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# Powdered *Echinacea purpurea*

**DEFINITION**

Powdered *Echinacea purpurea* consists of the dried rhizome and roots of *Echinacea purpurea* (L.) Moench (Fam. Asteraceae), harvested in the fall after three or more years of growth, and reduced to powder. It contains NLT 0.5% of total phenols, calculated on the dried basis as the sum of caftaric acid ($C_{13}H_{12}O_9$), chicoric acid ($C_{22}H_{18}O_{12}$), and chlorogenic acid ($C_{16}H_{18}O_9$). It contains NLT 0.025% of alkamides calculated as dodecatetraenoic acid isobutylamides ($C_{16}H_{25}NO$).

**IDENTIFICATION**

• **A. Thin-Layer Chromatography**

**Presence of chicoric acid and absence of echinacoside**

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

**Standard solution B:** 0.2 mg/mL of USP Caftaric Acid RS, 0.1 mg/mL of USP Chlorogenic Acid RS, and 0.2 mg/mL of USP Chicoric Acid RS in methanol

**Standard solution C:** 20 mg/mL of USP Powdered *Echinacea purpurea* Extract RS in methanol. Shake to disperse, sonicate for 5 min, and centrifuge. Use the supernatant.

**Sample solution:** Transfer 1 g of Powdered *Echinacea purpurea* to a centrifuge tube, add 10 mL of methanol, mix well, and sonicate for 10 min. Centrifuge, and use the supernatant.

**Chromatographic system**

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

**Adsorbent:** Chromatographic silica gel mixture with an average particle size of 5 µm (HPTLC plates)

**Application volume:** 5 µL *Standard solution C* and *Sample solution*, and 2 µL *Standard solution A* and *Standard solution B* as 8-mm bands

**Relative humidity:** Condition the plate to a relative humidity of about 33% using a suitable device.

**Developing solvent system:** A mixture of ethyl acetate, methylethyl ketone, water, and formic acid (5:3:1:1)

**Developing distance:** 6 cm

**Derivatization reagent:** 5 mg/mL of 2-aminoethyl diphenylborinate in ethyl acetate

**Analysis**

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

Apply the *Samples* as bands to a suitable thin-layer chromatographic plate, and dry in air. Develop the chromatograms in a saturated chamber. Remove the plate from the chamber, heat at 100° for 5 min, derivatize the plate while still warm with *Derivatization reagent*, dry in air, and examine under UV light at 366 nm.

**System suitability:** *Standard solution A* shows one major blue band in the lower third section of the chromatogram due to echinacoside. *Standard solution B* shows two major blue bands at about the middle of the chromatogram due to caftaric acid (higher $R_F$) and chlorogenic acid (lower $R_F$) that are clearly separated, and a blue band for chicoric acid in the upper third section of the chromatogram.

**Acceptance criteria:** The most prominent band in the *Sample solution* chromatogram is a blue band in the upper third section of the chromatogram at an $R_F$ corresponding to the chicoric acid band in the chromatograms of *Standard solution B* and *Standard solution C* (less prominent in *Echinacea pallida* and absent or almost absent in *Echinacea angustifolia*). The second most prominent band in the *Sample solution* chromatogram is a blue band at about the middle of the chromatogram due to caftaric acid, corresponding to a band in the chromatogram of *Standard solution C* (absent in *Echinacea angustifolia* and a minor band in *Echinacea pallida*). The *Sample solution* chromatogram does not exhibit a band at the $R_F$ of echinacoside in *Standard solution A* (difference from *Echinacea pallida* and *Echinacea angustifolia*). The *Sample solution* chromatogram may exhibit minor blue bands corresponding to similar bands in the chromatogram of *Standard solution C*. One of these is due to chlorogenic acid at an $R_F$ corresponding to chlorogenic acid in the *Standard solution B*.

• **B. Thin-Layer Chromatography**

**Presence of alkylamides**

**Standard solution A:** 0.2 mg/mL of USP β-Sitosterol RS in methanol

**Standard solution B:** 100 mg/mL of USP Powdered *Echinacea purpurea* Extract RS in dichloromethane. Shake to disperse, sonicate for 5 min, and centrifuge. Use the supernatant.

**Sample solution:** Transfer 1 g of Powdered *Echinacea purpurea* to a centrifuge tube, add 10 mL of dichloromethane, mix well, and sonicate for 10 min. Centrifuge, and use the supernatant.

