Angelica gigas Root

Proposed For Development Version 0.1

DEFINITION
The article consists of the dried roots of *Angelica gigas* Nakai (Family Apiaceae). It contains NLT 4.2% of caffeoylquinic acid and total coumarins calculated as the sum of chlorogenic acid, demethylsuberosin, decursin, and decursinol angelate, on the dried basis.

SYNONYMS
None known

POTENTIAL CONFOUNDING MATERIALS
*Angelica acutiloba* (Siebold & Zucc.) Kitag.
*Angelica sinensis* (Oliv.) Diels

SELECTED COMMON NAMES
Chinese: 朝鲜当归
English: Korean angelica, Korean tanggwi
Korean: 참당귀

CONSTITUENTS OF INTEREST
Coumarins: Decursin, decursinol, decursinol angelate, nodakenin

IDENTIFICATION
• A. BOTANICAL CHARACTERISTICS

  Macroscopic: Conical or narrow long conical in shape, usually branched, 15–25 cm in length and 2–5 cm in diameter. The external surface is pale yellowish brown to blackish brown with irregular longitudinal wrinkles and spot-shaped remains of fibrous roots. The crown is broad, usually with remains of stems and leaves. The texture is hard but fragile. The fractured surface has a pale brown or yellowish brown cortex, relatively sparse with numerous clefts, and the xylem is white or yellowish white. This has a slight, characteristic odor, and a slightly bitter and sweet taste.

  Microscopic: Under a microscope, the transverse section reveals cork consisting of 5–6 layers of cells, cells aligned transversely, parenchymas from primary cortex to xylem aligned systemically in rectangular shape. The cortex has schizogenous intercellular space, secretory canal with a yellowish brown ingredient and bast fiber bundles are sparsely scattered. Scalariform or spiral vessel is observed. Numerous starch grains are observed in parenchyma cells.

• B. HPTLC FOR ARTICLES OF BOTANICAL ORIGIN <203>

  Standard solution: 1.0 mg/mL each of USP Imperatorin RS, USP Osthole RS, and USP Isoimperatorin RS in methanol
  Sample solution: 1 g of *Angelica gigas* Root, finely powdered, in 5 mL of methanol. Sonicate for 10 min and centrifuge or filter. Use the supernatant or filtrate.

  Chromatographic system
  Adsorbent: Chromatographic silica gel F<sub>254</sub> mixture
  Application volume: 10 µL each of Standard solution and Sample solution, as 8-mm bands
  Relative Humidity: Condition the plate to a relative humidity of about 33% using a suitable device
  Developing solvent system: Toluene, ethyl acetate, and acetic acid (90:10:1)
  Developing distance: 6 cm

  Analysis
  Samples: Standard solution and Sample solution
  Apply the Samples as bands to a suitable HPTLC plate and dry in air. Develop the chromatograms in a saturated chamber (20 min with filter paper), remove the plate from the chamber, and examine under UV 365 nm light.

  System suitability: To Come
Acceptance criteria: Under UV 365 nm light, the chromatogram of the Sample solution exhibits about 8 fluorescence bands in the lower half of the chromatogram. Three pale fluorescence bands appear above the broad fluorescence band near R, 0.25 corresponding in color and R, to the bands due to imperatorin, osthole, and isoimperatorin, with increasing R, in the Standard solution.

C. HPLC
Analysis: Proceed as directed in the Assay for Content of Caffeoylquinic Acid and Total Coumarins.
Acceptance criteria: The Sample solution exhibits the most intense peak at a retention time corresponding to each Standard solution.

ASSAY
• CONTENT OF CAFFEYOYLQUINIC ACID AND TOTAL COUMARINS
  Solution A: 0.1% formic acid in water
  Solution B: 0.1% formic acid in acetonitrile
  Mobile phase: See Table 1.

