DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Tapentadol Capsules

Analytical Profile No: Tap 074/075/ AP 020

Tapentadol Capsules contains not less than 90 % and not more than 110 % of the stated amount of Tapentadol.

1. **Identification:** In the assay, the principle peak in the chromatogram obtained with the test solution should correspond to the peak in the chromatogram obtained with the reference solution of Tapentadol.

Tests:

2. Dissolution: *Determine by UV Spectrophotometry*

2.1 Dissolution Parameter:

Apparatus:	Basket
Medium:	900 ml of 0.1 N HCl
Speed and time:	75 rpm and 60 minutes
Temperature :	37˚C ± 0.5 ˚C

Withdraw the suitable volume of the medium and filter through 0.2 µm membrane filter.

2.2 Test Solution: Discard the first few ml of the filtrate. Dilute 10 ml of this solution to 20 ml with dissolution medium.

2.3 Reference Solution: Weigh accurately about 27.5 mg Tapentadol hydrochloride RS and transfer into 100 ml volumetric flask. Dissolve with dissolution medium and make up the volume to 100 ml with dissolution medium. Dilute 5 ml of the filtrate to 25 ml with dissolution medium.

2.4 Procedure: Measure the absorbance of test solution and reference solution at 272 nm using dissolution medium as blank. Calculate the percentage release of tapentadol in each capsule by comparison.

2.5 Limit: NLT 80 % (D) of the stated amount.

3. Assay: *determine by liquid chromatography*

3.1 Test Solution: Weigh individually 20 capsules & remove the content of each capsule. Mix the content of all the capsules. Weigh powder equivalent to 50 mg of Tapentadol and transfer into 50 ml volumetric flask. Add about 70 ml of mobile phase and dissolve by sonicating for about 10 minutes. Filter the resulting solution and dilute 5 ml of the filtrate to 25 ml with mobile phase. Again filter the resulting solution through 0.2 µm membrane filter paper.

3.2 Reference Solution: Weigh accurately about 25 mg Tapentadol hydrochloride reference standard and transfer into 25 ml volumetric flask. Dissolve with mobile phase and make up the volume to 25 ml with mobile phase. Dilute 5 ml of the resulting solution to 25 ml with mobile phase. Filter the resulting solution through 0.2 µm membrane filter paper.

3.3 Chromatographic System:

Column:	a stainless steel column 15 cm x 4.6 mm, octyl silane (5 µm),
Injection volume:	20 µl,
Flow rate:	1.0 ml per minute,
Detector:	spectrophotometer set at 215 nm,
Column temperature:	40˚c

Mobile phase: a mixture of 75 volumes of buffer and 25 volumes of methanol,

Buffer solution: dissolve 2.72 g of potassium dihydrogen orthophosphate in 1000 ml of water add 2 ml of triethylamine, and mix. Adjust the pH to 2.5 with orthophosphoric acid,

3.4 Procedure: Inject 20 µl of standard solution of Tapentadol five times as per above mentioned chromatographic condition. In the chromatogram obtained from the standard preparation, the column efficiency determined from the major peak should not be less than 2000 theoretical plates, the tailing factor should be not more than 2.0 and the relative standard deviation of replicate injections should not more be than 2.0 %. Inject 20 µl of the sample preparation and

chromatograph as per above mentioned chromatographic condition. Calculate the content of Tapentadol in each capsule.

4. Other tests: As per pharmacopoeial requirements.