e-ISSN: 2320-1215
p-ISSN: 2322-0112

RESEARCH AND REVIEWS: JOURNAL OF PHARMACY AND PHARMACEUTICAL SCIENCES

Method Development and Validation of Gliclazide in API and its Pharmaceutical Dosage Form by Uv-Visible Spectrophotometry.

Jugasmita Khatua, K Vanitha Prakash, G Manasa, and R Praveena.

Department of Pharmaceutical Chemistry, SSJ College of Pharmacy, Vattinagulapally, Gandipet, Andhra Pradesh, India.

Research Article

Received: 18/04/2014
Revised: 07/05/2014
Accepted: 13/05/2014

***For Correspondence**

Department of Pharmaceutical Chemistry, SSJ College of Pharmacy, Vattinagulapally, Gandipet, Andhra Pradesh, India.

Keywords: Gliclazide, UV-Visible Spectrophotometric Method, Bromo Cresol Green(BCG), Di Chloro Methane (DCM).

ABSTRACT

A simple, accurate, sensitive and reproducible visible spectrophotometric method has been developed for the determination of Gliclazide in bulk and also in its pharmaceutical dosage form. The proposed method was based on ion-complex of the drug with Bromo Cresol Green showing absorbance maxima at 411 nm respectively. Beer's law was obeyed in the range of 50-300 µg/mL , with molar absorptivity 1.044x 10^3 $L.mol^{-1}cm^{-1}$, relative standard deviation of the method was less than 1% and accuracy (average recovery %) was 94. All the variables were studied to optimize the reaction conditions. No interference was observed in the presence of common pharmaceutical excipients. The validity of the methods was tested by analyzing the drug in its pharmaceutical preparations. Good recoveries were also obtained. The developed method employed was successful for the determination of Gliclazide in various pharmaceutical preparation.

INTRODUCTION

Gliclazide[1] is an oral antihyperglycemic agent used for the treatment of non-insulin-dependent diabetes mellitus (NIDDM). Gliclazide chemically,1(Hexahydrocyclopenta(c)pyrrol-2(1H)yl)-3(p.tolylsulfonyl)urea (figure-1).

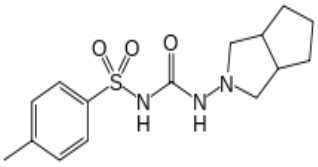


Figure 1: Gliclazide chemical structure.

Gliclazide is a white coloured powder, soluble in DCM, practically insoluble in water, freely soluble in methylene chloride, sparingly soluble in acetone, slightly soluble in alcohol. It belongs to the sulfonylurea class of insulin secretagogues, which act by stimulating β cells of the pancreas to release insulin. Gliclazide also has anti-platelet adhesive activity and reduces levels of free radicals, thereby preventing vascular complications.

The literature survey reveals that various methods has been reported for estimation of Gliclazide by UV spectrophotometric[2,3], RP-HPLC[4], simultaneous spectrophotometric method[5,6], HPLC[7], spectrometric method in combination with metformin[8,9], Analytical method[10].

e-ISSN: 2320-1215
p-ISSN: 2322-0112

MATERIALS AND METHODS

Instrument

Elico Double Beam - Ultra Violet - Visible Double Beam Spectrophotometer SL-244 with 1cm matched quartz cells was used for all spectral measurements.

Reagents

All the chemicals used were of *Analytical Reagent* grade and procured from SD Fine Chemicals (SDFC), Mumbai, India.

Preparation of Acetate buffer pH 3.0

Dissolve 12 g of sodium acetate in water, add 6 ml of glacial acetic acid and dilute with sufficient water to produce 100 ml.

Preparation of 0.1% Bromo Cresol Green (BCG) dye solution for 100 ml

Weigh 0.1 gm of dye sample and dissolve in small quantity of 20% ethanol or dissolve 0.04 g in 0.58 ml of 0.1M sodium hydroxide and make up to 100 ml with water and filter it using filter paper if necessary.

