(11:7:1:1) to a distance of about 12 cm, and air-dry the plate. Spray evenly potassium dichromate-sulfuric acid 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% (1 g, 105°C, 2 hours).

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

Assay Weigh accurately about 0.3 g of Imipramine Hydrochloride, previously dried, and dissolve in 20 mL of water. Add 5 mL of sodium hydroxide TS, and extract with three 20-mL portions of chloroform. Filter each extract through a pledget of absorbent cotton on which a small quantity of anhydrous sodium sulfate is placed. Combine the chloroform extracts, and titrate with 0.1 mol/L perchloric acid VS until the yellow solution changes to red-purple (indicator: 10 drops of metanil yellow TS). Perform a blank determination.

Each mL of 0.1 mol/L perchloric acid VS
= 31.687 mg of $C_{19}H_{24}N_2.HCl$

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

Imipramine Hydrochloride Tablets

塩酸イミプラミン錠

Imipramine Hydrochloride Tablets contain not less than 93% and not more than 107% of the labeled amount of imipramine hydrochloride ($C_{19}H_{24}N_2.HCl$: 316.87).

Method of preparation Prepare as directed under Tablets, with Imipramine Hydrochloride.

Identification (1) Weigh a quantity of powdered Imipramine Hydrochloride Tablets, equivalent to 0.25 g of Imipramine Hydrochloride according to the labeled amount, add 25 mL of chloroform, shake thoroughly, and filter. Evaporate the filtrate on a water bath, and proceed with the residue as directed in the Identification (1) under Imipramine Hydrochloride.

(2) Dissolve an amount of the residue obtained in (1), equivalent to 5 mg of Imipramine Hydrochloride, in 250 mL of 0.01 mol/L hydrochloric acid TS, and determine the absorption spectrum as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 249 nm and 253 nm, and a shoulder between 270 nm and 280 nm.

(3) Dry the residue obtained in (1) at 105°C for 2 hours: the residue melts between 170°C and 174°C (with decomposition).

Dissolution test Perform the test with 1 tablet of Imipramine Hydrochloride Tablet at 75 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of diluted pH 6.8 phosphate buffer solution (1 in 2) as the test solution. Take 20 mL or more of the dissolved solution after 60 minutes from the start of the dissolution test, and filter through a membrane filter with pore size of not more than 0.8 μm. Discard the first 10 mL of the filtrate, pipet the subsequent V mL, add diluted pH 6.8 phosphate buffer solution (1 in 2) to make exactly V' mL so that each mL of the filtrate contains about 10 μg of imipramine hydrochloride ($C_{19}H_{24}N_2.HCl$) according to the labeled amount, and use this solution as the sample solution. Separately, weigh accurately about 0.025 g of Imipramine Hydrochloride Reference Standard, previously dried at 105°C for 2 hours, dissolve in diluted pH 6.8 phosphate buffer solution (1 in 2) to make exactly 100 mL. Pipet 4 mL of this solution, add diluted pH 6.8 phosphate buffer solution (1 in 2) to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 250 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Imipramine Hydrochloride Tablets in 60 minutes should be not less than 75%.

Dissolution rate (%) with respect to the labeled amount of imipramine hydrochloride ($C_{19}H_{24}N_2.HCl$)

$$= W_S \times \frac{A_T}{A_S} \times \frac{V'}{V} \times \frac{1}{C} \times 36$$

W_S: Amount (mg) of Imipramine Hydrochloride Reference Standard.

C: Labeled amount (mg) of imipramine hydrochloride ($C_{19}H_{24}N_2.HCl$) in 1 tablet.

Assay Take 20 Imipramine Hydrochloride Tablets, add exactly 200 mL of 0.01 mol/L hydrochloric acid TS, and shake well until the tablets are completely disintegrated. After centrifuging the solution, pipet a volume of the supernatant liquid, equivalent to about 0.025 g of imipramine hydrochloride ($C_{19}H_{24}N_2.HCl$) according to the labeled amount, add 0.01 mol/L hydrochloric acid TS to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.025 g of Imipramine Hydrochloride for Assay, previously dried at 105°C for 2 hours, dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 100 mL, and use this solution as the standard solution. Pipet 3 mL each of these solutions into separators which contain 15 mL of potassium hydrogen phthalate buffer solution, pH 5.6, 8 mL of bromocresol green-sodium hydroxide TS and 30 mL of chloroform, and shake. Filter the chloroform layer through a pledget of absorbent cotton into a 100-mL volumetric flask. Repeat the extraction with two 30-mL portions of chloroform, combine the chloroform layers in the 100-mL volumetric flask, and add chloroform to make exactly 100 mL. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution obtained by proceeding with 3 mL of 0.01 mol/L hydrochloric acid TS in the same manner as the blank. Determine the absorbances, A_T and A_S, of these solutions at 416 nm.

Amount (mg) of imipramine hydrochloride ($C_{19}H_{24}N_2.HCl$)
= amount (mg) of Imipramine Hydrochloride for Assay

$$\times \frac{A_T}{A_S}$$

Containers and storage Containers—Tight containers.