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Capsicum Oleoresin

DEFINITION

Capsicum Oleoresin is an alcoholic extract of the dried ripe fruits of Capsicum. It contains NLT 6.5% of total capsaicinoids, calculated as the sum of capsaicin, dihydrocapsaicin, nordihydrocapsaicin, nonivamide, decanylvanillinamide, and homocapsaicin, all calculated on the anhydrous basis. The nonivamide content is NMT 5% of the total capsaicinoids, calculated on the anhydrous basis.

[**Caution—**Capsicum Oleoresin is a powerful irritant, and even in minute quantities produces an intense burning sensation when it comes in contact with the eyes and tender parts of the skin. Care should be taken to protect the eyes and to prevent contact of the skin with Capsicum Oleoresin.]

IDENTIFICATION

• **A. Thin-Layer Chromatography**

Standard solution A: 0.4 mg/mL of USP Capsaicin RS in methanol

Standard solution B: 0.4 mg/mL of USP Dihydrocapsaicin RS in methanol

Sample solution: 10 mg/mL of Capsicum Oleoresin in hexanes

Chromatographic system

Adsorbent: Chromatographic reverse phase octadecyl silyl silica gel with an average particle size of 5 µm (HPTLC plates)

Application volume: 2 µL, as 8-mm bands

Developing solvent system: A mixture of methanol and water (8:2)

Derivatization reagent A: 0.25 mg/mL of dichloroquinonechlorimide in ethyl acetate

Derivatization reagent B: Ammonium hydroxide solution

Analysis

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

Apply the *Samples* as bands to a suitable high-performance thin-layer chromatographic plate. Use a saturated chamber, and condition the plate to a relative humidity of about 33% using a suitable device. Develop the chromatograms over a distance of 6 cm. Remove the plate from the chamber, dry, derivatize with *Derivatization reagent A*, dry, expose to vapors of *Derivatization reagent B* until blue bands develop, and examine under white light.

System suitability: *Standard solution A* shows a blue band at about one-third of the chromatogram, and *Standard solution B* shows a blue band at an R_F right below that from *Standard solution A*.

Acceptance criteria: The *Sample solution* exhibits a blue band at about one-third of the chromatogram, similar in position and color to the capsaicin band in the chromatogram of *Standard solution A*, and exhibits a blue band at an R_F right below that of capsaicin, similar in position and color to the dihydrocapsaicin band in the chromatogram of *Standard solution B*. Other bands may be observed in the *Sample solution* chromatogram.

• **B. HPLC**

Analysis: Proceed as directed in the test for *Content of Total Capsaicinoids*.

Acceptance criteria: The *Sample solution* chromatogram exhibits the main capsaicinoid peak at the retention time corresponding to capsaicin in the chromatogram of *Standard solution A* and a peak of lower intensity corresponding to dihydrocapsaicin in the chromatogram of *Standard solution B*. The *Sample solution* chromatogram shows additional minor peaks corresponding to nordihydrocapsaicin, nonivamide, decanylvanillinamide, and homocapsaicin.

ASSAY

• **Content of Total Capsaicinoids**

Mobile phase: A mixture of acetonitrile and diluted phosphoric acid (1 in 1000) (2:3)

Standard solution A: 0.2 mg/mL of USP Capsaicin RS in methanol

Standard solution B: 0.1 mg/mL of USP Dihydrocapsaicin RS in methanol

Sample solution: 5 mg/mL of Capsicum Oleoresin in methanol. Pass a portion of this solution through a filter of 0.2-µm pore size, and use the filtrate as the *Sample solution*.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 281 nm

Column: 4.6-mm × 25-cm; end-capped, 5-µm, 150 Å, packing L11

Column temperature: 30°

Flow rate: 1.0 mL/min

Injection volume: 20 µL

System suitability

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

Suitability requirements

Resolution: NLT 1.5 between the capsaicin peak and the nonivamide peak that occurs at a retention time of 0.95 relative to 1.0 for capsaicin, *Sample solution*

Relative standard deviation: NMT 2.0% for the capsaicin peak, *Standard solution A*

Analysis

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

Identify the capsaicin and dihydrocapsaicin peaks in the *Sample solution* chromatogram by comparison with the chromatograms of *Standard solution A* and *Standard solution B*, respectively. Identify the peaks corresponding to nordihydrocapsaicin, nonivamide, decanylvanillinamide, and homocapsaicin using the approximate relative retention times provided in *Table 1*.

