

tra, dissolve Beclometasone Dipropionate and Beclometasone Dipropionate Reference Standard in ethanol (95), respectively, then evaporate the ethanol to dryness, and repeat the test on the residues.

**Optical rotation**  $[\alpha]_D^{20}$ : +88 – +94° (after drying, 0.1 g, 1,4-dioxane, 10 mL, 100 mm).

**Purity (1) Heavy metals**—Proceed with 0.5 g of Beclometasone Dipropionate according to Method 2, and perform the test. Prepare the control solution with 1.5 mL of Standard Lead Solution (not more than 30 ppm).

(2) Other steroids—Dissolve 0.020 g of Beclometasone Dipropionate in 5 mL of a mixture of chloroform and methanol (9:1), and use this solution as the sample solution. Pipet 1 mL of the sample solution, add a mixture of chloroform and methanol (9:1) to make exactly 50 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5  $\mu$ L each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1,2-dichloroethane, methanol and water (475:25:1) to a distance of about 15 cm, and air-dry the plate. Spray evenly alkaline blue tetrazolium TS on the plate: the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

**Loss on drying** Not more than 0.5% (0.5 g, 105°C, 3 hours).

**Residue on ignition** Not more than 0.1% (0.5 g).

**Assay** Weigh accurately about 0.02 g each of Beclometasone Dipropionate and Beclometasone Dipropionate Reference Standard, previously dried, and dissolve each in methanol to make exactly 50 mL. Pipet 10 mL each of these solutions, add exactly 10 mL of the internal standard solution and methanol to make 50 mL, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with 20  $\mu$ L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the ratios,  $Q_T$  and  $Q_S$ , of the peak area of beclometasone dipropionate to that of the internal standard, respectively.

$$\begin{aligned} &\text{Amount (mg) of } C_{28}H_{37}ClO_7 \\ &= \text{amount (mg) of Beclometasone Dipropionate} \\ &\quad \text{Reference Standard} \\ &\quad \times \frac{Q_T}{Q_S} \end{aligned}$$

**Internal standard solution**—A solution of testosterone propionate in methanol (1 in 4000).

**Operating conditions**—

**Detector:** An ultraviolet absorption photometer (wavelength: 254 nm).

**Column:** A stainless steel column 4.6 mm in inside diameter and 20 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5  $\mu$ m in particle diameter).

**Column temperature:** A constant temperature of about 25°C.

**Mobile phase:** A mixture of acetonitrile and water (3:2).

**Flow rate:** Adjust the flow rate so that the retention time of beclometasone dipropionate is about 6 minutes.

**System suitability**—

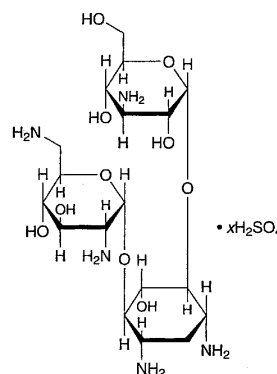
**System performance:** When the procedure is run with 20  $\mu$ L of the standard solution under the above operating conditions, beclometasone dipropionate and the internal standard are eluted in this order with the resolution between these peaks being not less than 8.

**System repeatability:** When the test is repeated 6 times with 20  $\mu$ L of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of beclometasone dipropionate to that of the internal standard is not more than 1.0%.

**Containers and storage** Containers—Tight containers.

## Bekanamycin Sulfate

硫酸ベカナマイシン



$C_{18}H_{37}N_5O_{10} \cdot xH_2SO_4$

O-3-Amino-3-deoxy- $\alpha$ -D-glucopyranosyl-(1 $\rightarrow$ 6)-O-[2,6-diamino-2,6-dideoxy- $\alpha$ -D-glucopyranosyl-(1 $\rightarrow$ 4)]-2-deoxy-D-streptomine sulfate [70550-99-1]

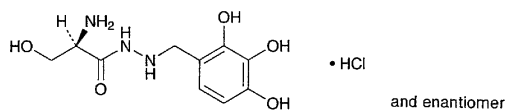
Bekanamycin Sulfate conforms to the requirements of Bekanamycin Sulfate in the Requirements for Antibiotic Products of Japan.

**Description** Bekanamycin Sulfate occurs as a white powder.

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

## Benserazide Hydrochloride

塩酸ベンセラジド



$C_{10}H_{15}N_3O_5 \cdot HCl$ : 293.70

(*RS*)-2-Amino-3-hydroxy-*N'*-(2,3,4-trihydroxybenzyl)propanoylhydrazide monohydrochloride [14919-77-8]