ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT
DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory

Cefoperazone & Sulbactam for injection

Analytical Profile No.: CEF SUL 075/076/AP 043

Cefoperazone and Sulbactam for injection contains 90% to 110% of Cefoperazone and 90% to 110% of /sulbactam of stated amount.

1. Identification:

1.1 Cefoperazone:

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 standard solution of Cefoperazone.

1.2 Sulbactam:

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 standard solution of Sulbactam.

2. pH : 4.5-7.0 **(**25% w/v of sample)

3. Water content: NMT 5%

4. Clarity of the solution

Constitute the powder in the vial with 10 ml of water for injection with the help of a hypodermic needle syringe. The solid dissolves completely, leaving no visible residue as undissolved matter. The constituted solution is not significantly less clear than an equal volume of diluents or Purified water contained in similar vessel and examined similarly. The solution is essentially free from foreign matter that can be observed on visual inspection.

5. Particulate matter (By Light Obscuration Particle Counter)

≥ 10 µm: NMT 6000 Particles/container

≤ 25 µm: NMT 600 Particles/container

6. Bacterial Endotoxin Test: NMT 0.2EU/mg

7. Sterility test: shows no growth of microorganisms

8. Assay: Determine by liquid chromatography

8.1 Chromatographic Condition

Column: C18, 150*4.6 mm, 5 µm

Flow rate: 0.8 ml/min

Detector: UV 230nm

Injection volume: 20 µl

Mobile Phase: Buffer:Acetonitrile (70:30)

Buffer: Transfer 3.3 ml of Tetrabutyl ammonium hydroxide (40% in water) in 1000ml of water and adjust the pH to 6.6 ± 0.05 with orthophosphoric acid.

8.3 Test Preparation:

Weigh twenty units taken randomly, and record their fill weight. Empty the content of the entire ten containers and mix all the content and keep the content in the air tight container. Weigh the sample equivalent to 40 mg of Cefoperazone and transfer into 20 ml volumetric flask. Dissolve it with mobile phase and make up the volume with same solvent. Dilute 5 ml of the resulting solution to 25 ml with mobile phase. Filter the resulting solution through 0.2 µm membrane filter paper.

8.2 Standard Solution:

Weigh accurately about 42 mg of Cefoperazone Sodium WS and 44 mg of Sulbactam Sodium WS in 20 ml volumetric flask. Dissolve with mobile phase and make up the volume with same solvent. Dilute 5 ml of the resulting solution to 25 ml with mobile phase. Filter the resulting solution through 0.2 µm membrane filter paper.

8.4 Procedure:

Inject the reference solution five/six 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, the relative standard deviation for replicate injections is not more than 2.0% and the resolution

between Sulbactam and Cefoperazone is not less than 5. Measure the peak responses. Calculate the content of Cefoperazone and Sulbactam per tablet.

8.5 Other Tests: As per pharmacopoieal requirement.