



JOURNAL OF PHARMACEUTICAL SCIENCE AND BIOSCIENTIFIC RESEARCH (JPSBR)

(An International Peer Reviewed Pharmaceutical Journal that Encourages Innovation and Creativities)

Development and Validation of UV Spectrophotometric Area under Curve (AUC) method for Dapoxetine HCl in Pharmaceutical Formulation

Anuja Kolsure*, Rucha Hiremath

JSPM's Jayawantrao Sawant College of Pharmacy & research, Hadapsar, Pune, India-411028

ABSTRACT:

Simple, precise and economical UV spectrophotometric methods have been developed for the estimation of Dapoxetine HCl in pharmaceutical dosage form. Area under curve was integrated in the wavelength range of 285-305 nm. Calibration curves were plotted. Beer's law obeyed in the Concentration range 10-60 μ g/ml and with correlation coefficient of 0.9981. Accuracy and precision studies were carried out and results were satisfactory. The proposed methods validated as per ICH analytical method development guidelines. The results of the analysis were validated statistically.

KEY WORDS: Dapoxetine HCl, Beer's law, Area under Curve, Validation, ICH guidelines

Article history:

Received 26 Jun 2015
Revised 09 July 2015
Accepted 20 July 2015
Available online 05 Oct 2015

Citation:

Kolsure A., Hiremath R., Development and Validation of UV Spectrophotometric Area under Curve (AUC) method for Dapoxetine HCl in Pharmaceutical Formulation. *J Pharm Sci Bioscientific Res.* 2015 5(5):475-478

INTRODUCTION:

Dapoxetine hydrochloride, a fast-acting serotonin reuptake transporter inhibitor, is generally prescribed for the treatment and management of premature ejaculation and erectile dysfunction in adult male¹. Dapoxetine hydrochloride prevents the reuptake of serotonin transporter. The drug binds with the reuptake transporters of norepinephrine and dopamine and inhibits the reuptake².

Chemically Dapoxetine ((+)-(S)-N, N-dimethyl-(α)-[2(1-naphthalenyloxy)ethyl]-benzenemethanamine hydrochloride), Dapoxetine hydrochloride is a water-soluble powder with a molecular weight of 341.88 and has a pKa of 8.63.

The literature study reveals that several spectrometric and HPLC methods available for Dapoxetine in combined tablet formulation [4-9]. No method has been reported for individual Dapoxetine HCl by using water as a solvent.

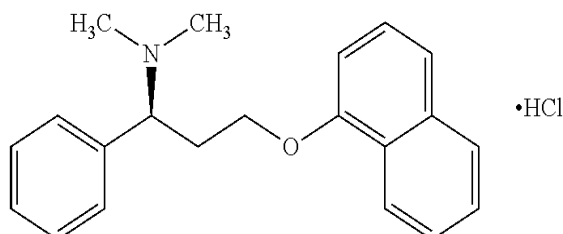


Figure 1: Chemical structure of Dapoxetine HCl

*For Correspondence:

Anuja Kolsure

Jspm's Jayawantrao Sawant College of Pharmacy and Research, Hadapsar, Pune-28.

Email: jpsbronline@rediffmail.com

(www.jpsbr.org)

MATERIAL AND METHODS:

APPARATUS AND INSTRUMENTATION

Shimadzu UV 1800 with matched quartz cells and equipped with UV Probe Software, was used for this work. Single panelectronic balance [Shimadzu, AX 200, (Japan)] was used for weighing purpose. Sonication of the solutions was carried out using an Ultrasonic Cleaning Bath (Spectra Lab. UCB 40, India). Calibrated volumetric glassware (Borosil) was used in this study.

Chemicals

Active pharmaceutical ingredient (API) Dapoxetine HCl was supplied as a gift sample by Sava Healthcare Ltd. Chinchwad, Pune Maharashtra, India. Commercially available tablets (Sustinex) containing 30 mg of Dapoxetine HCl were obtained from local pharmacy. Water has been selected as an analytical media for present research work.

PREPARATION OF STANDARD SOLUTION

The standard stock solution of Dapoxetine HCl was prepared by transferring, accurately weighed, 10 mg of API to 100ml of volumetric flask. The drug was dissolved with sonication in water and volume was made up to the mark by using water. The standard stock solution (100 µg/ml) was further diluted with water to get the concentration of 10 µg/ml.

