DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Dexlansoprazole Delayed Release Capsules

Analytical Profile No.: Dexlans 078/079/AP 106

Dexlansoprazole Delayed Release Capsules (Dexlansoprazole as enteric coated pellets) contains not less than 90.0% and not more than 110.0% of the stated amount of Dexlansoprazole.

Usual Strength: 60 mg

1. Identification:

In the Assay, the principle peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with the reference solution.

Tests:

2. Dissolution: *Deteremine by liquid chromatography*

A. Acidic Stage

Dissolution Parameters:

Apparatus: Basket

Medium: 900ml of 0.1N HCl

Speed and Time: 100 rpm and 120 minutes

Withdraw a suitable volume of the medium and filter.

Test Solution: Dilute 5ml of the filtrate to 20ml with mobile phase.

Reference Solution: Weigh accurately about 30 mg of Dexlansoprazole WS in 100 ml volumetric flask. Add about 70 ml of 0.1 M Sodium Hydroxide, sonicate to dissolve, cool to room temperature and make up the volume with same solvent. Further dilute 5 ml of this solution to 100 ml with mobile phase.

Procedure: Use the chromatographic system as described in the Assay. Inject the reference solution and the test solution.

Calculate the percent release of Dexlansoprazole.

Limit (D1): NMT 10% of the stated amount of Dexlansoprazole.

B. Buffer Stage (In phosphate buffer pH 6.0)

Dissolution Parameters:

Apparatus: Basket

Medium: 900ml of phosphate buffer pH 6.0

Speed and Time: 100 rpm and 60 minutes

I. Preparation of monobasic sodium phosphate dihydrate buffer solution: Weigh accurately and transfer 187.2 g of monobasic sodium phosphate dihydrate in 6500 ml of purified water.

II. Preparation of Dibasic sodium phosphate buffer solution: Weigh accurately and transfer 142 g of Dibasic sodium phosphate in 500 ml of purified water.

Medium (Preparation of pH 6.0 Buffer): Accurately measure about 4840 ml of buffer solution I & 660 ml of buffer solution II & transfer into a suitable beaker. To this solution add 16.5 g of SLS and if necessary, adjust pH to 6.0 with 1N NaOH or 0.1 N HCl solution.

Test Solution: After 120 minutes, discard the liquid media without loss of pellets. Again, add 900 ml of dissolution medium pH 6.0 buffer solution into each six vessels and transfer the pellets into each six vessel after attaining temperature of 37ºC ± 0.5°C and run the apparatus for 60 minutes at 100 rpm. After completion, withdraw a suitable volume of the medium and filter. Dilute 5ml of the filtrate to 20ml with mobile phase.

Reference Solution: Weigh accurately about 30 mg of Dexlansoprazole WS in 100 ml volumetric flask. Add about 70 ml of 0.1 M Sodium Hydroxide, sonicate to dissolve, cool to room temperature and make up the volume with same solvent. Further dilute 5 ml of this solution to 100 ml with mobile phase.

Procedure: Use the chromatographic system as described in the Assay. Inject the reference solution and the test solution.

Calculate the percent release of Dexlansoprazole.

Limit (D2): NMT 25% of the stated amount of Dexlansoprazole.

C. Buffer Stage (In phosphate buffer pH 7.4)

Dissolution Parameters:

Apparatus: Basket

Medium: 900ml of phosphate buffer pH 7.4

Speed and Time: 100 rpm and 5 hours

I. Preparation of monobasic sodium phosphate dihydrate buffer solution: Weigh accurately and transfer 187.2 g of monobasic sodium phosphate dihydrate in 6500 ml of purified water.

II. Preparation of Dibasic sodium phosphate buffer solution: Weigh accurately and transfer 142 g of Dibasic sodium phosphate in 500 ml of purified water.

Medium (Preparation of pH 7.4 Buffer): Accurately measure about 1210 ml of buffer solution I & 4290ml of buffer solution II & transfer into a suitable beaker. To this solution add 16.5 g of SLS and if necessary, adjust pH to 7.4 with 1N NaOH or 0.1 N HCl solution.

Test Solution: After 60 minutes, drain the pH 6.0 buffer solution carefully without loss of pellets, add 900 ml of pH 7.4 buffer solution into each six vessels and transfer the pellets into each six vessel after attaining temperature of 37ºC ± 0.5°C and run the apparatus for 5 hours at 100 rpm. After completion, withdraw a suitable volume of the medium and filter. Dilute 5ml of the filtrate to 20ml with mobile phase.

Reference Solution: Weigh accurately about 30 mg of Dexlansoprazole WS in 100 ml volumetric flask. Add about 70 ml of 0.1 M Sodium Hydroxide, sonicate to dissolve, cool to room temperature and make up the volume with same solvent. Further dilute 5 ml of this solution to 100 ml with mobile phase.

Procedure: Use the chromatographic system as described in the Assay. Inject the reference solution and the test solution.

Calculate the percent release of Dexlansoprazole.

Limit (D1+D2+D3): NLT 70% of the stated amount of Dexlansoprazole.

3. Assay: *Determine by liquid chromatography*

3.1 Solvent Mixture: 0.1M sodium hydroxide

3.2 Test Solution: Weigh a quantity of pellets equivalent to 30mg of Dexlansoprazole into 100 ml volumetric flask, add about 30 ml of solvent mixture, sonicate to disolve, cool & make volume to 100 ml with same solvent and centrifuge a portion of it at about 3000 rpm for 10 minutes. Dilute 5 ml of this solution to 50 ml with mobile phase & mix.

3.3 Reference Solution: Weigh accurately about 30 mg of Dexlansoprazole WS in 100 ml volumetric flask. Add about 70 ml of solvent mixture, sonicate to dissolve, cool to room temperature and make up the volume with same solvent. Further dilute 5 ml of this solution to 100 ml with mobile phase.

3.4 Chromatographic system:

- **Column:** C18, (250 x 4.6 mm), 5 µ particle size
- **Flow rate:** 1.0 ml/min
- **Wavelength:** 285 nm
- **Column Oven Temperature:** ambient
- **Injection volume:** 20 µl
- **Detector:** UV
- **Mobile Phase:** A mixture of 50 volumes of buffer solution prepared by dissolving 2.99 g of potassium dihydrogen orthophosphate in 1000 ml of water, adjusted to pH 7.4 with 1N potassium hydrogen solution and 50 volumes of acetonitrile

3.5 Procedure: Inject the reference solution five times and sample solutions. The test is not valid unless the column efficiency is not less than 2000 theoretical plates, tailing factor is not more than 2.0, and the relative standard deviation for replicate injections is not more than 2.0%. Measure the peak responses. Calculate the content of Dexlansoprazole in the capsules.

4. Other tests: As per pharmacopoeial requirements.