Table 1

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Solution A (%)</th>
<th>Solution B (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>88</td>
<td>12</td>
</tr>
<tr>
<td>10</td>
<td>82</td>
<td>18</td>
</tr>
<tr>
<td>25</td>
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<td>30</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>60</td>
<td>50</td>
<td>50</td>
</tr>
</tbody>
</table>

Solvent: Ethyl alcohol and water (7:3)
Standard solution A: 0.2 mg/mL of USP Chlorogenic Acid RS in methanol
Standard solution B: 0.8 mg/mL of USP Demethylsuberosin RS in methanol
Standard solution C: 2 mg/mL of USP Decursin RS in methanol
Standard solution D: 2 mg/mL of USP Decursinol Angelate RS in methanol
Sample solution: Accurately transfer about 1.0 g of finely powdered Angelica gigas Root to centrifugal tube, add 10 mL of Solvent, and weigh. Sonicate for 45 min, cool, and compensate the weight loss with Solvent. Before injection, pass through a membrane filter of 0.45-μm pore size.

Chromatographic system
(See Chromatography <621>, System Suitability.)
  Detector: UV 325 nm
  Column: 4.6-mm × 25-cm; packing L1
  Column temperature: 40°
  Flow rate: 1.0 mL/min
  Injection volume: 10 μL
System suitability: To Come
Analysis
Samples: Standard solutions A–D and Sample solution
Calculate the percentage of chlorogenic acid, demethylsuberosin, decursin, and decursinol angelate in the portion of Angelica gigas Root taken:

\[ \text{Result} = \left( \frac{r_u}{r_s} \right) \times C_s \times \left( \frac{V}{W} \right) \times 100 \]

\( r_u \) = peak area of the corresponding analyte from the Sample solution
\( r_s \) = peak area of the corresponding analyte from the Standard solution
\( C_s \) = concentration of the Standard solution (mg/mL)
\( V \) = volume of the Sample solution, mL
\( W \) = weight of Angelica gigas Root taken to prepare the Sample solution, mg
Acceptance criteria: NLT 4.2% of caffeoylquinic acid and coumarins on the dried basis

CONTAMINANTS
- **Elemental Impurities—Procedures** <233>

Acceptance criteria
- Arsenic: NMT 3.0 µg/g
- Cadmium: NMT 0.3 µg/g
- Lead: NMT 5.0 µg/g
- Mercury: NMT 0.2 µg/g
- **Articles of Botanical Origin** <561>, Pesticide Residue Analysis: Meets the requirements

- **Microbial Enumeration Tests** <61>: The total aerobic bacterial count does not exceed $10^5$ cfu/g, the total combined molds and yeasts count does not exceed $10^3$ cfu/g, and the bile-tolerant Gram-negative bacteria does not exceed $10^3$ cfu/g.
- **Tests for Specified Microorganisms** <62>: Meets the requirements of the tests for absence of *Salmonella* species and *Escherichia coli*
- **Articles of Botanical Origin** <561>, Test for Aflatoxins: Meets the requirements

SPECIFIC TESTS
- **Articles of Botanical Origin** <561>, Methods of Analysis, Foreign Organic Matter: NMT 5.0%
- **Articles of Botanical Origin** <561>, Methods of Analysis, Alcohol-Soluble Extractives, Method 1: To Come
- **Articles of Botanical Origin** <561>, Methods of Analysis, Water-Soluble Extractives, Method 2: To Come
- **Loss on Drying** <731>: To Come
- **Articles of Botanical Origin** <561>, Methods of Analysis, Total Ash: NMT 6.0%
- **Articles of Botanical Origin** <561>, Methods of Analysis, Acid-Insoluble Ash: To Come

ADDITIONAL REQUIREMENTS
- **Packaging and Storage**: Preserve in well-closed containers, protected from light and moisture, and store at room temperature.
- **Labeling**: The label states the Latin binomial and the part(s) of the plant contained in the article.

**USP Reference Standards** <11>
- USP Chlorogenic Acid RS
- USP Decursin RS
- USP Decursinol Angelate RS
- USP Demethylsuberosin RS
- USP Imperatorin RS
- USP Isoimperatorin RS
- USP Osthole RS