SELECTION OF WAVELENGTH RANGE

The standard solution of 10µg/ml was scanned between 400 nm to 200 nm in UV spectrophotometer against water as blank after baseline correction. Wavelength range was selected around wavelength maxima (236 nm). Different working standards were prepared between 10-60 µg/ml. various wavelength range were tried and final range between 285-305 nm was selected on the basis of linear relationship between area and corresponding concentration (Figure 2).

PREPARATION OF CALIBRATION CURVE

Working solutions were prepared from standard stock solution by further dilution with water to obtain the Concentration of 10, 20, 30, 40, 50, 60 µg/ml, respectively. These solutions were scanned from 400 to 200 nm and Area under Curve (AUC) was integrated in the range of

285-305 nm. The calibration curve was plotted between Areas against concentration (Fig.3)

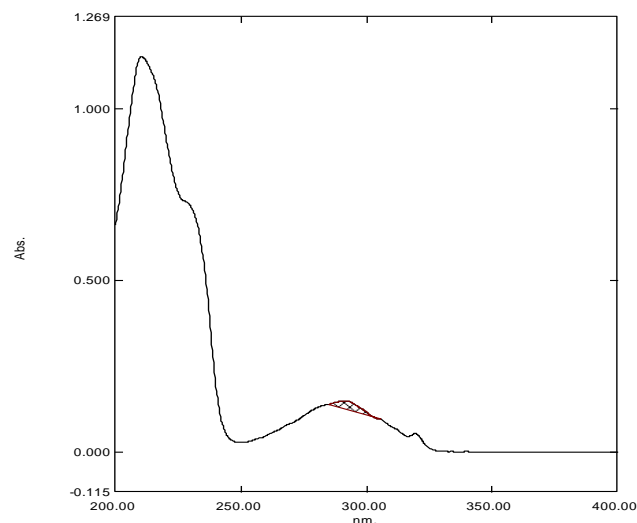


Figure 2: Wavelength range selected for Dapoxetine HCl

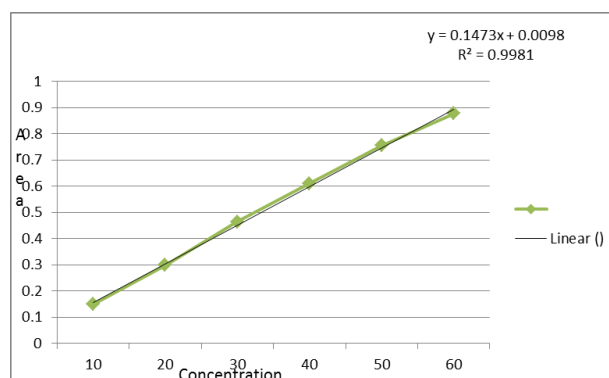


Figure 3: Calibration Curve

ASSAY OF TABLET FORMULATION

Twenty tablets were weighed and average weight was calculated. These tablets were crushed and powdered in a glass mortar. The tablet powder equivalent to 10 mg of Dapoxetine HCl was accurately weighed and transferred to a 100 ml of volumetric flask and diluted up to mark with water. The solution was filtered with Whatmann filter paper and sonicated for 15 min. This solution was further diluted to obtain 10µg/ml solution with same solvent and subjected for UV analysis (Table no.1)

METHOD VALIDATION

Linearity

The linearity of the drug was found to be between 10-60 µg/mL concentrations. The calibration graphs were obtained and treated by linear regression analysis. The

Table 1: Results of Samples (ASSAY)

Sample solution concentration (µg/ml)	Amount Found (%)	% RSD
10 µg/ml	99.5	0.49

equation of the calibration curve for Dapoxetine HCl is $y = 0.1473x + 0.0098$, and correlation coefficient (r^2) 0.9981(Figure 3)

Precision

Method repeatability was determined by six times repetitions of assay procedure. For intra-day precision method was repeated 6 times in a day and the average % RSD was determined. Similarly the method was repeated on 6 different days for inter-day precision and average % RSD was determined (Table 2)

Table 2: Results of Precision Study

Parameter	Inter- day Precision		Intra-day Precision	
	SD*	% RSD*	SD*	% RSD*
Area Under Curve	0.041312	0.15688	0.027539	0.07671

*n=6

Accuracy (Recovery study)

The accuracy of the proposed method was checked by recovery studies, by addition of standard drug solution to preanalysed sample solution at three different concentration levels (80 %, 100 % and 120 %) within the range of linearity for both the drugs (Table 3)

RSD: Relative standard deviation,

*Average of 3 observations

RESULTS AND DISCUSSION

No any spectrophotometric methods have been described for the determination of Dapoxetine HCl in water. Simple, accurate, linear, precise and fast Area under Curve spectrophotometric method was developed for the routine determination of Dapoxetine HCl.

