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Development and validation of UV spectrophotometric method for the determination of Gliclazide in tablet dosage form

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ABSTRACT

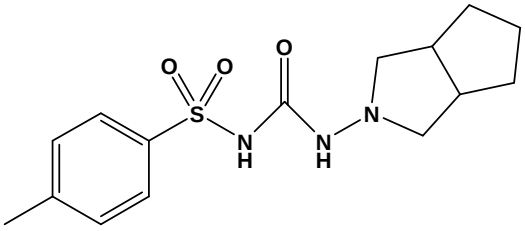
The article describes a simple, sensitive, rapid, accurate and precise spectrophotometric method for the estimation of gliclazide in bulk and pharmaceutical dosage forms. The wavelength maxima for gliclazide was found to be 229.5 nm with molar absorptivity of 1.4962×10^{4}l/mol/cm. Beer's law was obeyed in the concentration range of 7-27 µg/ml. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.31 µg/ml and 0.92 µg/ml, respectively. The percentage recovery of the drug for the proposed method ranged from 98.68-100.12% indicating no interference of the tablet excipients. The results demonstrate that proposed method is accurate, precise, and reproducible while being simple and rapid too for the determination of gliclazide in tablet dosage form.

Keywords: Gliclazide, UV spectroscopy, validation, tablets.

INTRODUCTION

Gliclazide [1-(1-azabicyclo (3, 3, 0) octyl)-3-(p-tolylsulphonylurea)] (Figure 1), is an oral hypoglycemic drug, belonging to second-generation sulphonylureas [1, 2], used in type-II diabetes [2].

Figure 1 Chemical structure of gliclazide



Gliclazide may reverse insulin resistance in type-II diabetic patients and also improves defective insulin secretion. It is readily absorbed from the gastro-intestinal tract with peak concentrations in plasma occurring about 2-4 h and it is highly protein bound [3].

Different analytical methods including radioimmunoassay [4], gas chromatography [5], HPLC [6, 7], Evaporative Light Scattering Detection [8], Charged Aerosol Detection [8] and simultaneous spectrophotometric estimation of gliclazide and metformin hydrochloride in combined dosage forms [9] have been reported for determination of gliclazide. However, no UV spectrophotometric method is available for quantitative determination of gliclazide in its pharmaceutical dosage forms. Some reported analytical methods involve time consuming and laborious extraction steps [5, 6], lengthy retention time or large volume of biological samples[5, 6], use of mass spectroscopy for detection and identification of drug [10] or solid phase extraction processes[11].

The objective of work was to develop simple, rapid, accurate and specific UV spectrophotometric method for the estimation of gliclazide in pharmaceutical dosage forms. The method was further validated for the parameters like precision, accuracy, sensitivity and linearity. The limit of detection (LOD) and limit of quantification (LOQ) were also determined. The results of analysis were validated statistically and by recovery studies. The proposed method is recommended for routine analysis since it is rapid, simple, accurate, sensitive and also specific by no heating and no organic solvent extraction.

MATERIALS AND METHODS

2.1. Materials

Gliclazide was obtained as a gift sample from Ajanta Pharmaceuticals Ltd., Mumbai, India. Gliclazide tablets were procured from local pharmacy. All the reagents were of analytical grade. Glass double distilled water was used throughout the experiment. A Shimadzu UV/VIS 1700 spectrophotometer with 1 cm matched quartz cells were used for the estimation.

2.2. Preparation of standard stock solution of gliclazide

An accurately weighed 5 mg of gliclazide was dissolved in 10 ml of methanol in a 50 ml volumetric flask and the volume was adjusted up to the mark with distilled water to obtain a stock solution of 100 µg/ml. The solution was filtered through Whatman filter paper No. 41.

2.3 Determination of λ_{max}

An appropriate aliquot portions of 0.7 to 2.7 ml of stock solution were transferred to a series of 10 ml volumetric flasks and volume in each flask were adjusted to 10 ml with distilled water to obtain a concentration of range of 7-27 µg/ml. One of the solutions was scanned in UV range of 200-400 nm using methanol: distilled water (1:4) as a blank and wavelength of maximum absorption was found to be 229.5 nm. The absorbance of solutions was measured at 229.5 nm against blank and calibration curve of gliclazide was constructed.

2.4. Preparation of Sample

Twenty tablets of gliclazide were weighed and finely powdered. Amount equivalent to 5 mg was transferred to 50 ml volumetric flask, dissolved in 10 ml of methanol and made up the volume with distilled water to obtain a concentration of 100 µg/ml. The solution was filtered through Whatman filter paper No. 41 and filtrate was diluted to obtain concentration in between linearity range. The absorbance of sample solution was measured and amount of gliclazide was determined by referring to the calibration curve. Recovery studies were carried out to judge the accuracy of the method at 80, 100 and 120% level by adding a known quantity of pure drug to the preanalyzed formulation and the proposed method was followed. From the amount of drug found, percentage recovery was calculated.

RESULTS AND DISCUSSION

3.1. Optical characteristics

Tab. 1 represents the optical characteristics and precision of the proposed method for estimation of gliclazide.