**Chromatographic system**

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

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**Adsorbent:** Chromatographic silica gel with an average particle size of 5 µm (HPTLC plates)

**Application volume:** 5 µL *Standard solution B* and *Sample solution*, and 2 µL *Standard solution A* as 8-mm bands

**Relative humidity:** Condition the plate to a relative humidity of about 33% using a suitable device.

**Developing solvent system:** A mixture of toluene, ethyl acetate, cyclohexane, and formic acid (8: 2: 1: 0.3)

**Developing distance:** 6 cm

**Derivatization reagent:** Place 85 mL of methanol in a 100-mL glass bottle and cool it down in a water–ice cubes–salt bath or in a freezer. To the ice-cold methanol, slowly and carefully add 10 mL of acetic acid and 5 mL of sulfuric acid, and mix well. Allow the mixture to cool to room temperature, then add 0.5 mL of *p*-anisaldehyde.

**Analysis**

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

Apply the *Samples* as bands to a suitable thin-layer chromatographic plate, and dry in air. Condition the plate to a relative humidity of about 33% using a suitable device. Develop the chromatograms in a saturated chamber. Remove the plate from the chamber, dry in air, derivatize with *Derivatization reagent*, heat at 100° for 3–5 min, set aside to cool, and examine under visible light.

**System suitability:** The chromatogram of *Standard solution B* exhibits the most prominent band as a pinkish violet band at about the middle of the chromatogram, and just below this pinkish band, a violet band at a lower $R_F$ similar in position and color to the β-sitosterol band in the chromatograms of *Standard solution A*. These two bands are clearly separated from each other. The chromatogram of *Standard solution B* also shows a broad pink violet band close to the solvent front.

**Acceptance criteria:** The most prominent band of the *Sample solution* chromatogram is a pinkish violet band at about the middle of the chromatogram similar in position and color to a band in the *Standard solution B* chromatogram (much less prominent in *Echinacea angustifolia* and *Echinacea pallida*), a violet band corresponding to β-sitosterol band in the chromatograms of *Standard solution A* and *Standard solution B*, and a broad pink violet band close to the solvent front similar in position and color to the band in the chromatogram of *Standard solution B*. The *Sample solution* chromatogram does not exhibit a yellow band below the β-sitosterol band (difference from *Echinacea angustifolia*) or a prominent violet band at about two thirds of the chromatogram (difference from *Echinacea pallida*).

• **C.** The retention time of the major peak in the *Sample solution* corresponds to that of the chicoric acid peak in *Standard solution A*, and the second most prominent peak corresponds to that of the caftaric acid peak in *Standard solution B*. The *Sample solution* chromatogram shows no or a very minor peak at the retention time corresponding to the echinacoside peak in the *Standard solution D* chromatogram. All peaks as obtained in the test for *Content of Total Phenols*.

**COMPOSITION**

• **Content of Total Phenols**

**Solution A:** Phosphoric acid (0.1 in 100) in water

**Solution B:** Acetonitrile

**Mobile phase:** See *Table 1*.

**Table 1**

| Time (min) | Solution A (%) | Solution B (%) |
|---|---|---|
| 0 | 90 | 10 |
| 13 | 78 | 22 |
| 14 | 60 | 40 |
| 17.5 | 60 | 40 |
| 18 | 90 | 10 |
| 30 | 90 | 10 |

**Solvent:** Alcohol and water (7:3)

**Standard solution A:** 30 µg/mL of USP Chicoric Acid RS in *Solvent*

**Standard solution B:** 20 µg/mL of USP Caftaric Acid RS in *Solvent*

**Standard solution C:** 20 µg/mL of USP Chlorogenic Acid RS in *Solvent*

**Standard solution D:** 20 µg/mL of USP Echinacoside RS in *Solvent*

**Sample solution:** Transfer about 125 mg of Powdered *Echinacea purpurea* (capable of passing through a 40-mesh sieve), accurately weighed, to a round-bottom flask equipped with a condenser. Add 25.0 mL of *Solvent*, and heat under reflux while shaking by mechanical means for 15 min. Centrifuge, or pass through a membrane filter of 0.45-µm or finer pore size.

**Chromatographic system**

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

**Mode:** LC

**Detector:** UV 330 nm

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