Peucedani Decursivi Radix

Figure 1  A photograph of Peucedani Decursivi Radix
1. NAMES

Official Name: Peucedani Decursivi Radix

Chinese Name: 紫花前胡

Chinese Phonetic Name: Zihuaqianhu

2. SOURCE

Peucedani Decursivi Radix is the dried root of Peucedanum decursivum (Miq.) Maxim. (Apiaceae). The root is collected in autumn and winter when stem and leaves withered, freed of aerial part, soil and rootlet, washed clean, then dried under the sun or at ambient temperature to obtain Peucedani Decursivi Radix.

3. DESCRIPTION

Irregular cylindrical, conical or fusiform, slightly twisted, branched at the lower part, 3-18 cm long, 5-22 mm in diameter. Externally yellowish-brown to dark brown, with longitudinally wrinkles, rootlet scars and transverse lenticel-like cicatrices. The top part is stubby, frequently with stem scars and fibrous leaf-bases, annular striaion occasionally at the upper end. Texture hard, easily broken; fracture uneven, off-white or pale yellow, yellow oil spots scattered in cortex. Odour strongly aromatic; taste slightly bitter and pungent (Fig. 1).

4. IDENTIFICATION

4.1 Microscopic Identification (Appendix III)

Transverse section
Cork consists of 10 or more layers of flattened cells. Cortex narrow, with scattered oil cavities. Phloem broad, cells arranged somewhat densely, frequently with oil cavities, subrounded with 5-10 secretory cells, 42-166 µm in diameter. Phloem ray slightly curved. Cambium in a ring. Xylem shows vessels arranged radially, xylem rays relatively wide. Starch granules abundantly present in the parenchymatous cells (Fig. 2).
Powder
Colour greyish-yellow to yellowish-brown. Starch granules abundant, simple granules subrounded or polygonal, hilum dotted or slit-shaped visible in large granules, 5-20 μm in diameter, compound granules composed of 2-10 units; black and cruciate in shape under the polarized microscope. Fibre in bundles or scattered singly, long-strip shaped, 8-35 μm in diameter, pit canals distinctly visible; yellowish-white under the polarized microscope. Vessels mainly reticulate and scalariform in type, 14-80 μm in diameter. Oil cavities mostly fractured, containing yellowish-brown secretions. Cork cells subpolygonal or irregular (Fig. 3).

4.2 Thin-Layer Chromatographic Identification [Appendix IV(A)]

Standard solution
_Nodakenin standard solution_
Weigh 0.5 mg of nodakenin CRS (Fig. 4) and dissolve in 5 mL of methanol.

Developing solvent system
Prepare a mixture of ethyl acetate, acetone and formic acid (6:3:1, v/v).

Test solution
Weigh 0.2 g of the powdered sample and place it in a 50-mL conical flask, then add 10 mL of methanol. Sonicate (90 W) the mixture for 15 min. Filter the mixture.

Procedure
Carry out the method by using a HPTLC silica gel _F<sub>254</sub>_ plate, a twin trough chamber and a freshly prepared developing solvent system as described above. Apply separately nodakenin standard solution and the test solution (2 μL each) to the plate. Before the development, add the developing solvent to one of the troughs of the chamber and place the HPTLC plate in the other trough. Cover the chamber with a lid and let equilibrate for about 15 min. Carefully tilt the chamber to allow sufficient solvent to pass from the trough containing the solvent to the other containing the HPTLC plate for development. Develop over a path of about 8.5 cm. After the development, remove the plate from the chamber, mark the solvent front and dry in air. Examine the plate under UV light (366 nm). Calculate the _R_f value by using the equation as indicated in Appendix IV(A).

For positive identification, the sample must give spots or bands with chromatographic characteristics, including the colour and the _R_f value, corresponding to those of nodakenin.
Figure 2  Microscopic features of transverse section of Peucedani Decursivi Radix

A. Sketch    B. Section illustration    C. Oil cavity

Figure 3  Microscopic features of powder of Peucedani Decursivi Radix

5. Fragments of oil cavity  6. Cork cells

a. Features under the light microscope   b. Features under the polarized microscope
4.3 High-Performance Liquid Chromatographic Fingerprinting (Appendix XII)

**Standard solution**

*Nodakenin standard solution for fingerprinting, Std-FP (14 mg/L)*

Weigh 0.14 mg of nodakenin CRS and dissolve in 10 mL of methanol.

**Test solution**

Weigh 0.1 g of the powdered sample and place it in a 125-mL conical flask, then add 50 mL of methanol. Sonicate (90 W) the mixture for 30 min. Filter through a 0.45-µm nylon filter.

**Chromatographic system**

The liquid chromatograph is equipped with a DAD (325 nm) and a column (4.6 × 250 mm) packed with ODS bonded silica gel (5 µm particle size). The flow rate is about 1.0 mL/min. Programme the chromatographic system as follows (Table 1) –

**Table 1  Chromatographic system conditions**

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Water (% v/v)</th>
<th>Methanol (% v/v)</th>
<th>Elution</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 – 15</td>
<td>60 → 50</td>
<td>40 → 50</td>
<td>linear gradient</td>
</tr>
<tr>
<td>15 – 20</td>
<td>50 → 35</td>
<td>50 → 65</td>
<td>linear gradient</td>
</tr>
<tr>
<td>20 – 60</td>
<td>35 → 10</td>
<td>65 → 90</td>
<td>linear gradient</td>
</tr>
</tbody>
</table>

**System suitability requirements**

Perform at least five replicate injections, each using 10 µL of nodakenin Std-FP. The requirements of the system suitability parameters are as follows: the RSD of the peak area of nodakenin should not be more than 5.0%; the RSD of the retention time of nodakenin peak should not be more than 2.0%; the column efficiency determined from nodakenin peak should not be less than 10000 theoretical plates.
The $R$ value between peak 1 and the closest peak in the chromatogram of the test solution should not be less than 1.5 (Fig. 5).