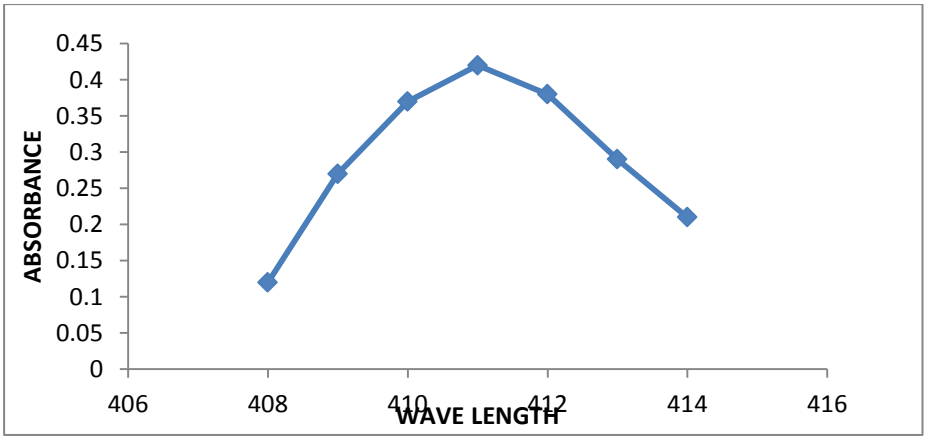
Preparation of standard solution of 1mg/ml stock solution

Weigh 0.1 gm of bulk drug (Gliclazide) and dissolve in 10 ml of DCM, shake well till it dissolves and make up to 100 ml with the same.

Determination of maximum absorbance

The standard solution of Gliclazide was scanned in the range of 400-800 nm which shows maximum absorbance at 411 nm **(Fig 2)**. Therefore, 411 nm wavelength was selected for the construction of calibration curve.

Figure 2: Absorption spectrum of Gliclazide with Bromocresol green



Procedure for pharmaceutical preparation (Tablet)

The method was extended for the determination of Gliclazide from tablet formulation. Ten tablets of Gliclazide were accurately weighed and powdered. Tablet powder equivalent to 100 mg of Gliclazide was dissolved in 10 ml of di-chloro methane, sonicated for 15 mins and filtered. The filtrate is combined and the final volume was made to 100 ml with di-chloro methane for the above method. The solution was suitably diluted and analyzed as given under the assay procedure for bulk sample.

e-ISSN: 2320-1215
p-ISSN: 2322-0112

RESULTS AND DISCUSSION

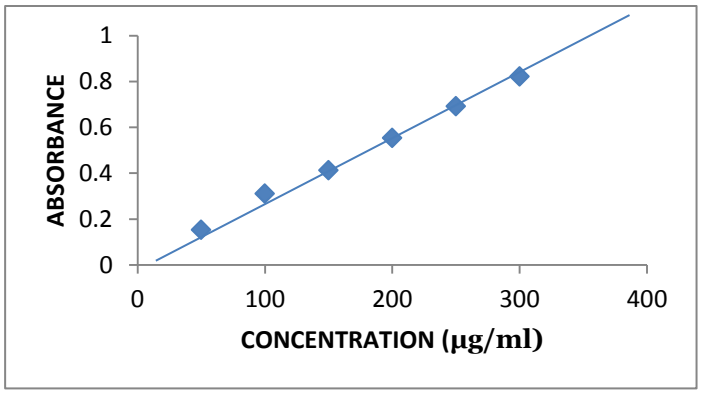
The chromogenic reagent is added to the drug i.e test solution and blank solution (here DCM is taken as blank) & it is shaken. To it suitable amount of chloroform and acetate buffer is added & shaken vigorously, so that the drug is extracted in chloroform layer then this is tested for absorbance in UV spectrophotometer.

The appropriate wavelength in the visible region (411 nm) was selected for the measurement of the active ingredients in the proposed method. The method was validated by linear fit curve and all the parameters were calculated. Linearity of the Gliclazide is plotted against absorbance and concentration and the linearity was graphically represented in **(table 1, figure 3)**

Table 1: Calibration curve

CONCENTRATION (µg/ml)	ABSORBANCE
50	0.153
100	0.311
150	0.412
200	0.554
250	0.691
300	0.821

Figure 3: Calibration curve for Gliclazide



The proposed method was satisfactorily applied to the determination of Gliclazide in its pharmaceutical preparations tablets, the results of the assay of the pharmaceutical preparation reveals that there is close agreement between the results obtained by the proposed method and the label claim (**Table 2**) and the results of the recovery values were close to 100% .

