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UV spectrophotometric estimation of carbamazepine in bulk and tablet dosage form

Manoj P. Borse* and Sugandha V. Mulgund

Department of Quality Assurance Techniques, STES's Sinhgad College of Pharmacy, Vadgaon (Bk), Pune, India

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ABSTRACT

A UV spectrophotometric method has been developed for estimation of Carbamazepine which is simple, precise, accurate and economical. Initially the standard and sample stock solutions were prepared by using methanol and further dilutions were made with distilled water as a solvent. Quantitative analysis of drug was carried out at 284 nm. The linearity was obtained over the concentration range of 8-18 µg/ml. The regression equation was found to be y=0.049 x+0.009 with value of R^2 as 0.999. The result of analysis has been validated as per ICH Q2 (R1) guidelines. The proposed method can be used for the reliable quantification of Carbamazepine in tablet formulation.

Keywords: Carbamazepine, UV Spectrophotometry, Validation, Zero – order.

INTRODUCTION

Carbamazepine (CBZ) is an antiepileptic drug and it is used for the treatment of trigeminal neuralgia. IUPAC name of Carbamazepine is 5H-dibenzo[b,f]azepine-5-carboxamide, its structure is shown in fig. 1. Carbamazepine is official in IP 2010, USP 2007 [1,2,3].

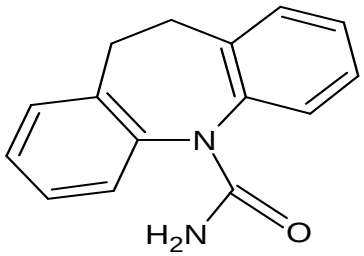


Figure 1: Chemical structure of Carbamazepine

Several methods such as HPLC [4], FTIR [5], UV-Visible spectroscopy [5], HPTLC [6], have been developed for determination of CBZ in pharmaceutical preparations, plasma fluids and human serum. In this method we developed a simple, precise and accurate spectroscopic method for determination CBZ in tablet dosage form.

MATERIALS AND METHODS

Apparatus and Instruments

In this study Shimadzu UV 1800(Japan) double beam spectrophotometer with match quartz cells, connected to computer loaded UV Prob Software was used. Single pan electronic balance (Shimadzu, AX 200, Japan), Ultrasonicator (Spectra lab UCB 40, India) and Calibrated volumetric glasswares (Borosil®) were used for this work.

Materials

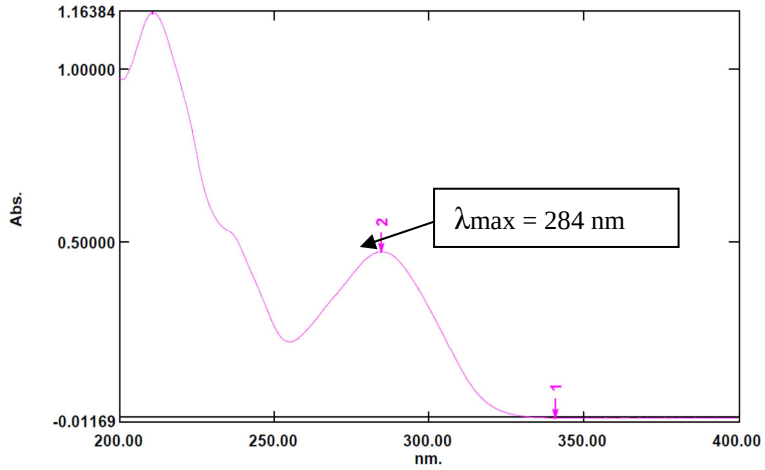
Carbamazepine was procured as a gift sample from a reputed company in India. The commercially available tablets (TEGRITAL® 400 mg, Novartis India Ltd., Batch No.132003EH) were obtained from local pharmacy. Methanol HPLC Grade used as solvent for stock solution was obtained from Fisher Scientific India, and distilled water was used as a diluent was obtained from ELGA water purification unit.

Methods

Preparation of standard stock solution

The standard stock solution was prepared by dissolving 50 mg CBZ in 30ml methanol by sonication for 10 minutes and volume was made up to the 50 mL mark using methanol. From the standard stock solution (1000 µg/ml), different aliquots were diluted with distilled water separately to prepare a series of concentrations from range 8-18 µg/ml. The standard solution of CBZ (10 µg/ml) was scanned in the range of 400-200 nm against distilled water as a blank. The λmax of this solution was found to be 284 nm (figure 2). Absorbance of all solutions was measured at 284 nm against distilled water as a blank. The calibration curve was prepared by plotting absorbance versus concentration of CBZ.

Figure 2: UV Spectrum of CBZ (10µg/ml).



Assay of tablet formulation

Twenty tablets were accurately weighed and average weight was calculated, they were crushed to fine powder. The powder equivalent to 50 mg CBZ was dissolved in 30 ml of methanol with help of sonication and volume was made up using methanol upto the mark of 50 mL volumetric flask. The solution was filtered using whatmann filter paper. This solution was further diluted to obtain 10 µg/ml concentration of the solution by using distilled water as a solvent and observed by UV analysis. This procedure was repeated in triplicate (Tablet 1).

