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Research Article

# ESTIMATION OF FLUOXETINE IN CAPSULE DOSAGE FORM BY HPTLC METHOD

M. JAGADEESWARAN[1], S. MAHIBALAN[2] AND N. GOPAL.*[3]

[1]Department of Pharmaceutical Analysis, Nandha College of Pharmacy, Erode-52, Tamil Nadu.
[2]Department of Pharmaceutical Chemistry, Balaji Institute of Pharmacy, Narsampet, Warangal, Andhra Pradesh.
[3]Department of Pharmaceutical Chemistry, Balaji Institute of Pharmaceutical Sciences, Narsampet, Warangal, Andhra Pradesh.
E-mail: ngo8pharm@gmail.com
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## ABSTRACT

A simple, accurate, low cost and specific HPTLC method for estimation of Fluoxetine in capsule has been developed. It was performed on Silica gel $G_{60}$ $F_{254}$ aluminium foil using acetonitrile: chloroform in the ratio of 1:9 as mobile phase. The mobile phase having chamber was saturated for 15 minutes at room temperature. The $R_f$ value of Fluoxetine was found to be 0.4. The plate was scanned and quantified at 254 nm. The calibration curve response was observed between 4-20 µg. The linear regression data showed good linear relationship of r = 0.9986. The percent recovery was found to be 100.0 ± 0.01.The developed method was validated for its accuracy and precision with suitable parameters.

**Key words:** HPTLC, Fluoxetine, $R_f$ value, Silica gel $G_{60}$ $F_{254}$.

## INTRODUCTION

Fluoxetine[1] is chemically known as N-methyl-3-[(α, α, α-Trifluro-P-tolyl) oxy] propyl amine. It acts as a selective serotonin reuptake inhibitor and used as Anti-Depressant. It has basically toluene derivative. From the literature review many analytical methods[2, 3, 4, 5] have been reported for the determination of fluoxetine such as spectrophotometry, HPLC, spectrofluorimetry, MS and capillary zone electrophoresis. There is no reported HPTLC method for the determination of Fluoxetine in capsule dosage form. The objective of this work is report a simple, precise, accurate and cost effective HPTLC method for estimation of Fluoxetine is quantified at 254 nm.

## MATERIALS AND METHODS

A Camag, Linomat 5 sample applicator was used. The scanner used was Camag TLC Scanner 3 and CATS 4 software for interpretation of data. Acetonitrile and Chloroform used were of AR grade purchased from S.D Fine Chemicals Ltd, Boisar.

### Standard preparation

Accurately weighed 10 mg of fluoxetine was transferred into10 ml volumetric flask; methanol was added to dissolve and made up to mark with the same (1µg/1µl).

### Chromatographic conditions

Stationary phase-silica gel $G_{60}F_{254}$ TLC precoated plates (10*10), Mobile phase-acetonitrile: chloroform in ratio of 1:9, saturation time -15 Minutes, Migration distance-85 cm, Band width-6mm, Source of radiation-Deuterium lamp, Detection wavelength- 254nm using slit dimension 5x6.5 mm.

### Calibration curve response

Aliquots of 4, 8, 12, 16 and 20 µl of standard solution of Fluoxetine were applied on the chromatographic plates. The plate was developed using acetonitrile: chloroform (1:9) dried and scanned at 254 nm between peak areas/Concentration was observed for Fluoxetine.

**Sample preparation**

Twenty capsules were taken and and average weight was calculated. The capsule shell was removed then the content of Fluoxetine was weighed equivalent to 10 mg and it was taken in a 10 ml volumetric flask and dissolved with small portion of methanol. The solution was shaken well and filtered through whattman filter paper. Then the volume was made up to mark using methanol.

**Assay**

From the sample solution aliquots was spotted (8 µl and 12 µl) on the plate by using Linomat 5 applicator. Developed chromatogram was scanned. A triplicate of those was carried out the peak areas were noted and the amount present formulation was calculated using standard calibration curve. The result of assay is displayed in Table 1.

**Recovery study**

To study the accuracy and precision of the method recovery experiment to determine if there are positive or negative interferences from excipients present in formulation. The recovery of added standard was studied at 3 different levels, each being analyzed in a manner similar to described for assay. Each set of addition was reported seven times and the recovery of added standard was calculated.

**Validation**

The developed method was validated as per ICH guidelines for specificity and accuracy (Table 2).

The method is found to be specific for Fluoxetine since it resoled the peak ($R_f$ = 0.40) in presence of other excipients in the formulation (Fig.1).

