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NOTE

Development and Validation of A Reverse Phase HPLC Method for the Analysis of Fluoxetine in Pharmaceutical Dosage Form

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A rapid and sensitive high performance liquid chromatographic method was developed for the estimation of fluoxetine in pharmaceutical dosage form. Fluoxetine was chromatographed on a reverse phase C_{18} column in a mobile phase consisting of methanol:water (40:60). The mobile phase was pumped at a flow rate of 1 mL/ min. The calibration curve was linear in the range of 1-10 µg/mL. The intra and interday variation was found to be less than 2 % showing high precision of the assay method. The mean recovery of the drug from the solution containing 10 µg/mL was 97 % indicating high accuracy of the proposed method. Due to its simplicity, rapidness, high precision and accuracy, the proposed method may be used for determining fluoxetine in bulk samples and pharmaceutical dosage forms.

Key Words: Fluoxetine, RP-HPLC, Dosage forms.

Fluoxetine is chemically N-methyl-γ-[4(trifluoromethyl)phenoxy]-benzene propamine[1,2]. An isocratic HPLC with a single waters 510 pump, waters 486 tunable absorbance detector and C_{18} column was used. The HPLC system was equipped with software spinchrom.

The contents of the mobile phase methanol:water (40:60) were filtered before use through a 0.4 µm membrane filter and degassed for 0.5 h.

The components of the mobile phase were pumped from the solvent reservoir to the column at a flow rate of 1 mL/min that yielded column back pressure of 140-150 kg/cm^2. The column temperature was maintained at 40ºC. The eluents were monitored 268 nm. Prior to the injection of the drug solutions, the column was calibrated for at least 0.5 h with the mobile phase flowing through the system.

The solutions were prepared on weight basis and volumetric flasks were used to minimize solvent evaporation. Stock solution of the drug was prepared by dissolving 100 mg of fluoxetine in 100 mL of methanol.

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Each of the sample was injected five times into the column and the peak area of the drug was recorded.

Assay of fluoxetine capsules: An accurately weighed sample of powdered capsules equivalent to 100 mg of fluoxetine was placed in a100 mL volumetric flask. 70 mL of methanol was added, shaken well and allowed to stand for 4 h with intermittent sonication to ensure complete solubility of drug. The mixture was then made up to volume with methanol, thoroughly mixed and then filtered through a 0.4 µm membrane filter. An aliquot of the filtrate was transferred to a volumetric flask and made upto volume with methanol to give an expected concentration of fluoxetine. All determinations were made in triplicate.

Precision: The precision of the assay was determined in terms of intra and inter day variation in the peak area for the set of drug solutions on three different days (n = 5).

Accuracy: The accuracy of the HPLC method was assessed by adding known amount of the drug to a drug of solution of known concentration and subjecting the samples to the proposed HPLC method. The amount of drug solution was also added to the volumetric flask containing the powder sample of the capsule formulation with known amount of drug. The drug was estimated as in the procedure described above for the estimation of fluoxetine in the capsule formulations. In both the cases the recovery studies were replicated five times. The accuracy was expressed in terms of the recovery and calculated by multiplying the ratio of measured drug concentration to the expected drug concentration with 100, so as to give the per cent recovery.

The run time of the method was set at 10 min and fluoxetine appeared on the chromatogram at 2.4 min. When the same drug solution was injected 5 times, the retention time of the drug was same the peak area of fluoxetine was calculated and the averages of five determinations were given in Table-1. The results showed that the proposed HPLC method is highly reproducible. When a known amount of drug solution was added to a known concentration of drug solution, there was a high recovery of fluoxetine indicating the high accuracy of the proposed method (Table-2).

TABLE-1
CALIBRATION OF FLUOXETINE BY HPLC METHOD

Concentration of fluoxetine (µg/mL)	Peak area
2	16200
4	32500
6	49000
8	64413
10	80101

TABLE-2
RECOVERY OF FLUOXETINE

Amount of drug added (μg)	Mean amount recovered	Mean recovery (%)
10	9.86	98.6
20	19.52	97.6

The HPLC method developed in the present study has also been used to quantify in capsule dosage forms. Fluoxetine capsules were analyzed as per the procedure described above (Table-3). The average drug content was found to be 97 % of the laballed amount. No interfering peaks were found in the chromatogram indicating excipients used in the formulation did not interfere in the estimation of drug by proposed HPLC method.

TABLE-3
ANALYSIS OF FORMULATION BY PROPOSED HPLC METHOD

Brand	Lebelled amount	Observed amount	Purity
Flutine	10	9.41	94.1

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