ly dried, and dissolve each in 30 mL of the mobile phase. To each add exactly 10 mL of the internal standard solution and the mobile phase to make 50 mL, and use these solutions as the sample solution and the standard solution. Perform the test with 10 μL 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 diclofenamide to that of the internal standard, respectively.

Amount (mg) of $C_6H_6Cl_2N_2O_4S_2$

$$= \text{amount (mg) of Diclofenamide Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of butyl para-hydroxy benzoate in the mobile phase (3 in 5000).
Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 280 nm).

Column: A stainless steel column about 4 mm in inside diameter and about 30 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (10 μm in particle diameter).

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

Mobile phase: A mixture of sodium phosphate TS and acetonitrile (1:1).

Flow rate: Adjust the flow rate so that the retention time of diclofenamide is about 7 minutes.

Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of diclofenamide and the internal standard in this order with the resolution between these peaks being not less than 9.

Containers and storage   Containers—Tight containers.

Diclofenamide Tablets

Dichlorphenamide Tablets

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Diclofenamide Tablets contain not less than 92% and not more than 108% of the labeled amount of diclofenamide ($C_6H_6Cl_2N_2O_4S_2$: 305.16).

Method of preparation   Prepare as directed under Tablets, with Diclofenamide.

Identification   To a quantity of powdered Diclofenamide Tablets, equivalent to 0.2 g of Diclofenamide according to the labeled amount, add 20 mL of methanol, shake, and filter. Evaporate the filtrate on a water bath to dryness, and dissolve 0.01 g of the residue in 100 mL of 0.01 mol/L sodium hydroxide TS. To 10 mL of this solution add 0.1 mL of hydrochloric acid TS, and determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 284 nm and 288 nm, and between 293 nm and 297 nm.

Dissolution test   Perform the test with 1 tablet of Diclofenamide Tablets at 50 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of water as the test solution. Take 20 mL or more of the dissolved solution 60 minutes after starting the 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, and use the subsequent as the sample solution. Separately, weigh accurately about 0.055 g of Diclofenamide Reference Standard, previously dried in vacuum at a pressure not exceeding 0.67 kPa at 100°C for 5 hours, dissolve in 10 mL of methanol, and add water to make exactly 100 mL. Pipet 10 mL of this solution, add water 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 285 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Diclofenamide Tablets in 60 minutes is not less than 70%.

Dissolution rate (%) with respect to
the labeled amount of diclofenamide ($C_6H_6Cl_2N_2O_4S_2$)

$$= W_S \times \frac{A_T}{A_S} \times \frac{1}{C} \times 90$$

W_S: Amount (mg) of Diclofenamide Reference Standard.
C: Labeled amount (mg) of diclofenamide
($C_6H_6Cl_2N_2O_4S_2$) in 1 tablet.

Assay   Weigh accurately, and powder not less than 20 tablets of Diclofenamide Tablets. Weigh accurately a portion of the powder, equivalent to about 0.05 g of diclofenamide ($C_6H_6Cl_2N_2O_4S_2$), add exactly 25 mL of the mobile phase, shake for 15 minutes, and centrifuge. Pipet 10 mL of the supernatant liquid, add exactly 4 mL of the internal standard solution and the mobile phase to make 20 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.05 g of Diclofenamide Reference Standard, previously dried at 100°C in vacuum at a pressure not exceeding 0.67 kPa for 5 hours, dissolve in 30 mL of the mobile phase, add exactly 10 mL of the internal standard solution and the mobile phase to make 50 mL, and use this solution as the standard solution. Proceed as directed in the Assay under Diclofenamide.

Amount (mg) of diclofenamide ($C_6H_6Cl_2N_2O_4S_2$)

$$= \text{amount (mg) of Diclofenamide Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of butyl parahydroxybenzoate in the mobile phase (3 in 5000).

Containers and storage   Containers—Well-closed containers.