

**International Journal of Pharma and Bio Sciences**

RESEARCH ARTICLE

ANALYTICAL CHEMISTRY

**A VALIDATED UV SPECTROPHOTOMETRIC DETERMINATION OF AN ANTIDEPRESSANT DRUG – DULOXETINE HYDROCHLORIDE FROM CAPSULE FORMULATIONS**



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**ABSTRACT**

Simple, sensitive and specific spectrophotometric method was developed and validated for quantification of Duloxetine hydrochloride<sup>3</sup> in capsule dosage form. Drug showed the absorption maxima in methanol at 292nm and was linear for a range of 5-25µg/ml with a correlation coefficient of 0.9999. The validation of the above method was done by carrying out precision and accuracy studies. The Limit of detection and Limit of Quantification for Duloxetine hydrochloride was found to be 0.82mcg/ml and 2.76mcg/ml. The percentage recovery was found to be 99.3% and showed good repeatability with relative standard deviation less than 2. So, the proposed method can be applied for the routine analysis of Duloxetine hydrochloride from formulations.

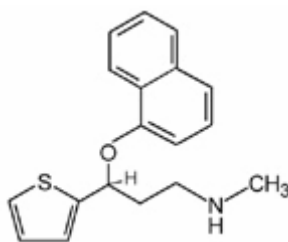
## KEY WORDS

Spectrophotometry, Duloxetine hydrochloride, Methanol, Beer's law.

## INTRODUCTION

Duloxetine hydrochloride, is chemically known as (+)-(S)-N-Methyl-3-(naphthalen-1-yloxy)-3-(thiophen-2-yl)propan-1-amine (fig-1), is a serotonin-norepinephrine reuptake inhibitor<sup>7</sup>, effective for major depressive disorder which is given orally. For the estimation of Duloxetine hydrochloride in pharmaceutical formulations, literature survey reveals many analytical

methods include UV-Spectrophotometry<sup>4,5</sup>, HPLC and LC-MS and fluorimetric methods. To the best of our knowledge, there is no report of UV-Visible spectrophotometric method for its estimation. Therefore, an attempt was made to develop a simple spectrophotometric method for the estimation of the present drug in formulations i.e. capsules.



**Figure 1**  
**Chemical structure of Duloxetine hydrochloride**

## EXPERIMENTAL

Shimadzu UV-VIS (1700 series) double beam spectrophotometer equipped with 10mm matched quartz cells.

## CHEMICALS

Duloxetine hydrochloride was obtained from Hetero drugs Ltd., Hyd, was used as such without further purification. Different brands of capsules of given drug were supplied from local pharmacy.

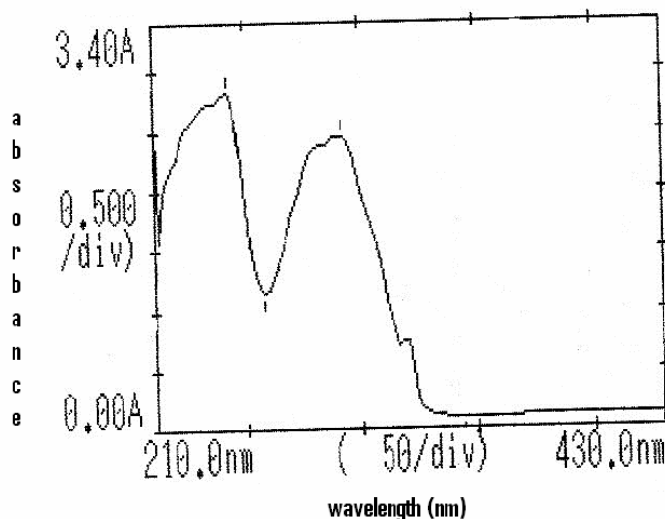
## RECOMMENDED PROCEDURE AND CALIBRATION CURVE

DXH (100 mg) was accurately weighed and dissolved in 100 mL of methanol to form a stock solution (1000 µg/mL). The stock solution was further diluted suitably with methanol to get a working standard solution of concentration 100 µg/mL. This working standard solution was suitably diluted to give a concentration of 10 µg/mL and this was then scanned in UV range. This showed an absorption maximum at 292 nm (Figure 2). Aliquots (0.5, 1.0, 1.5, 2.0 and 2.5) µg/mL of working standard solution (100 µg/mL)

corresponding to 5-25  $\mu\text{g}$  were taken in a series of 10 mL volumetric flask and volume made up with methanol. The absorbance measurements of these solutions were carried out against methanol as blank at 292 nm. A calibration curve

