section, the *Sample solution* exhibits an intense red-brown band.

• 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 peak corresponding to ferulic acid in *Standard solution B*, and peaks due to senkyunolide I, senkyunolide H, coniferyl ferulate, senkyunolide A (a distinction from *A. sinensis* root, which does not exhibit a significant senkyunolide A peak), *E*-ligustilide, and Z-butylidenephthalide corresponding to the same constituents in *Standard solution C*. The peak intensity of senkyunolide I is much higher than senkyunolide H. The peak intensity of senkyunolide A is lower than Z-ligustilide. The content ratio of senkyunolide A to Z-ligustilide is between 0.4 to 0.9. *E*-Ligustilide and Z-butylidenephthalide are minor peaks. Beside the above mentioned peaks, no other peak between ferulic acid and senkyunolide I and between senkyunolide H and coniferyl ferulate is more intense than senkyunolide H.

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 [USP Z-Ligustilide RS](#) 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: 15 mg/mL of [USP Ligusticum chuanxiong Rhizome Powder RS](#) in *Solvent*. Sonicate for 30 min, centrifuge, and pass through a suitable membrane filter of 0.45- μ m or finer pore size.

Sample solution: Transfer about 150 mg of Sichuan Lovage Rhizome 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- μ m 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

Column: 4.6-mm $\times$ 15-cm; 3.5- μ m packing [L1](#)