**Procedure**

Separately inject nodakenin Std-FP and the test solution (10 µL each) into the HPLC system and record the chromatograms. Measure the retention time of nodakenin peak in the chromatogram of nodakenin Std-FP and the retention times of the four characteristic peaks (Fig. 5) in the chromatogram of the test solution. Identify nodakenin peak in the chromatogram of the test solution by comparing its retention time with that in the chromatogram of nodakenin Std-FP. The retention times of nodakenin peaks from the two chromatograms should not differ by more than 2.0%. Calculate the RRTs of the characteristic peaks by using the equation as indicated in Appendix XII.

The RRTs and acceptable ranges of the four characteristic peaks of Peucedani Decursivi Radix extract are listed in Table 2.

<table>
<thead>
<tr>
<th>Peak No.</th>
<th>RRT</th>
<th>Acceptable Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (marker, nodakenin)</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>2.61</td>
<td>± 0.04</td>
</tr>
<tr>
<td>3</td>
<td>4.13</td>
<td>± 0.03</td>
</tr>
<tr>
<td>4</td>
<td>4.23</td>
<td>± 0.03</td>
</tr>
</tbody>
</table>

Figure 5  A reference fingerprint chromatogram of Peucedani Decursivi Radix extract
For positive identification, the sample must give the above four characteristic peaks with RRTs falling within the acceptable range of the corresponding peaks in the reference fingerprint chromatogram (Fig. 5).

5. **TESTS**

5.1 **Heavy Metals** *(Appendix V)*: meet the requirements.

5.2 **Pesticide Residues** *(Appendix VI)*: meet the requirements.

5.3 **Mycotoxins** *(Appendix VII)*: meet the requirements.

5.4 **Foreign Matter** *(Appendix VIII)*: not more than 1.0%.

5.5 **Ash** *(Appendix IX)*

Total ash: not more than 7.0%.
Acid-insoluble ash: not more than 1.0%.

5.6 **Water Content** *(Appendix X)*

Oven dried method: not more than 11.0%.

6. **EXTRACTIVES** *(Appendix XI)*

Water-soluble extractives (cold extraction method): not less than 20.0%.
Ethanol-soluble extractives (hot extraction method): not less than 21.0%.

7. **ASSAY**

Carry out the method as directed in Appendix IV(B).

**Standard solution**

*Nodakenin standard stock solution, Std-Stock (400 mg/L)*

Weigh accurately 4.0 mg of nodakenin CRS and dissolve in 10 mL of methanol.

*Nodakenin standard solution for assay, Std-AS*

Measure accurately the volume of the nodakenin Std-Stock, dilute with methanol to produce a series of solutions of 5.2, 10, 15.2, 20, 40 mg/L for nodakenin.
Test solution
Weigh accurately 0.1 g of the powdered sample and place it in a 125-mL conical flask, then add 50 mL of methanol. Sonicate (90 W) the mixture for 30 min. Filter and transfer the filtrate to a 250-mL round-bottomed flask. Repeat the extraction for one more time. Combine the filtrates and evaporate the solvent to dryness at reduced pressure in a rotary evaporator. Dissolve the residue in methanol. Transfer the solution to a 50-mL volumetric flask and make up to the mark with methanol. Filter through a 0.45-µm nylon filter.

Chromatographic system
The liquid chromatograph is equipped with a DAD (335 nm) and a column (4.6 × 250 mm) packed with ODS bonded silica gel (5 µm particle size). The flow rate is about 1.0 mL/min. The mobile phase is a mixture of methanol and water (45:55, v/v). The elution time is about 15 min.

System suitability requirements
Perform at least five replicate injections, each using 10 µL of nodakenin Std-AS (15.2 mg/L). The requirements of the system suitability parameters are as follows: the RSD of the peak area of nodakenin should not be more than 5.0%; the RSD of the retention time of nodakenin peak should not be more than 2.0%; the column efficiency determined from nodakenin peak should not be less than 5000 theoretical plates.

The R value between nodakenin peak and the closest peak in the chromatogram of the test solution should not be less than 1.5.

Calibration curve
Inject a series of nodakenin Std-AS (10 µL each) into the HPLC system and record the chromatograms. Plot the peak areas of nodakenin against the corresponding concentrations of nodakenin Std-AS. Obtain the slope, intercept and the \( r^2 \) value from the 5-point calibration curve.

Procedure
Inject 10 µL of the test solution into the HPLC system and record the chromatogram. Identify nodakenin peak in the chromatogram of the test solution by comparing its retention time with that in the chromatogram of nodakenin Std-AS. The retention times of nodakenin peaks from the two chromatograms should not differ by more than 2.0%. Measure the peak area and calculate the concentration (in milligram per litre) of nodakenin in the test solution, and calculate the percentage content of nodakenin in the sample by using the equations indicated in Appendix IV(B).
**Limits**

The sample contains not less than 0.90% of nodakenin (C$_{20}$H$_{24}$O$_9$), calculated with reference to the dried substance.