

acid (1 in 1000) as the blank: it exhibits a maximum between 271 nm and 275 nm.

**Assay** Weigh accurately not less than 20 Etilefrine Hydrochloride Tablets, and powder. Weigh accurately a portion of the powder, equivalent to about 5 mg of etilefrine hydrochloride ( $C_{10}H_{15}NO_2 \cdot HCl$ ), add 60 mL of diluted hydrochloric acid (1 in 1000), shake for 10 minutes, add diluted hydrochloric acid (1 in 1000) to make exactly 100 mL, and filter. Discard the first 20 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, weigh accurately about 0.05 g of etilefrine hydrochloride for assay (previously determine the loss on drying at 105°C for 4 hours), dissolve in diluted hydrochloric acid (1 in 1000) to make exactly 1000 mL, and use this solution as the standard solution. Measure exactly 5 mL each of the sample solution and the standard solution, add exactly 5 mL of acetone and 25 mL of boric acid-potassium chloride-sodium hydroxide buffer solution, pH 9.2, to each solution, cool these solution in ice water below 5°C, add exactly 10 mL each of a solution of 4-nitrobenzenediazonium fluoroborate (1 in 2000) with shaking, and allow to stand for 2 minutes. Further, allow to stand at room temperature for 30 minutes, and add boric acid-potassium chloride-sodium hydroxide buffer solution, pH 9.2, to make exactly 50 mL. Determine the absorbances,  $A_T$  and  $A_S$ , of the subsequent solutions of the sample solution and the standard solution at 505 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared with 5 mL of diluted hydrochloric acid (1 in 1000) in the same manner as the sample solution, as the blank.

Amount (mg) of etilefrine hydrochloride ( $C_{10}H_{15}NO_2 \cdot HCl$ )  
= amount (mg) of etilefrine hydrochloride for assay,  
calculated on the dried basis

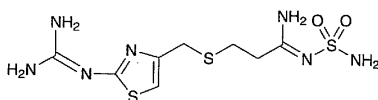
$$\times \frac{A_T}{A_S} \times \frac{1}{10}$$

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

Storage—Light-resistant.

## Famotidine

ファミチジン



$C_8H_{15}N_7O_2S_3$ : 337.45

*N*-(1-Amino-3-[[2-(diaminomethyleneamino)-1,3-thiazol-4-yl]methylsulfanyl]propylidene)sulfamide  
[76824-35-6]

Famotidine, when dried, contains not less than 98.5% of  $C_8H_{15}N_7O_2S_3$ .

**Description** Famotidine occurs as white to yellowish white crystals.

It is freely soluble in acetic acid (100), slightly soluble in ethanol (95), and very slightly soluble in water.

It dissolves in 0.5 mol/L hydrochloric acid TS.

It is gradually colored by light.

Melting point: about 164°C (with decomposition).

**Identification** (1) Determine the absorption spectrum of a solution of Famotidine in 0.05 mol/L potassium dihydrogenphosphate TS (1 in 50,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(2) Determine the infrared absorption spectrum of Famotidine, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

**Purity** (1) Clarity and color of solution—Dissolve 0.5 g of Famotidine in 10 mL of 0.5 mol/L hydrochloric acid TS: the solution is clear and colorless to pale yellow.

(2) Heavy metals—Proceed with 2.0 g of Famotidine according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Related substances—Dissolve 0.20 g of Famotidine in 10 mL of acetic acid (100), and use this solution as the sample solution. Pipet 1 mL of the sample solution, and add acetic acid (100) to make exactly 100 mL. Pipet 1 mL, 2 mL and 3 mL of this solution, add acetic acid (100) to make exactly 10 mL, respectively, and use these solutions as the standard solution (1), the standard solution (2) and the standard solution (3). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5  $\mu$ L each of the sample solution, the standard solutions (1), (2) and (3) on a plate of silica gel (5 to 7  $\mu$ m) with fluorescent indicator for thin-layer chromatography, and dry in a stream of nitrogen.

Develop the plate with a mixture of ethyl acetate, methanol, toluene and ammonia solution (28) (40:25:20:2) to a distance of about 8 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot and other than the spot of the starting point from the sample solution are not more intense than the spot from the standard solution (3). Total intensity of the spots other than the principal spot and other than the spot of the starting point from the sample solution is not more than 0.5% calculated on the basis of intensities of the spots from the standard solution (1) and the standard solution (2) (each spot is equivalent to 0.1% and 0.2%, respectively).

**Loss on drying** Not more than 0.5% (1 g, in vacuum, phosphorus (V) oxide, 80°C, 4 hours).

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

**Assay** Weigh accurately about 0.3 g of Famotidine, previously dried, dissolve in 50 mL of acetic acid (100), 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  
= 16.873 mg of  $C_8H_{15}N_7O_2S_3$

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

Storage—Light-resistant.