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Maritime Pine Extract

DEFINITION

Maritime Pine Extract is prepared from the pulverized Maritime Pine using suitable solvents. It contains NLT 65% and NMT 75% of procyanidins, calculated on the dried basis.

IDENTIFICATION

• A. PRESENCE OF PROCYANIDINS

Sample solution: Dissolve 50 mg of Extract in 6 mL of a mixture of butanol and hydrochloric acid (95:5).

Analysis: Heat for 2 min in a water bath.

Acceptance criteria: The solution turns red.

• B. THIN-LAYER CHROMATOGRAPHIC IDENTIFICATION TEST

Standard solution A: 25 mg/mL of [USP Maritime Pine Extract RS](#) in methanol

Standard solution B: 1 mg/mL each of ferulic acid and protocatechuic acid in methanol

Sample solution: 25 mg/mL of Extract in methanol

Chromatographic system

(See [Chromatography \(521\)](#), [Thin-Layer Chromatography](#).)

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 5 µL

Developing solvent system: Methylene chloride, methanol, glacial acetic acid, and water (80:15:2:2)

Spray reagent: 5% ferric chloride solution in methanol

Analysis

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

Develop the chromatogram, dry the plate at 110°, and examine the plate under short-wavelength and long-wavelength UV light. The chromatograms of *Standard solution A* and *Standard solution B* exhibit bands in the middle third and upper third that correspond to protocatechuic acid and ferulic acid, respectively. Spray the plate with the *Spray reagent*, and heat at 115° for 15 min. The bands due to ferulic acid and protocatechuic acid turn grayish green. Grayish-green bands become visible in the chromatogram of *Standard solution A* above and below protocatechuic acid, indicating the presence of caffeic acid and catechin, respectively.

Acceptance criteria: The chromatogram of the *Sample solution* exhibits bands due to catechin, protocatechuic acid, caffeic acid, and ferulic acid that correspond in color and R_f values to those in the chromatogram of *Standard solution A* and *Standard solution B*.

• C. THIN-LAYER CHROMATOGRAPHIC IDENTIFICATION TEST

Standard solution: Use *Standard solution A*, prepared as directed for *Identification test B*.

Sample solution: Use the *Sample solution*, prepared as directed for *Identification test B*.

Chromatographic system

(See [Chromatography \(521\)](#), [Thin-Layer Chromatography](#).)

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 5 µL

Developing solvent system: Ethyl acetate, formic acid, and water (50:5:3)

Spray reagent: Phosphoric acid and alcohol (1:1), containing 1% of vanillin

Analysis

Samples: *Standard solution* and *Sample solution*

Proceed as directed in the chapter, except to dry the plate with the aid of a current of air, spray the plate with the *Spray reagent*, and heat at 115° for 15 min. Three red bands appear in the middle third of the chromatogram of the *Standard solution* corresponding to two dimeric procyanidins and catechin. The chromatogram of the *Standard solution* also exhibits a blue band between the upper band due to upper dimeric procyanidins and the band due to catechin.

Acceptance criteria: The chromatogram of the *Sample solution* contains bands that correspond to those found in the chromatogram of the *Standard solution*.

• **D. HPLC IDENTIFICATION TEST**

Solution A: Methanol

Solution B: 1 mg/mL of phosphoric acid in water

Mobile phase: See [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	8	92
40	34	66
45	2	98
50	2	98
52	8	92
57	8	92

Standard solution: 2 mg/mL of [USP Maritime Pine Extract RS](#) in *Solution A*. Pass through a membrane having a 0.45-µm or finer pore size.

Sample solution: Add 20 mg of Extract to 10 mL of *Solution A*, and sonicate for 10 min to dissolve. Pass through a membrane having a 0.45-µm or finer pore size, discarding the first 4 mL of the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 15-cm; base-deactivated packing L7, having less than 5-µm particle size

Column temperature: 40°

Flow rate: 1 mL/min

Injection size: 10 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Chromatogram similarity: The chromatogram is similar to the reference chromatogram provided with the lot of [USP Maritime Pine Extract RS](#) being used.

Resolution: NLT 3.0 between taxifolin and ferulic acid

Tailing factor: NMT 2.0 for taxifolin

Analysis

Samples: *Standard solution* and *Sample solution*

Identify the peaks for catechin, caffeic acid, taxifolin, and ferulic acid by comparison of the chromatogram of the *Standard solution* with the reference chromatogram.

Acceptance criteria: The chromatogram of the *Sample solution* exhibits peaks for catechin, caffeic acid, taxifolin, and ferulic acid at the retention times corresponding to those in the chromatogram of the *Standard solution*.

COMPOSITION

• **CONTENT OF PROCYANIDINS**

Reagent solution A: Butanol and hydrochloric acid (95:5)

[NOTE—Prepare this solution on the day of use.]

Reagent solution B: Dissolve 2 g of ferric ammonium sulfate in a mixture of 100 mL of water and 17.5 mL of hydrochloric acid. [NOTE—This solution can be used within 15 days of preparation.]

Standard solution: 95 µg/mL of procyanidins from [USP Maritime Pine Extract RS](#) in methanol

Sample stock solution: Transfer 250 mg of Extract to a 100-mL volumetric flask. Dissolve with methanol, and dilute with the same solvent to volume.

Sample solution: Dilute the *Sample stock solution* with methanol (1 in 20).