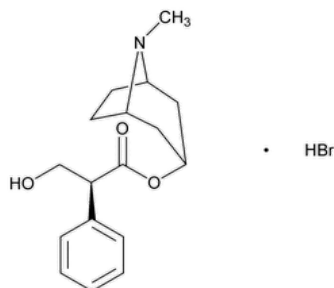


Status: Currently Official on 15-Feb-2025  
 Official Date: Official Prior to 2013  
 Document Type: USP Monographs  
 DocId: GUID-87DAF6FA-B000-404B-B76B-6511B7288571\_1\_en-US  
 DOI: [https://doi.org/10.31003/USPNF\\_M39650\\_01\\_01](https://doi.org/10.31003/USPNF_M39650_01_01)  
 DOI Ref: h47tf

© 2025 USPC  
 Do not distribute

## Hyoscyamine Hydrobromide



$C_{17}H_{23}NO_3 \cdot HBr$  370.28

Benzeneacetic acid,  $\alpha$ -(hydroxymethyl)-, 8-methyl-8-azabicyclo[3.2.1]oct-3-yl ester, hydrobromide [3(S)-endo]-;

1 $\alpha$ H,5 $\alpha$ H-Tropan-3 $\alpha$ -ol (-)-tropate (ester) hydrobromide CAS RN®: 306-03-6; UNII: IWT50P9S79.

### DEFINITION

Hyoscyamine Hydrobromide contains NLT 98.5% and NMT 100.5% of hyoscyamine hydrobromide ( $C_{17}H_{23}NO_3 \cdot HBr$ ), calculated on the dried basis.

[CAUTION—Handle Hyoscyamine Hydrobromide with exceptional care, since it is highly potent.]

### IDENTIFICATION

#### • A.

**Standard solution:** Transfer 36 mg of [USP Hyoscyamine Sulfate RS](#) to a 60-mL separator with the aid of 5 mL of water. Add 1.5 mL of 1 N sodium hydroxide and 10 mL of chloroform to the separator. Shake for 1 min, allow the layers to separate, and pass the chloroform extracts through a filter of about 2 g of anhydrous granular sodium sulfate supported on a pledget of glass wool. Extract the aqueous layer with two additional 10-mL portions of chloroform, filtering and combining with the main extract. Evaporate the chloroform solution, under reduced pressure, to dryness, and dissolve the residue in 10 mL of carbon disulfide.

**Sample solution:** Prepare as directed in the *Standard solution* using 30 mg of Hyoscyamine Hydrobromide.

**Acceptance criteria:** The IR absorption spectrum (determined in a 1-mm cell) of the *Sample solution* exhibits maxima only at the same wavelengths as those of the *Standard solution*.

#### • B.

**Sample solution:** 1 in 20

**Analysis:** To 1 mL of the *Sample solution*, add gold chloride TS, dropwise with shaking, until a definite precipitate separates. Add a small amount of 3 N hydrochloric acid, dissolve the precipitate with the aid of heat, and then allow the solution to cool.

**Acceptance criteria:** Lustrous reddish-brown scales that may be accompanied by reddish-brown needles are formed (distinction from atropine and scopolamine).

#### • C.

**Sample solution:** 1 in 20

**Analysis:** To the *Sample solution*, add silver nitrate TS.

**Acceptance criteria:** A yellowish-white precipitate is formed, and it is insoluble in nitric acid.

### ASSAY

#### • PROCEDURE

**Sample:** 700 mg

**Analysis:** Dissolve the *Sample* in a mixture of 50 mL of glacial acetic acid and 10 mL of mercuric acetate TS. Add 1 drop of crystal violet TS. Titrate with 0.1 N perchloric acid VS to a blue-green endpoint. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 37.03 mg of hyoscyamine hydrobromide ( $C_{17}H_{23}NO_3 \cdot HBr$ ).

**Acceptance criteria:** 98.5%–100.5% on the dried basis

### IMPURITIES

• [RESIDUE ON IGNITION \(281\)](#): NMT 0.2%

• OTHER ALKALOIDS

**Sample solution:** Dissolve 250 mg of Hyoscyamine Hydrobromide in 1 mL of 0.1 N hydrochloric acid, and dilute with water to 15 mL.

**Analysis 1:** To 5 mL of the *Sample solution*, add a few drops of platinic chloride TS.

**Acceptance criteria 1:** No precipitate is formed immediately.

**Analysis 2:** To another 5-mL portion of the *Sample solution*, add 2 mL of 6 N ammonium hydroxide.

**Acceptance criteria 2:** The mixture may develop a slight opalescence, but no turbidity or precipitate is formed immediately.

SPECIFIC TESTS

- [MELTING RANGE OR TEMPERATURE <741>](#): NLT 149°
- [OPTICAL ROTATION, Specific Rotation <781S>](#)  
**Sample solution:** 50 mg/mL in water  
**Acceptance criteria:** NLT -24°
- [LOSS ON DRYING <731>](#)  
**Analysis:** Dry at 105° for 2 h.  
**Acceptance criteria:** NMT 1.0%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers.
- [USP REFERENCE STANDARDS <11>](#)  
[USP Hyoscyamine Sulfate RS](#)

Auxiliary Information - Please [check for your question in the FAQs](#) before contacting USP.

Topic/Question	Contact	Expert Committee
HYOSCYAMINE HYDROBROMIDE	<a href="#">Documentary Standards Support</a>	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services <a href="mailto:RSTECH@usp.org">RSTECH@usp.org</a>	SM32020 Small Molecules 3

Chromatographic Database Information: [Chromatographic Database](#)

Most Recently Appeared In:

Pharmacopeial Forum: Volume No. 45(6)

Current DocID: GUID-87DAF6FA-B000-404B-B76B-6511B7288571\_1\_en-US

DOI: [https://doi.org/10.31003/USPNF\\_M39650\\_01\\_01](https://doi.org/10.31003/USPNF_M39650_01_01)

DOI ref: [h47tf](#)