The correlation co-efficient (r) and other validation parameters are given in Table2. The precision of method was studied by Intra-day and Inter-day assays. The %RSD was found to be 0.7908 and 0.7999 respectively. Precision of the instrument was checked by repeated scanning of two different concentrations for 3 times on the same day. Accuracy of the method was developed by carrying out recovery studies. Repeatability of the method was checked by analyzing a standard solution of Fluoxetine after application on a TLC plate.

**RESULTS AND DISCUSSION**

The developed method was precise and drug is resolved in well chromatographic system. From the standard deviation, it was observed that the method was precise. The content of Fluoxetine was found to be 19.9 ± 0.05 and the percent recovery 100.0 ± 0.01 using precoated silica gel $G_{60}F_{254}$ on aluminium foil and a mobile phase comprising acetonitrile: chloroform (1:9) which gives good separation of Fluoxetine ( $R_f$ = 0.40). The result of assay is displayed in Table 1.

**Table 1 : Analysis for formulation**

| S. No | Drug | Label claim (mg/Capsule) | Assay | % Label claim |
|---|---|---|---|---|
| 1 | Fluoxetine | 20 | 19.9 ± 0.05 | 99.5 ± 0.04 |

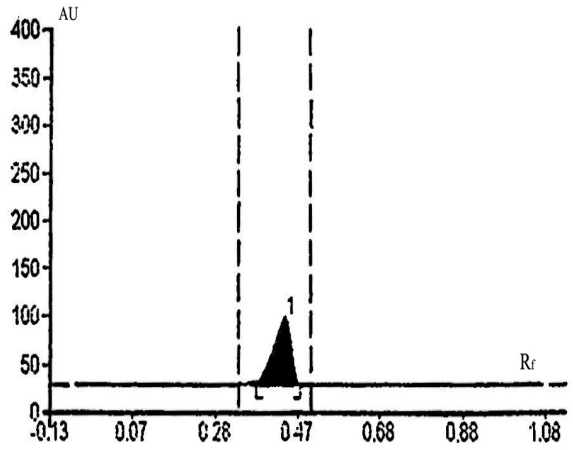
The detector response of Fluoxetine was found to be linear in the range of 4-20 µg/spot. The correlation co-efficient obtain for the linearity Fluoxetine was 0.9986. The result of assay is displayed in Table 2.

**Table 2 : Validation Parameters**

| Parameter | Fluoxetine |
|---|---|
| Precision: | |
| Intra-day (%RSD) | 0.7908 |
| Inter-day (%RSD) | 0.7999 |
| Repeatability | 0.005 |
| Specificity | Specific |
| Linearity range | 4-20 µg/spot |
| Correlation co-efficient (r) | 0.9986 |

Low standard deviation indicated that the present method is more accurate, so the method can be used for routine analysis of Fluoxetine in dosage forms (Fig.1).

**Fig. 1 : Chromatogram for Fluoxetine in capsule dosage form.**




## CONCLUSION

There are several methods existing for the estimation of Fluoxetine viz HPLC, spectrophotometry, spectrofluorimetry, MS and capillary zone electrophoresis. These methods are either costlier or cannot detect impurity whereas the HPTLC method developed can simultaneously run standards and formulation. Therefore it is concluded that the HPTLC method is cost effective, less time consuming, precise and accurate.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## REFERENCES


1. Bhudarari S: Eds, In: The Merck Index, 13th edition, Merck and co., 4241; PageNo.741.
2. Prabhakar A H, Patel V B, Girighar: Spectrophotometric determination of fluoxetine hydrochloride in bulk and in pharmaceutical formulations. J Pharm Biomed Anal 1999; 20 (3): 427-32.
3. Erturk S, Cetin S M, Atmaca S, Ersoy L, Baktr G: A sensitive HPLC method for the determination of fluoxetine and norfluoxetine in human plasma with fluorescence detection. Ther Drug Monit 2005; Feb 27 (1): 38-43.
4. Mandrioli R, Pucci V, Visini D, Varani G, Raggi M A: Rapid methods for determination of fluoxetine in pharmaceutical formulations. J Pharm Biomed Anal 2002; Aug 29 (6): 1127-34.
5. Cherkaoui S, Veuthey J L: Nonaqueous capillary electrophoresis - electrospray-mass spectrometry for the analysis of fluoxetine and its related compounds. Electrophoresis 2002 Feb; 23 (3): 442-448.