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Propantheline Bromide Tablets

DEFINITION
Propantheline Bromide Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of propantheline bromide ($C_{23}H_{30}BrNO_3$).

IDENTIFICATION

• **A.**

Standard solution: 6 mg/mL of USP Propantheline Bromide RS in chloroform

Sample solution: Triturate the equivalent to 90 mg of propantheline bromide, from finely powdered Tablets, with 10 mL of chloroform. Filter, and wash the filter with 10 mL of chloroform, collecting the filtrate and washing in a separator. Add 10 mL of water, shake, and discard the chloroform layer. Wash the aqueous layer with two 10-mL portions of ether, and discard the ether washings. Filter the aqueous solution, and evaporate on a steam bath with the aid of a current of dry air to dryness. Dissolve the residue in 5 mL of chloroform.

Analysis: In a well-ventilated hood, apply 2 mL each of the *Standard solution* and *Sample solution* dropwise to separate salt plates while continuously evaporating the solvent with the aid of an IR heat lamp and a current of dry air. Heat the residues at 105° for 15 min.

Acceptance criteria: The IR absorption spectrum of the residue on the single salt plate exhibits maxima only at the same wavelengths as those of a similar preparation of USP Propantheline Bromide RS, treated in the same manner.

• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Buffer: 17.3 g of sodium dodecyl sulfate in 1000 mL of water containing 10 mL of phosphoric acid in a 2000-mL volumetric flask. Add 250 mL of 0.5 M sodium hydroxide, and while stirring, adjust with 0.5 M sodium hydroxide or dilute phosphoric acid (1 in 10) to a pH of 3.5 ± 0.05. Dilute with water to volume.

Mobile phase: Acetonitrile and *Buffer* (55:45)

Standard solution: 0.3 mg/mL of USP Propantheline Bromide RS in *Mobile phase*

Sample solution: Weigh and finely powder NLT 20 Tablets. Transfer a portion of the powder equivalent to 15 mg of propantheline bromide to a 50-mL volumetric flask, and dissolve in *Mobile phase*. Dilute with *Mobile phase* to volume, and filter.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; packing L7

Flow rate: 2.0 mL/min

Injection volume: 50 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 propantheline bromide ($C_{23}H_{30}BrNO_3$) in the portion of Tablets 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 USP Propantheline Bromide RS in the *Standard solution* (mg/mL)

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution,** *Procedure for a Pooled Sample* ⟨711⟩

Medium: pH 4.5 ± 0.05 acetate buffer prepared by mixing 1.64 g of anhydrous sodium acetate and 1.25 mL of glacial acetic acid with 500 mL of water, and diluting with water to obtain 1000 mL of solution having a pH of 4.50 ± 0.05; 500 mL

Apparatus 2: 50 rpm

Time: 45 min

Standard solution: USP Propantheline Bromide RS at a known concentration in *Medium*

Sample solution: Filtered portion of solution under test

Buffer, Mobile phase, Chromatographic system, and **System suitability:** Prepare as directed in the *Assay*.

Analysis: Determine the amount of propantheline bromide ($C_{23}H_{30}BrNO_3$) dissolved by proceeding as directed in the *Assay.*

Tolerances: NLT 75% (*Q*) of the labeled amount of propantheline bromide ($C_{23}H_{30}BrNO_3$) is dissolved.

• **Uniformity of Dosage Units** ⟨905⟩**:** Meet the requirements

IMPURITIES

• **Organic Impurities**

Buffer: 17.3 g of sodium dodecyl sulfate in 1000 mL of water containing 10 mL of phosphoric acid in a 2000-mL volumetric flask. Add 250 mL of 0.5 M sodium hydroxide, and while stirring, adjust with 0.5 M sodium hydroxide or dilute phosphoric acid (1 in 10) to a pH of 3.5 ± 0.05. Dilute with water to volume.

Mobile phase: Acetonitrile and *Buffer* (55:45)

Standard solution: 12.0 µg/mL of USP Propantheline Bromide Related Compound A RS, 3.0 µg/mL of USP Xanthanoic Acid RS, and 3.0 µg/mL of USP Xanthone RS in *Mobile phase*

Sample solution: Prepare as directed in the *Assay*.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; packing L7

Flow rate: 2.0 mL/min

Injection volume: 50 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Resolution: NLT 1.2 between the least resolved peaks

Relative standard deviation: NMT 6.0% for each component

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of xanthanoic acid, xanthone, and propantheline bromide related compound A in the portion of Tablets taken:

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

r_U = peak response for xanthanoic acid, xanthone, or propantheline bromide related compound A from the *Sample solution*

r_S = peak response for xanthanoic acid, xanthone, or propantheline bromide related compound A from the *Standard solution*

C_S = concentration of xanthanoic acid, xanthone, or propantheline bromide related compound A in the *Standard solution* (µg/mL)

C_U = nominal concentration of propantheline bromide in the *Sample solution* (µg/mL)

Acceptance criteria: NMT 4.0% of propantheline bromide related compound A; NMT 1.0% each of xanthone and xanthanoic acid. Disregard any peak less than 0.1%.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

• **USP Reference Standards ⟨11⟩**

USP Propantheline Bromide RS

USP Propantheline Bromide Related Compound A RS

9-Hydroxypropantheline bromide.
$C_{23}H_{30}BrNO_4$ 464.39

USP Xanthanoic Acid RS $C_{14}H_{10}O_3$ 226.23

USP Xanthone RS $C_{13}H_8O_2$ 196.21

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

Topic/Question	Contact	Expert Committee
PROPANTHELINE BROMIDE TABLETS	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

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