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Bromocriptine Mesylate Capsules

DEFINITION

Bromocriptine Mesylate Capsules contain bromocriptine mesylate ($C_{32}H_{40}BrN_5O_5 \cdot CH_4SO_3$) equivalent to NLT 90.0% and NMT 110.0% of the labeled amount of bromocriptine ($C_{32}H_{40}BrN_5O_5$).

IDENTIFICATION

• **A.** The principal spot of the *Sample solution* corresponds, in R_F value and color, to that of the *Standard solution*, as obtained in the test for *Organic Impurities*.

ASSAY

• **Procedure**

Conduct this procedure without exposure to daylight and with minimum exposure to artificial light.

Buffer: 0.125 g/L of ammonium carbonate in water

Mobile phase: Acetonitrile and *Buffer* (3:2)

Standard solution: 1.0 mg/mL of bromocriptine from USP Bromocriptine Mesylate RS in dehydrated alcohol. Sonicate as needed.

Sample solution: 1.0 mg/mL of bromocriptine in methanol, prepared as follows. Remove, as completely as possible, the contents of NLT 10 Capsules. Weigh and determine the average weight per Capsule. Mix the combined contents, and transfer a weighed quantity of the powder, nominally equivalent to 50 mg of bromocriptine, to a 50-mL volumetric flask. Add 30 mL of dehydrated alcohol, and shake for 15 min. Dilute with dehydrated alcohol to volume, mix, and filter. Use this solution without delay.

Chromatographic system

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

Mode: LC

Detector: UV 300 nm

Column: 4-mm × 25-cm; packing L7

Flow rate: 2 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Column efficiency: NLT 1000 theoretical plates

Tailing factor: NMT 2.0

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount bromocriptine ($C_{32}H_{40}BrN_5O_5$) in the portion of Capsules taken:

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

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of bromocriptine, from USP Bromocriptine Mesylate RS, in the *Standard solution* (mg/mL)

C_U = nominal concentration of bromocriptine in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Medium: 0.1 N hydrochloric acid; 500 mL

Apparatus 2: 50 rpm

Time: 60 min

Standard solution: USP Bromocriptine Mesylate RS in *Medium*, at a concentration similar to the *Sample solution*. [NOTE—A volume of alcohol not to exceed 5% of the total volume of the *Standard solution* may be used to bring the Standard into solution before dilution with *Medium*.]

Sample solution: Sample per *Dissolution ⟨711⟩*, passed through a glass-fiber filter.

Instrumental conditions

(See *Fluorescence Spectroscopy ⟨853⟩*.)

Mode: Fluorometry

Excitation wavelength: 315 nm

Emission wavelength: 445 nm

Blank: *Medium*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of bromocriptine ($C_{32}H_{40}BrN_5O_5$) dissolved.

Tolerances: NLT 75% (*Q*) of the labeled amount of bromocriptine ($C_{32}H_{40}BrN_5O_5$) is dissolved.

Change to read:

• **UNIFORMITY OF DOSAGE UNITS ⟨905⟩:** ▲Meet the requirements▲ (CN 1-Aug-2023)

Procedure for content uniformity

Protect all solutions from light.

Diluent: Dissolve 1.0 g of tartaric acid in 500 mL of water, add 500 mL of methanol, and mix.

Standard solution: 0.04 mg/mL of USP Bromocriptine Mesylate RS in *Diluent*

Sample solution: Transfer the contents of 1 Capsule into a 25-mL volumetric flask. Add 15 mL of *Diluent*, and shake by mechanical means for 20 min. Dilute with *Diluent* to volume, and mix. Filter, and dilute 10.0 mL of the clear filtrate with *Diluent* to 50.0 mL.

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy ⟨857⟩*.)

Mode: UV

Analytical wavelength: Maximum absorbance (about 306 nm)

Cell: 1 cm

Blank: *Diluent*

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*

Calculate the percentage of the labeled amount of bromocriptine ($C_{32}H_{40}BrN_5O_5$) in the Capsule taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of USP Bromocriptine Mesylate RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of bromocriptine in the *Sample solution* (mg/mL)

M_{r1} = molecular weight of bromocriptine, 654.59

M_{r2} = molecular weight of bromocriptine mesylate, 750.70

▲▲ (CN 1-Aug-2023)

IMPURITIES

• **ORGANIC IMPURITIES**

Conduct this test without exposure to daylight and with minimum exposure to artificial light. Perform the test rapidly, preparing and spotting the *Sample solution* last.

Standard stock solution: 2.3 mg/mL of USP Bromocriptine Mesylate RS in methanol, equivalent to 2 mg/mL of bromocriptine

Standard solution 1: 0.06 mg/mL (3.0%) of bromocriptine in methanol, from *Standard stock solution*

Standard solution 2: 0.04 mg/mL (2.0%) of bromocriptine in methanol, from *Standard stock solution*

Standard solution 3: 0.02 mg/mL (1.0%) of bromocriptine in methanol, from *Standard stock solution*

Standard solution 4: 0.01 mg/mL (0.50%) of bromocriptine in methanol, from *Standard stock solution*

Sample solution: 2.0 mg/mL of bromocriptine in methanol, prepared as follows. Transfer a quantity of the Capsule contents, equivalent to 20 mg of bromocriptine, to a conical flask. Add 10 mL of methanol, and stir by mechanical means for 20 min. Centrifuge the suspension for 10 min at about 3500 rpm. Use the clear supernatant.

Chromatographic system

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

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 50 µL as 1.5-cm bands

Developing solvent: Methylene chloride, dioxane, alcohol, and ammonium hydroxide (180:15:5:1)

Spray reagent: 0.2% *o*-phthalaldehyde in sulfuric acid

Analysis

Samples: *Standard stock solution*, *Standard solutions*, and *Sample solution*

Develop under the exclusion of light in a tank lined with filter paper, previously equilibrated for 30 min, using *Developing solvent* until the solvent front has moved a distance of 15 cm on the plate. Dry the plate briefly in a current of cold air. Spray evenly with the *Spray reagent*, and view the plate under long-wavelength UV light.

Acceptance criteria: Any major secondary spot, other than the principal spot, obtained from the *Sample solution* is not greater in size and intensity than the spot obtained from *Standard solution 1* (3.0%). Any remaining spots are not greater in size and intensity than the spot obtained from *Standard solution 3* (1.0%). The sum of the organic impurities is NMT 5.0%.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light-resistant containers.
- **USP Reference Standards** ⟨11⟩

  USP Bromocriptine Mesylate RS

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

Topic/Question	Contact	Expert Committee
BROMOCRIPTINE MESYLATE CAPSULES	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

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