Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-May-2018
Document Type: Dietary Supplement Monographs
DocId: GUID-BB0E4C10-0FC8-4660-BBFD-9A97427A1065_3_en-US
DOI: https://doi.org/10.31003/USPNF_M10457_03_01
DOI Ref: yiu5p

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Tangerine Peel Powder

DEFINITION

Tangerine Peel Powder 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, reduced to a fine or very fine powder. 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 Powder in 5 mL of methanol. Sonicate for 20 min, centrifuge, and use the supernatant.

Chromatographic system

Adsorbent: Chromatographic silica gel F_{254} 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*.

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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 didymin (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 didymin 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 Tangerine Peel Powder 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*

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