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Simple UV Spectrophotometric Determination of Duloxetine Hydrochloride in Bulk and in Pharmaceutical Formulations

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Abstract: A new, simple and sensitive spectrophotometric method in ultraviolet region has been developed for the determination of duloxetine hydrochloride in bulk and in pharmaceutical formulations. Duloxetine hydrochloride exhibits absorption maxima at 288 nm with apparent molar absorptivity of 0.97×10^4 L/mol.cm in 0.1 N HCL. Beer's law was found to be obeyed in the concentration range of 5-30 µg/mL. The method is accurate, precise and economical. The proposed method has been applied successfully for the analysis of the drug in pure and in its capsule dosage forms. In this method, there is no interference from any common pharmaceutical additives and diluents. Results of the analysis were validated statistically and by recovery studies.

Keywords: UV Spectrophotometry, Duloxetine hydrochloride, Beer's law, Capsule.

Introduction

Duloxetine hydrochloride, chemically known as (+) - (*S*) - *N*-methyl - *γ*- (1- naphthyloxy) - 2-thiophenepropylamine hydrochloride[1] (Figure 1), is an antidepressant agent[2]. It is indicated for the treatment of major depressive disorder (MDD).It is a selective serotonin and nor-epinephrine reuptake inhibitor (SSNRI) for oral administration. A survey of literature showed few analytical methods has been reported for determination of duloxetine hydrochloride in human serum and biological fluids using tandem MS and LC-MS methods[3] and HPLC[4-5] method for the estimation of duloxetine hydrochloride in pharmaceutical preparations. Determination of duloxetine hydrochloride by spectroflourimetric method[6-7] has also been reported. To the best of our knowledge, there is no report on UV–Visible spectrophotometric method for its estimation. Therefore, an attempt was made to develop a

simple, rapid, accurate and sensitive UV spectrophotometric method for the estimation of duloxetine hydrochloride in pure drug and in formulations *i.e*. capsules. UV analysis of duloxetine hydrochloride was performed in 0.1 N HCL. The spectrum was recorded from 200 nm to 400 nm. The quantitative analysis was carried out at 288 nm. The method was validated and applied for the determination of duloxetine hydrochloride in capsule dosage form.

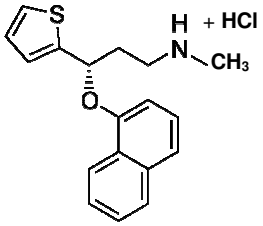

Figure 1. Chemical structure of duloxetine hydrochloride.

Experimental

ELICO SL-164 UV-Visible double beam spectrophotometer equipped with 10 mm matched quartz cells. A sartorius analytical balance was used.

Chemicals

Pure duloxetine hydrochloride (DXH) obtained from Orchid Chemicals and Pharmaceuticals Ltd., Chennai, was used as such without further purification. Different brands of capsules of DXH were supplied from local pharmacy.

Recommended procedure and calibration curve

DXH (100 mg) was accurately weighed and dissolved in 100 mL 0.1 N HCl to form a stock solution (1000 µg/mL). The stock solution was further diluted suitably with 0.1 N HCl to get a working standard solution of concentration 100 µg/mL. This working standard solution was suitably diluted to give a concentration of 20 µg/mL and this was then scanned in UV range. This showed an absorption maximum at 288 nm (Figure 2). Aliquots (0.5,1.0,1.5,2.0,2.5 and 3.0) mL of working standard solution (100 µg/mL) corresponding to 5-30 µg were taken in a series of 10 mL volumetric flask and volume made up with 0.1 N HCl. The absorbance measurements of these solutions were carried out against 0.1 N HCl as blank at 288 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.

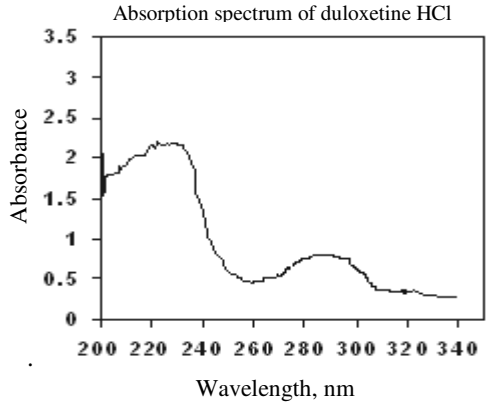

Figure 2. Duloxetine hydrochloride scanned in UV range (in 0.1 N HCl).

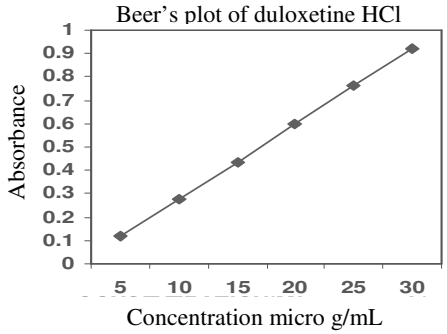

Figure 3. Standard plot of duloxetine hydrochloride.

Procedure for capsules

Two commercial formulation, dupact (M/s Wockhardt) and duxet (M/s Unichem) 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 0.1 N HCl was added and the contents of the flask were shaken for 5 min. The solution was then diluted to the mark with the 0.1 N HCl 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 0.1 N HCl 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 0.1 N HCl. 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 x 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 0.9998. 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 2.

Table 1. Optical characteristics of duloxetine hydrochloride.

Parameters	Results
λ_{max}, nm	288
Beer's law limit, µg/mL	5-30
Molar absorptivity, L $mole^{-1}$ cm^{-1}	$0.97x10^4$
Sandell's sensitivity	0.033
(µg cm^{-2} / 0.001 absorbance unit)	
Regression equation (Y = a + bC)	
Slope (b)	0.0322
Intercept (a)	0.0042
Correlation coefficient (r)	0.9998
% Range of error (Confidence limits)	
0.05 level	0.1923
0.01 level	0.2845

**Average of six determinations.*

Table 2. Statistical analysis of duloxetine hydrochloride capsules.

Brand	Labeled amount mg/capsule	Amount found mg/capsule	% Label claim ± SD*
Dupact 20 mg	20	19.9965	99.98± 0.14
Duxet 20 mg	20	19.9997	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*
Dupact, 20 mg	5	29.9972	99.99± 0.22
Duxet, 20 mg	5	29.9925	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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