744

(100) and 30 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

> Each mL of 0.1 mol/L perchloric acid VS  $= 44.04 \text{ mg of } C_{21}H_{30}BrNO_4$

Containers and storage Containers—Tight containers.

## Scopolamine Hydrobromide

臭化水素酸スコポラミン

C<sub>17</sub>H<sub>21</sub>NO<sub>4</sub>.HBr.3H<sub>2</sub>O: 438.31 (1S,2S,4R,5R,7s)-9-Methyl-3-oxa-9-azatricyclo- $[3.3.1.0^{2,4}]$ non-7-yl (2S)-3-hydroxy-2-phenylpropanoate monohydrobromide trihydrate [6533-68-2]

Scopolamine Hydrobromide, when dried, contains not less than 98.5% of C<sub>17</sub>H<sub>21</sub>NO<sub>4</sub>.HBr (mol. wt.: 384.26).

**Description** Scopolamine Hydrobromide occurs as colorless or white crystals, or white granules or powder. It is odor-

It is freely soluble in water, sparingly soluble in ethanol (95) and in acetic acid (100), and practically insoluble in diethyl ether.

Identification (1) To 1 mg of Scopolamine Hydrobromide add 3 to 4 drops of fuming nitric acid, evaporate on a water bath to dryness, and cool. Dissolve the residue in 1 mL of N, N-dimethylformamide, and add 6 drops of tetraethylammonium hydroxide TS: a red-purple color is produced.

(2) A solution of Scopolamine Hydrobromide (1 in 20) responds to the Qualitative Tests for bromide.

**Optical rotation**  $[\alpha]_D^{20}$ :  $-24.0 - 26.0^{\circ}$  (after drying, 0.5) g, water, 10 mL, 100 mm).

Melting point 195 - 199°C (after drying; previously heat the bath to 180°C).

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Scopolamine Hydrobromide in 10 mL of water: the solution is clear and colorless.

- (2) Acid—Dissolve  $0.50 \, \mathrm{g}$ of Scopolamine Hydrobromide in 15 mL of water, and add 0.50 mL of 0.02 mol/L sodium hydroxide and 1 drop of methyl red-methylene blue TS: a green color develops.
- (3) Apoatropine—Dissolve 0.20 g of Scopolamine Hydrobromide in 20 mL of water, add 0.60 mL of 0.002 mol/L potassium permanganate VS, and allow to stand for 5 minutes: the red color in the solution does not disappear.
- (4) Other alkaloids—Dissolve 0.15 g of Scopolamine Hydrobromide in 3 mL of water, and use this solution as the

- (i) To 1 mL of the sample solution add 2 to 3 drops of ammonia TS: no turbidity is produced.
- (ii) To 1 mL of the sample solution add 2 to 3 drops of potassium hydroxide TS: a transient white turbidity might be produced, and disappears clearly in a little while.

Loss on drying Not more than 13.0% [1.5 g; first dry in a desiccator (silica gel) for 24 hours, then dry at 105°C for 3 hoursl.

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.5 g of Scopolamine Hydrobromide, previously dried in 10 mL of acetic acid (100) by warming. After cooling, add 40 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

> Each mL of 0.1 mol/L perchloric acid VS  $= 38.427 \text{ mg of } C_{17}H_{21}NO_4.HBr$

Containers and storage Containers—Tight containers. Storage—Light-resistant.

## Secretin

セクレチン

His-Ser-Asp-Gly-Thr-Phe-Thr-Ser-Glu-Leu-Ser-Arg-Leu-Arg-Asp-Ser-Ala-Arg-Leu-Gln-Arg-Leu-Leu-Gin-Gly-Leu-Val-NH2

 $C_{130}H_{220}N_{44}O_{41}$ : 3055.41 [1393-25-5]

Secretin is a peptide obtained from the upper part of hog small intestine (duodenum mucous membrane), having a pancreatic juice secretion-stimulating activity. It contains not less than 16,000 secretin Units and not more than 21,500 secretin Units per 1 mg, calculated on the de-acetic acid basis.

**Description** Secretin occurs as a white to pale yellow-white powder.

Identification Dissolve an amount of Secretin in bovine serum albumin TS for secretin so that each mL of the solution contains 20 secretin Units, and use this solution as the sample solution. Separately, dissolve an amount of Secretin Reference Standard in bovine serum albumin TS for Secretin Reference Standard so that each mL of the solution contains 20 secretin Units, and use this solution as the standard solution. Anesthetize a male Wistar rat, weighing 300 to 400 g, starved in advance for 24 hours, by injecting 1.2 g per kg body mass of ethyl carbamate into the abdominal cavity. Fix the animal on the back, cut and open the skin to reveal the femoral vein, insert a cannula filled with isotonic sodium chloride solution into the vein, and sew up the incision. Through the cannula inject 0.2 mL of the standard solution. Shave off the fur of the abdominal region, cut and open the region 3 to 4 cm below the central xiphoid process, and tie up the common bile duct at the duodenal ostial and the stomach at the pylorus, then insert a cannula into an upper