Table 2: Assay of Gliclazide in Tablet Formulation

Tablet Formulation	Amount Claim (mg/tablet)	*Amount obtained(mg) By Proposed Method	**%Recovery By the Proposed Method
1	80	75.6	94.5
2	80	76.2	95.3
3	80	74.7	93.4

The optical characteristics such as Maximum absorbance, Beer's law limits, Molar absorptivity and Sandell's sensitivity are presented in (**Table 3).**

The regression analysis using the method of least squares was made for slope (m), intercept (b) and correlation coefficient and the results are summarized in (**Table 3).**

LOD and LOQ

The LOD and LOQ were calculated from the equations,

e-ISSN: 2320-1215
p-ISSN: 2322-0112

LOD =3.3 σ/S and LOQ = 10 σ/S, where σ is the standard deviation of the lowest standard concentration and S is the slope of the standard curve. The results are shown in **table 3**

Table 3: Optical Characteristics and Regression Analysis of the Proposed Method Developed

Parameters	Results
Beers law limit (µg/ml)	50-300
Molar absorptivity(L.mol^{-1}cm^{-1})	1.044x 10^3
Sandell's sensitivity (micrograms/cm^2/0.001 Absorbance unit)	0.3095
Slope (m)	0.22
Intercept (c)	0.72
Standard error of estimate	0.0133
Correlation coefficient	0.986
% RSD	0.18
Limit of Detection (µg/ml)	0.010
Limit of Quantification (µg/ml)	0.036

CONCULSION

The proposed method was simple, economical, sensitive, precise reliable and reproducible for the routine estimation of Gliclazide in bulk as well as in tablet formulation.

ACKNOWLEDGMENT

The authors are very thankful to Principal and management of SSJ College of Pharmacy for providing necessary facilities to carry out research work.

REFERENCES

1. Merck Index. An encyclopedia of chemical drugs and biologicals, twelth edition 1996, 9811.
2. McGavin JK, Perry CM and Goa KL. Gliclazide modified release. Drugs. 2002; 62 (9): 1357-1364.
3. Amruta B Loni, Minal R Ghante, SD Sawant. Simultaneous UV Spectrophotometric Method for Estimation of Gliclazide and Metformine Hydrochloride in Tablet Dosage Form. Der Pharma Chemica, 2012, 4 (3): 854-859.
4. Samina A Jamadar, Snehal P Mulye, Poonam S Karekar, Yogesh V Pore, Kishor B Burade. Development and validation of UV spectrophotometric method for the determination of Gliclazide in tablet dosage form. Der Pharma Chemica. 2011;3(4):338-343.
5. BVV Ravi Kumar, et al. A RP-HPLC Method Development and Validation for the Estimation of Gliclazide in bulk and Pharmaceutical Dosage Forms. J App Pharm Sci. 2013;3(4):059-062.
6. K Moitra, PN Murthy, and Uma S. Mishra Simultaneous spectrophotometric analysis of rosiglitazone maleate and gliclazide in bulk drug and pharmaceutical formulation. Der Pharmacia Sinica, 2012;3(2):170-176.
7. Uttam Singh Baghel. Gradient HPLC method development and validation for Simultaneous estimation of Rosiglitazone and Gliclazide Department of Pharmaceutical Analysis, Journal of Tropical Disease. Asian Pacific Journal of Tropical Disease. 2012:S756-S760.
8. Ketan P Dadhania, Parthika. Simultaneous estimation of Gliclazide and metformin hydrochloride in bulk and tablet dosage form, source Der Pharma Chemica. 2011;3(4):338.
9. PN Dhabale, CR Seervi. Simultaneous UV Spectrophotometric Method for Estimation of Gliclazide and MetformineHydrochloride in Tablet Dosage Form. Int J Chem Tech Res.
10. Harish Kumar Arjariya. Analytical method development approach for gliclazide tablets. J Pharm Pharm Sci (Accepted).