The generated regression equation was $305 \int 285 \text{ Ad} = y = 0.1473x + 0.0098$ ($R^2 = 0.9981$) Where, $305 \int 285$ Is Area

Table 3: Results of Recovery Study

Recovery Level	Amount Spiked (µg/mL)	% Mean Recovery	SD*	% RSD*
80%	8	99.46	0.033	0.034
100%	10	99.49	0.013	0.013
120%	12	99.57	0.029	0.029

under Curve (AUC) between 285 to 305 nm, R^2 is correlation coefficient. The method can be used for the routine analysis of Dapoxetine HCl in bulk and tablet dosage form (Table 4).

Table 4: Results of Validation parameters

Parameters	Dapoxetine HCl
wavelength (nm)	285-305nm
Linearity (µg/mL)	10-60 µg/mL
Intercept*(c)	0.0098
Slope* (m)	0.1473
Correlation coefficient*	0.9981
Inter-day* (Precision) (%RSD)	0.15688
Intra-day* (Precision) (%RSD)	0.07671
Accuracy (n=3)	
80%	99.46±0.033
100%	99.49±0.013
120%	99.57±0.029

CONCLUSION

The authors are thankful to Sava Healthcare Ltd. Chinchwad, Pune Maharashtra, India for providing us gift sample and managements of Jspm’s Jayawantrao Sawant College of Pharmacy and Research, Hadapsar, Savitribai Phule Pune University for providing needed facilities for this work.

REFERENCES:

1 Dapoxetine: an evidence-based review of its effectiveness in treatment of premature ejaculation [www.ncbi.nlm.nih.gov/PubMed Central \(PMC\)](http://www.ncbi.nlm.nih.gov/PubMed Central (PMC))

2 Dapoxetine hydrochloride information from Drugs Update
www.drugsupdate.com/generic/view/1088/Dapoxetine-hydrochloride

3 Anurag Jhanjee, Pankaj Kumar, M.S. Bhatia, Shruti Srivastava. Dapoxetine — A Novel Drug for Premature Ejaculation, Delhi Psychiatry Journal 2011; 14(1).

4 CA Prajapati, BS Patel. Development and Validation of Spectrophotometric Method for Simultaneous Determination of Sildenafil Citrate and Dapoxetine Hydrochloride in Their Combined Dosage Formulation, PharmaTutor; 2014; 2(11); 84-88

5 Anuruddha P. Chabukswar, Bhanudas S. Kuchekar, Sonali L. Patil, Swagati A. Moon, Subhash G. Chate, Bharat D. Pagare. D.Spectrophotometric Simultaneous Determination of Dapoxetine and Sildenafil in Combined Tablet Dosage Form by Absorbance Corrected Method, Der Pharma Chemica, 2012;4 (4):1404-1407

6 Albin Pt, Y Haribabu, Sosamma Cicy Eapen, Sheeja Velayudhan Kutty, Kumar P, Nithyamol P. Validated Spectrophotometric Methods for Simultaneous Estimation of Sildenafil Citrate and Dapoxetine Hcl in Tablet Dosage Form, the Pharma Innovation – Journal, 2013; 2(4):40-45.

7 K.S. Nataraj, K.Kesinath Reddy, D.Kiran Kumar, S.Suresh Kumar. Simple Validated UV Spectrophotometric Method for Estimation of Dapoxetine, Journal of Chemical and Pharmaceutical sciences, July – September 2011;4(3):105-107.

8 Abha D. Giri, Vidhya K. Bhusari, Sunil R. Dhaneshwar. Validated Hplc Method for Simultaneous Quantitation of Tadalafil and Dapoxetine Hydrochloride In Bulk Drug And Formulation, Int J Pharm Sci, 2012; 4(2), 654-658.

9 T. Rohith, S. Ananda. A Validated Chiral Liquid Chromatographic Method for the Enantiomeric Separation of Dapoxetine Hydrochloride, International Journal of Advanced Research in Pharmaceutical & Bio Science, 2012; 2(3):311-319.