Table 1. Assay of tablet dosage form

Sample solution concentration(µg/ml)	Amount found (%)	Mean ±SD	% RSD*
10	100.64	101.15±0.0459	0.04539
10	101.31		
10	101.51		

*SD: standard deviation, RSD: relative standard deviation, (*n=3).*

RESULTS AND DISCUSSION

The CBZ was estimated by using ultraviolet spectroscopic method. The method obeyed Beer's law in the concentration range of 8-18 µg/ml and its detection wavelength was 284 nm. Statistical evaluation of analysis and recovery study was carried out. The data obtained from proposed method showed accuracy of method. The values of standard deviation and recovery study were in the given limits (NMT 2 %).

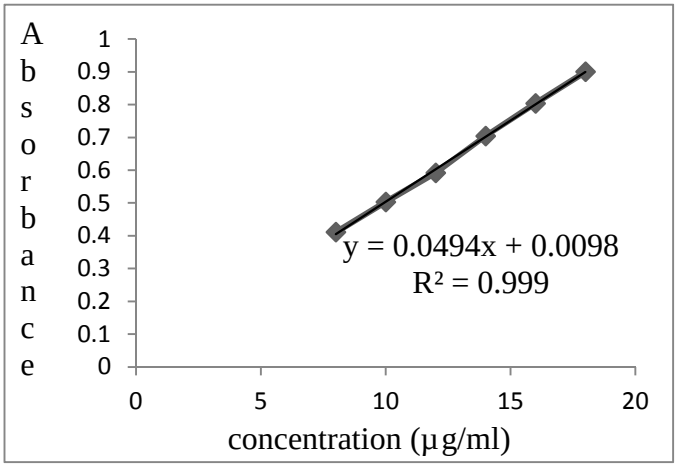
Method validation

The proposed method was validated for various parameters such as Linearity and Range, Accuracy, Precision, Limit of Detection (LOD), Limit of Quantitation (LOQ), and Robustness according to ICH Q2 (R1) guideline.

Linearity and Range

The linearity of concentrations (8-18 µg/ml) was scanned for absorbance at λmax 284 nm. The absorbance range was determined and calibration curve was obtained (Figure 3).

Figure 3: Calibration curve of CBZ (8-18µg/ml).



Accuracy

The accuracy for the analytical procedure was determined at 80% (8 µg/ml), 100% (10 µg/ml), 120% (12 µg/ml) levels of tablet solution. Absorbance was measured at 284 nm and results were expressed in terms of % recoveries. Performed each level in triplicates and % RSD was calculated at each level (Table 2).

Table 2. Accuracy results of CBZ

Accuracy level	Amount added (µg/ml)	Amount recovered (µg/mL)	Average mean recovery ± SD	% RSD*
80	8	7.98	101.388±0.1079	0.1065
	8	8.15		
	8	8.19		
100	10	10.06	101.150±0.0459	0.0453
	10	10.13		
	10	10.15		
120	12	11.82	98.482±0.0112	0.0114
	12	11.80		
	12	11.82		

** n=3*

Method Precision

Repeatability

The precision of the method was checked repeatedly by analysis of 3 concentrations in 3 replicates (i.e. 10 µg/ml, 12 µg/ml, 14 µg/ml) standard solutions of CBZ. Absorbance of each concentration was measured at 284 nm. Percentage relative standard deviation values (% RSD) were calculated (Table 3).

Intermediate Precision

The inter-day and intra-day precision of this method was determined by analysing the corresponding responses 3 times on a same day and on 3 different days over a period of 1 week for standard solution of 14 µg/ml of CBZ. The results were reported in terms of relative standard deviation (RSD) (Table 3).

Table 3. Precision studies of CBZ

Drug	Concentration	Absorbance (Mean ± SD)	% RSD*
CBZ	10	0.5096 ± 0.0068	1.335
	12	0.5986 ± 0.0065	1.086
	14	0.7063 ± 0.0025	0.356
Intraday (n=3)	14	0.7006 ± 0.0015	0.2180
Interday (n=3)	14	0.7126 ± 0.0035	0.492

**n=3*

Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Six sets of known concentrations (8-18 µg/ml) were prepared. Calibration curve was plotted for each set. LOD and LOQ were calculated using the formulae as LOD= 3.3x SD/S and LOQ= 10x SD/S, where S is the average value of slopes of calibration plots and SD is calculated using values of y intercepts of regression equations (Table 4).

Robustness

Robustness of this method was determined by analysing the CBZ standard solution of 10µg/ml at different λmax (i.e. ±1) of actual λmax. Absorbance was measured. The standard deviation and percent relative standard deviation was calculated. There was no change in percent relative standard deviation of actual wavelength and different wavelength so the method is robust at different wavelength (Table 4).

Table 4. Summary of Validation Parameters

Parameters	Results
λmax	284 nm
Linearity Range (µg/ml)	8-18 (µg/ml)
Regression Equation (y=mx+c)	y =0.049x+0.009
Correlation Coefficient	0.999
Precision (% RSD) a. Repeatability (n=3) b. Intraday (n=3) c. Interday (n=3)	 0.356 0.2180 0.492
Accuracy (Mean % Recovery)	100.34
Limit of Detection (LOD) (µg/ml)	0.4546
Limit of Quantitation (LOQ) (µg/ml)	1.377
Robustness (% RSD)	1.1896

CONCLUSION

The proposed UV-spectrophotometric method was simple, precise, and accurate for determination of Carbamazepine in tablet dosage form. The method was validated as per the ICH guidelines for linearity, accuracy, precision, limit of detection (LOD), limit of quantitation (LOQ) and robustness. This method can be used for routine quality control analysis of Carbamazepine in bulk and pharmaceutical formulations.

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