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Tangerine Peel

DEFINITION

Tangerine Peel consists of the dried exocarp and mesocarp of the ripe fruit of *Citrus reticulata* Blanco (Fam. Rutaceae), partly freed from the white spongy tissue of the mesocarp. It contains NLT 5.0% of total dihydroflavone glycosides, calculated as the sum of narirutin ($C_{27}H_{32}O_{14}$), hesperidin ($C_{28}H_{34}O_{15}$), and didymin ($C_{28}H_{34}O_{14}$) on the anhydrous basis, and NLT 0.1% of total polymethoxylated flavones, calculated as the sum of nobiletin ($C_{21}H_{22}O_8$), 3,5,6,7,8,3',4'-heptamethoxyflavone ($C_{22}H_{24}O_9$), and tangeretin ($C_{20}H_{20}O_7$) on the anhydrous basis.

IDENTIFICATION

• A. HPTLC FOR ARTICLES OF BOTANICAL ORIGIN (203)

Standard solution A: 1.0 mg/mL of [USP Hesperidin RS](#) in [methanol](#). Sonicate to dissolve.

Standard solution B: 50 mg/mL of [USP Citrus reticulata Pericarp Dry Extract RS](#) in [methanol](#). Sonicate for 20 min, centrifuge, and use the supernatant.

Sample solution: 500 mg of Tangerine Peel, finely powdered, in 5 mL of [methanol](#). Sonicate for 20 min, centrifuge, and use the supernatant.

Chromatographic system

Adsorbent: Chromatographic silica gel F₂₅₄ mixture

Application volume: *Standard solution A*, 10 µL; *Standard solution B* and *Sample solution*, 5 µL for each, 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: [Ethyl acetate](#), [formic acid](#), and [water](#) (100:15:13)

Derivatization reagent A: 10 mg/mL of [2-aminoethyl diphenylborinate](#) in [methanol](#)

Derivatization reagent B: 50 mg/mL of [polyethylene glycol 4000](#) in [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 the plate at 100° for 3 min. Treat the plate with *Derivatization reagent A* and dry for 5 min with a stream of cool air. Immediately, treat the plate with *Derivatization reagent B*, dry for 5 min with a stream of cool air, and examine under UV light at 366 nm.

System suitability

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

Suitability requirements: *Standard solution A* exhibits a yellowish-brown band due to hesperidin in the lower half section. *Standard solution B* exhibits a band corresponding in R_f and color to the band due to hesperidin in *Standard solution A*, a yellow band below hesperidin, a blue band below the yellow band, another blue band above hesperidin, and a light yellowish-brown band between hesperidin and the blue band above. In the upper-third section, *Standard solution B* exhibits a bright blue band due to the coelution of nobiletin with some other components, and a faint green band above the bright blue band.

Acceptance criteria: The *Sample solution* exhibits a yellowish-brown band corresponding in R_f and color to the band due to hesperidin in *Standard solution A* and *Standard solution B*, a yellow band below hesperidin, a blue band below the yellow band, another blue band above hesperidin, and a light yellowish-brown band between hesperidin and the blue band above corresponding in R_f and color to the same bands in *Standard solution B*. In the upper-third section, the *Sample solution* exhibits a bright blue band and a faint green band above the bright blue band corresponding in R_f and color to the same bands in *Standard solution B*. In the lower-third section, the *Sample solution* exhibits a couple of yellow bands close to the starting position and a couple of faint bands above the yellow bands corresponding in R_f and color to the same bands in *Standard solution B*.

• B. HPLC

Analysis: Proceed as directed in the test for *Content of Dihydroflavone Glycosides and Polymethoxylated Flavones*.

Acceptance criteria: The *Sample solution* exhibits the most intense peak at the retention time corresponding to hesperidin in *Standard solution A* and peaks due to narirutin, didymin, nobiletin, 3,5,6,7,8,3',4'-heptamethoxyflavone, and tangeretin at retention times corresponding

to the same constituents in *Standard solution B*. No other peak between narirutin and tangeretin is more intense than the peak corresponding to didymnin (a distinction from other *Citrus* species; *Citrus maxima* peel and *Citrus wilsonii* fruit show a principal peak for naringin). The content ratios of narirutin and didymnin relative to hesperidin are within the ranges listed in [Table 2](#).

COMPOSITION

• CONTENT OF DIHYDROFLAVONE GLYCOSIDES AND POLYMETHOXYLATED FLAVONES

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	85	15
8	81	19
10	81	19
17	60	40
28	56	44

[NOTE—The *Standard solutions* and the *Sample solution* are stable for 24 h at room temperature.]

Standard solution A: 0.40 mg/mL of [USP Hesperidin RS](#) in [methanol](#)

Standard solution B: 0.05 mg/mL of [USP Nobiletin RS](#) in [methanol](#)

Standard solution C: 5 mg/mL of [USP Citrus reticulata Pericarp Dry Extract RS](#) in [methanol](#). Sonicate for 15 min, centrifuge, and pass through a suitable membrane filter of 0.22-μm pore size.

Sample solution: Accurately transfer about 100 mg of finely powdered Tangerine Peel to a suitable flask, accurately add 10.0 mL of [methanol](#), and close tightly. Weigh the filled flask accurately and sonicate for 30 min. Cool to room temperature and adjust to the initial weight by adding [methanol](#) if needed. Before injection, pass through a suitable membrane filter of 0.22-μm pore size and discard the first portion of the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 283 nm (0–17 min) and 330 nm (17–28 min)

Column: 4.6-mm × 5-cm; 1.8-μm packing [L1](#)

Column temperature: 25°

Flow rate: 0.7 mL/min

Injection volume: 2 μL

System suitability

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

Suitability requirements

Resolution: NLT 1.5 between the peak of hesperidin and the small peak before it, *Standard solution C*

Tailing factor: NMT 1.5 for the hesperidin and nobiletin peaks, *Standard solution A* and *Standard solution B*

Relative standard deviation: NMT 2.0% for the hesperidin and nobiletin peaks in repeated injections, *Standard solution A* and *Standard solution B*

Chromatographic similarity: The chromatogram of *Standard solution C* is similar to the reference chromatogram provided with the lot of [USP Citrus reticulata Pericarp Dry Extract RS](#) being used.

Analysis

For dihydroflavone glycosides

Samples: *Standard solution A*, *Standard solution C*, and *Sample solution*

Using the chromatograms of *Standard solution A*, *Standard solution C*, and the reference chromatogram provided with the lot of [USP Citrus reticulata Pericarp Dry Extract RS](#) being used, identify the peaks corresponding to narirutin, hesperidin, and didymnin in the *Sample solution*. [NOTE—See [Table 2](#) for the relative retention times.]