ESTIMATION OF FLUOXETINE IN CAPSULE DOSAGE FORM BY HPTLC METHOD

M. JAGADEESWARAN1, S. MAHIBALAN2 AND N. GOPAL.*3
1Department of Pharmaceutical Analysis, Nandha College of Pharmacy, Erode-52, Tamil Nadu.
2Department of Pharmaceutical Chemistry, Balaji Institute of Pharmacy, Narsampet, Warangal, Andhra Pradesh.
3Department of Pharmaceutical Chemistry, Balaji Institute of Pharmaceutical Sciences, Narsampet, Warangal, Andhra Pradesh.
E-mail: ngo8pharm@gmail.com
Received – 17th May, 2009, Revised and Accepted – 20th July 2009

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-[(\(\alpha\), \(\alpha\), \(\alpha\)-Trifluro-P-tolyl) oxy] propyl amine. It acts as a selective serotonin reuptake inhibitor and used as Anti-Depressant. It has basically tolune 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 into 10 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 whatman 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).

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

<table>
<thead>
<tr>
<th>S. No</th>
<th>Drug</th>
<th>Label claim (mg/Capsule)</th>
<th>Assay</th>
<th>% Label claim</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fluoxetine</td>
<td>20</td>
<td>19.9 ± 0.05</td>
<td>99.5 ± 0.04</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Fluoxetine</th>
</tr>
</thead>
<tbody>
<tr>
<td>Precision:</td>
<td></td>
</tr>
<tr>
<td>Intra-day (%RSD)</td>
<td>0.7908</td>
</tr>
<tr>
<td>Inter-day (%RSD)</td>
<td>0.7999</td>
</tr>
<tr>
<td>Repeatability</td>
<td>0.005</td>
</tr>
<tr>
<td>Specificity</td>
<td>Specific</td>
</tr>
<tr>
<td>Linearity range</td>
<td>4-20 µg/spot</td>
</tr>
<tr>
<td>Correlation co-efficient (r)</td>
<td>0.9986</td>
</tr>
</tbody>
</table>

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).

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.

ACKNOWLEDGEMENT
Nandha College of Pharmacy, Erode and SNR & Sons Charitable Trust, Coimbatore for providing facility to carryout the work and Cadila Pharmaceuticals, Ahmedabad for procuring the gift sample.

REFERENCES