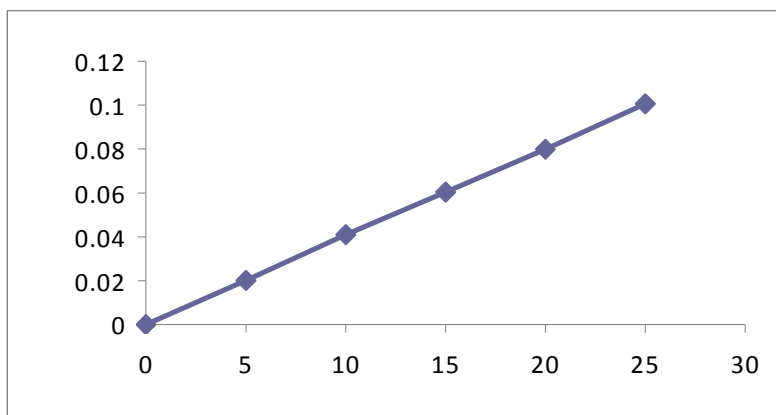
of DXH was plotted (Figure 3). The concentration of the unknown was read from the calibration graph or computed from the regression equation.

**Absorption spectrum of duloxetine HCl**



**Figure 2**  
*Duloxetine hydrochloride scanned in UV range (in methanol).*

**Beer's plot of duloxetine hydrochloride**



**Figure 3**  
*Standard plot of duloxetine hydrochloride.*

## PROCEDURE FOR CAPSULES

Two commercial formulations, Dulot (Lupin Pharmaceuticals) and Duzela (Sun Pharmaceutical Industries Ltd) were purchased from local pharmacy. The contents of 20 capsules were mixed and accurately weighed amount of the contents equivalent to 100 mg of DXH was transferred into a 100 mL volumetric flask. 70 mL of methanol was added and the contents of the flask were shaken for 5 min. The solution was then diluted to the mark with the methanol to get a stock solution of 1000 µg/mL. The content of the flask was filtered through Whatman filter paper No.1 and 10 mL of the filtrate was diluted to 100 mL with methanol in a 100 mL volumetric flask to give a concentration of 100 µg/mL. Suitable volume of this solution was taken in 10 mL volumetric flask and volume was made up with methanol. Absorbances were read and concentrations of DXH determined

using the calibration curve. Calculations were then made with the dilution factor to find out the concentration of the drug in capsules. The experiments were repeated six times to check its reproducibility.

## RESULTS AND DISCUSSION

The proposed method for determination of Duloxetine hydrochloride showed molar absorptivity of  $0.97 \times 10^4$  L/mol.cm. Linear regression of absorbance on concentration gave the equation  $y = 0.0322x + 0.0042$  with a correlation coefficient (r) of 0.9999. The optical characteristics such as Beer's law limit, Sandell's sensitivity, % Range of error (0.05 and 0.01 confidence limits) were calculated and are summarized in Table 1. Statistical analysis of commercial formulations has been shown in Table 1.

**Table 1**  
*Statistical analysis of Duloxetine hydrochloride capsules.*

Parameters	Results
$\lambda_{max}$ , nm	292
Beer's law limit, µg/mL	5-25
Molar absorptivity, L mole <sup>-1</sup> cm <sup>-1</sup>	$0.97 \times 10^4$
Sandell's sensitivity (µg cm <sup>-2</sup> / 0.001 absorbance unit)	0.036
Regression equation (Y = a + bC)	
Slope (b)	0.0040
Intercept (a)	0.0001
Correlation coefficient (r)	0.9999
% Range of error (Confidence limits)	
0.05 level	0.1893
0.01 level	0.2753

**Table 2**  
**Statistical analysis of duloxetine hydrochloride capsules.**

Brand	Labelled amount mg/capsule	Amount found mg/capsule	% claim + SD*
Dulot	20	19.9978	99.98±0.14
Duzela	20	19.9984	99.99±0.23

\*average of six determinations.

**Table 3**  
**Recovery studies of Duloxetine hydrochloride capsules.**

Brand	Amount added, mg	Amount found, mg	% recovery ± SD*
Dulot	5	29.9981	99.99±0.22
Duzela	5	29.9949	99.97±0.15

\*average of six determinations.

## CONCLUSION

In this study a simple, rapid, sensitive, accurate and precise UV spectrophotometric method for the determination of Duloxetine hydrochloride in bulk and pharmaceutical formulation has been

developed and validated. It was found that the common excipients present in the formulation did not interfere with the proposed method and can be used for the routine quality control analysis of Duloxetine hydrochloride in bulk as well as in marketed capsules.

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