Table 1

Analyte	Approximate Relative Retention Time
Nordihydrocapsaicin	0.89
Nonivamide	0.95
Capsaicin	1.00
Decanylvanillinamide	1.34
Homocapsaicin	1.40

Calculate the percentage of capsaicin in the portion of Capsicum Oleoresin taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak area of capsaicin from the *Sample solution*

r_S = peak area of capsaicin from *Standard solution A*

C_S = concentration of capsaicin in *Standard solution A* (mg/mL)

C_U = concentration of Capsicum Oleoresin in the *Sample solution* (mg/mL)

Calculate the percentage of dihydrocapsaicin in the portion of Capsicum Oleoresin taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak area of dihydrocapsaicin from the *Sample solution*

r_S = peak area of dihydrocapsaicin from *Standard solution B*

C_S = concentration of dihydrocapsaicin in *Standard solution B* (mg/mL)

C_U = concentration of Capsicum Oleoresin in the *Sample solution* (mg/mL)

Calculate the percentage of nonivamide, expressed as capsaicin, in the portion of Capsicum Oleoresin taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak area of nonivamide from the *Sample solution*

r_S = peak area of capsaicin from *Standard solution A*

C_S = concentration of capsaicin in *Standard solution A* (mg/mL)

C_U = concentration of Capsaicin Oleoresin in the *Sample solution* (mg/mL)

Calculate the sum of the percentages of nordihydrocapsaicin, decanylvanillinamide, and homocapsaicin, expressed as capsaicin, in the portion of Capsicum Oleoresin taken:

$$\text{Result} = (\Sigma r_U / r_S) \times (C_S / C_U) \times 100$$

Σr_U = sum of the peak areas of nordihydrocapsaicin, decanylvanillinamide, and homocapsaicin from the *Sample solution*

r_S = peak area of capsaicin from *Standard solution A*

C_S = concentration of capsaicin in *Standard solution A* (mg/mL)

C_U = concentration of Capsicum Oleoresin in the *Sample solution* (mg/mL)

Calculate the content of total capsaicinoids as the sum of the percentages of capsaicin, dihydrocapsaicin, nordihydrocapsaicin, nonivamide, decanylvanillinamide, and homocapsaicin.

Acceptance criteria: NLT 6.5% on the anhydrous basis

CONTAMINANTS

• **Elemental Impurities—Procedures ⟨233⟩**

Acceptance criteria

Arsenic: NMT 0.5 µg/g

Cadmium: NMT 1.0 µg/g

Lead: NMT 5.0 µg/g

Mercury: NMT 0.1 µg/g

Change to read:

• **▲Articles of Botanical Origin,** *Pesticide Residue Analysis* ⟨561⟩▲ (CN 1-May-2019) **:** Meets the requirements

• **Articles of Botanical Origin,** *Test for Aflatoxins* ⟨561⟩**:** Meets the requirements

SPECIFIC TESTS

• **Limit of Nonivamide**

Analysis: Use the chromatograms and calculations obtained in the test for *Content of Total Capsaicinoids*.

Calculate the content of nonivamide as percentage of total capsaicinoids:

$$\text{Result} = (PN/PTC) \times 100$$

PN = percentage of nonivamide as calculated in the test for *Content of Total Capsaicinoids*

PTC = percentage of total capsaicinoids as calculated in the test for *Content of Total Capsaicinoids*

Acceptance criteria: NMT 5% on the anhydrous basis

• **Water Determination,** *Method Ia* ⟨921⟩

Sample: 5.0 g of Capsicum Oleoresin

Acceptance criteria: NMT 8%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

• **Labeling:** Label it to indicate that if separation occurs, it should be warmed and mixed before use.

• **USP Reference Standards ⟨11⟩**

USP Capsaicin RS

USP Dihydrocapsaicin RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
CAPSICUM OLEORESIN	Nam-Cheol Kim Scientific Liaison	BDSHM2020 Botanical Dietary Supplements and Herbal Medicines

Chromatographic Database Information: Chromatographic Database

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