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Bromocriptine Mesylate Tablets

DEFINITION

Bromocriptine Mesylate Tablets 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 stock solution*, as obtained in the test for *Organic Impurities*.

ASSAY

• **Procedure**

Buffer: 0.01 M ammonium carbonate in water

Mobile phase: Acetonitrile and *Buffer* (65:35)

Standard solution: 0.22 mg/mL of USP Bromocriptine Mesylate RS in methanol

Sample solution: Transfer a quantity of powdered Tablets (NLT 20), equivalent to 10 mg of bromocriptine, to an appropriate container. Add 40 mL of methanol, and stir for 20 min, protected from light. Quantitatively filter through a fine glass filtering funnel into a 50-mL volumetric flask. Rinse the filter with methanol, adding the rinsing to the filtrate, and dilute with methanol to volume.

Chromatographic system

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

Mode: LC

Detector: UV 300 nm

Column: 4-mm × 25-cm; packing L1

Flow rate: 2 mL/min

Injection volume: 50 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Coefficient of variation: NMT 3.0% for 3 replicate injections

Analysis

Samples: *Standard solution* and *Sample solution*

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

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

r_U = peak response from the *Sample solution*

r_S = peak response from 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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Test 1: If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 1*.

Medium: 0.1 N hydrochloric acid; 500 mL

Apparatus 1: 120 rpm

Time: 60 min

Standard solution: USP Bromocriptine Mesylate RS at a known concentration in *Medium*

[Note—A volume of alcohol not to exceed 5% of the total volume of the *Standard solution* may be used to dissolve the Standard before dilution with *Medium*.]

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

Blank: *Medium*

Instrumental conditions

(See *Fluorescence Spectroscopy* ⟨853⟩.)

Mode: Fluorometry

Excitation wavelength: 315 nm

Emission wavelength: 445 nm

Analysis

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

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

Tolerances: NLT 80% (*Q*) of the labeled amount of bromocriptine ($C_{32}H_{40}BrN_5O_5$) is dissolved.

Test 2: If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 2*.

Medium: 0.1 N hydrochloric acid; 500 mL

Apparatus 2: 50 rpm

Time: 30 min

Buffer: 0.01 M ammonium carbonate in water

Mobile phase: Acetonitrile and *Buffer* (65:35)

Standard solution: Dissolve USP Bromocriptine Mesylate RS in methanol, and quantitatively dilute with *Medium* to obtain a solution having a known concentration similar to the expected concentration of the *Sample solution*.

Sample solution: Sample per *Dissolution* ⟨711⟩.

Chromatographic system

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

Mode: LC

Detector: UV 300 nm

Column: 3.9-mm × 30-cm; packing L1

Flow rate: 1.5 mL/min

Injection volume: 100 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

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 80% (*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

[Note—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 1 Tablet into a 25-mL volumetric flask. Add 15 mL of *Diluent*, and shake by mechanical means for 30 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: 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 Tablet 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**

[Note—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: 1.2 mg/mL of USP Bromocriptine Mesylate RS in methanol, equivalent to 1 mg/mL of bromocriptine

Standard solution 1: 0.50 mg/mL (5%) of bromocriptine in methanol, from *Standard stock solution*

Standard solution 2: 0.30 mg/mL (3%) of bromocriptine in methanol, from *Standard stock solution*

Standard solution 3: 0.10 mg/mL (1%) of bromocriptine in methanol, from *Standard stock solution*

Sample solution: Transfer an equivalent to 20 mg of bromocriptine, from powdered Tablets, to a conical flask. Add 10 mL of methanol, and mix for 20 min. Centrifuge the suspension for 10 min at 4000 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 volumes

Standard solutions: 10-µL, as 1.5-cm bands

Sample solution: 50-µL, as 1.5-cm bands

Developing solvent system: Methylene chloride, dioxane, alcohol, and ammonium hydroxide (180:15:5:0.1)

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

Analysis

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

Proceed as directed in *Chromatography ⟨621⟩, Thin-Layer Chromatography*. Dry the plate for 5 min in a current of cold air. Develop in a tank lined with filter paper, previously equilibrated for 20 min, using *Developing solvent system* until the solvent front has moved a distance of 10 cm on the plate. Dry the plate under vacuum at room temperature for 15 min. Spray evenly with the *Spray reagent*, and view the plate under long-wavelength UV light.

Acceptance criteria: Any spot, other than the principal spot, from the *Sample solution* is not greater in size and intensity than the spot from *Standard solution 2* (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.

• **Labeling:** The labeling indicates the *Dissolution* test with which the product complies.

• **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 TABLETS	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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