DEPARTMENT OF DRUG ADMINISTRATION

National Medicines Laboratory

ANALYTICAL METHOD VALIDATION COMMITTEE

Cefpodoxime and Potassium Clavulanate Powder for Oral Suspension

Analytical Profile No.: CCP 074/075/ AP 034

Cefpodoxime and Potassium Clavulanate Powder for Oral Suspension contains 90-125% of the stated amount of Clavulanic Acid and 90-110% of the stated amount of Cefpodoxime Proxetil per 5ml suspension.

1. Identification:

1.1 Cefpodoxime proxetil

In the assay, the principle peak in the chromatogram obtained with the sample solution should correspond to the peak in the chromatogram obtained with the reference solution of Cefpodoxime proxetil.

1.2 Potassium Clavulanate

In the assay, the principle peak in the chromatogram obtained with the sample solution should correspond to the peak in the chromatogram obtained with the reference solution of Potassium Clavulanate.

2. pH

4 to 5.5

3. wt/ml

As per manufacturer's instruction.

4. Assay [Use freshly prepared solutions and carry out the test protected from light**]**

4.1 Cefpodoxime proxetil

4.1.1 Chromatographic system:

Column : C18 (250 × 4.6) mm; 5 micron

Flow rate: 1 ml/ min.

Injection volume: 20 µl

Wavelength: 235 nm

Column Temperature: 30 ºC

Detector: UV

Mobile phase: Dissolve 0.463g of Ammonium acetate in 300 ml of water and 200 ml of acetonitrile, mix. Filter through 0.45 µm membrane filter.

Diluents: 40% v/v solution of Acetonitrile in water

4.1.2 Reference Solution:

Weigh accurately about 10 mg of working standard of Cefpodoxime proxetil in 50 ml volumetric flask. Add accurately measured 5 ml of methanol and 25 ml of diluents then sonicate for 5 minutes in an ice cooled bath and make the volume to 50ml with diluents, mix. Dilute 5 ml of the resulting solution to 25ml with diluents, mix. Filter the resulting solution through 0.2 µm membrane filter.

4.1.3 Test Solution:

Weigh accurately about 2.7 g of sample of reconstituted suspension and transfer into a 50 ml volumetric flask. Add accurately measured 10 ml of water and 20 ml of acetonitrile then sonicate for 15 minutes in an ice cooled bath (swirl the flask in every 3 minutes) and make the volume to 50 ml with diluents and filter. Dilute 5 ml of the resulting solution to 100 ml with diluents. Filter through 0.2 micron filter paper.

4.1.4 Procedure:

Inject the reference solution. The relative retention times are about 0.9 for Cefpodoxime proxetil S-epimer and 1.0 for Cefpodoxime proxetil R-epimer; the resolution, R, between Cefpodoxime proxetil S-epimer and Cefpodoxime proxetil R-epimer is not less than 2.0, the tailing factor is not more than 2.0, and the relative standard deviation determined from the sum of the areas of the Cefpodoxime proxetil S-epimer and Cefpodoxime proxetil R-epimer peaks for replicate injections is not more than 2.0%.

Separately inject standard and sample preparation. Record the chromatogram and measure the responses of the major peaks. Calculate the quantity of Cefpodoxime per 5 ml of the reconstituted suspension.

4.2 Potassium Clavulanate

4.2.1 Chromatographic system:

Column: C18 (250 × 4.6) mm; 5 micron

Flow rate: 1 ml/ min.

Injection volume: 20 µl

Wavelength: 220nm

Column Temperature: Ambient

Mobile phase: Dissolve 1.704 g of disodium hydrogen phosphate in 400 ml of water and mix. Adjust the pH to 6.5 with phosphoric acid. Add 100 ml of methanol. Filter through 0.45 µm membrane filter

Diluents: HPLC Grade water

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4.2.2 Test Solution:

Weigh accurately about 2.7 g of sample of reconstituted suspension and transfer into a 50 ml volumetric flask. Add 40 ml of diluents and sonicate for 10 minutes in an ice cooled bath (swirl the flask in every 3 minutes) and make the volume to 50 ml with diluents, mix and filter. Dilute 5 ml of the resulting solution to 100 ml with diluents. Filter through 0.2 micron filter paper.

4.2.3 Reference Solution:

Weigh accurately about 10 mg of working standard of Potassium Clavulanate in 50 ml volumetric flask. Add 40 ml of diluents and sonicate for 10 minutes in an ice cooled bath and make the volume to 50 ml with diluents, mix. Dilute 5 ml of the resulting solution to 25 ml with diluents. Filter through 0.2 micron filter paper

4.2.4 Procedure:

Inject the reference solution. The test is not valid unless the column efficiency is not less than 2000 theoretical plates and the tailing factor is not more than 2.0. Separately inject standard and sample preparation. Record the chromatogram and measure the responses of the major peaks. Calculate the quantity of Clavulanic acid per 5 ml of the reconstituted suspension.

5. Other Tests: As per pharmacopoieal requirements.