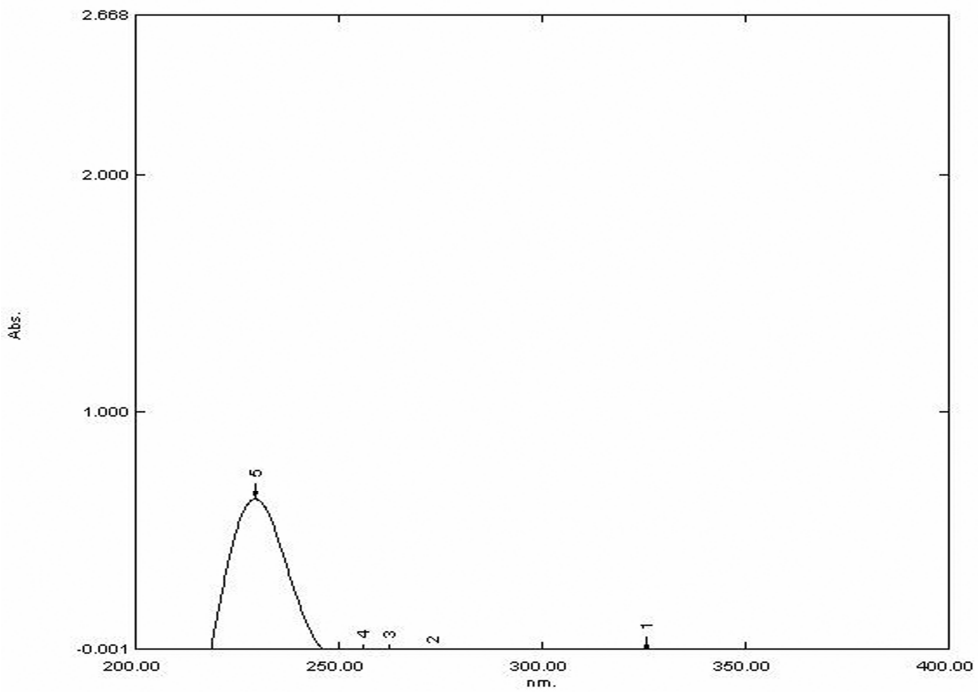
Table 1Validation parameters for standard gliclazide

Parameter	Value
λ_{max} (nm)	229.5
Beer's range (µg/ml)	7-27
Molar absorptivity (l/mol/cm)	1.4962×10^4
Sandell's sensitivity (µg/cm^2/0.001AU)	0.021616
Correlation coefficient (r^2)	0.9998
Regression equation	y = 0.0463x-0.2104
Intercept (a)	-0.2104
Slope (b)	0.0463
Limit of detection (LOD µg/ml)	0.31
Limit of quantification (LOQ µg/ml)	0.92
Precision (% RSD)*	0.56

** Indicates mean of six determinations (n=6); RSD: Relative standard deviation*

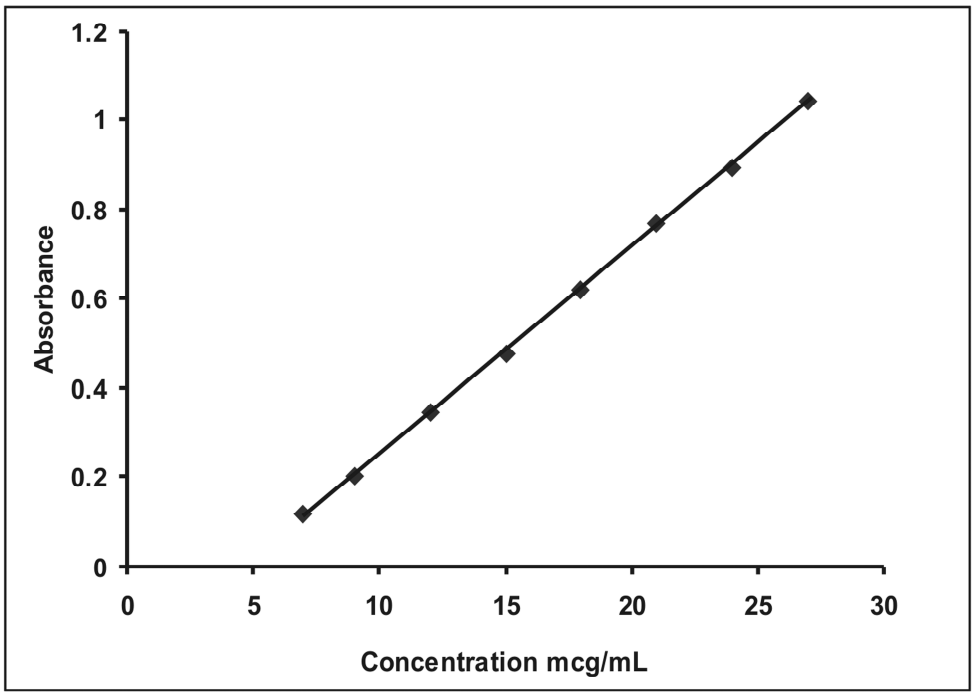
The proposed method of determination of gliclazide showed molar absorptivity of 1.4962×10^4 l/mol/cm and Sandell's sensitivity 0.021616mcg/Sq.cm/0.001-absorbance units. Gliclazide exhibits its maximum absorption at 229.5 nm (Figure 2).

Figure 2 λ_{max} of gliclazide in methanol: distilled water (1:4)



The linearity range for gliclazide at this wavelength was found to be 7-27 µg/ml (Figure 3).

Figure 3 Calibration curve of gliclazide in methanol: distilled water (1:4) at 229.5 nm



Linear regression of absorbance on concentration gave equation y = 0.0463x–0.2104 with a correlation coefficient of 0.9998. Relative standard deviation of 0.56 was observed for analysis of 6 replicate samples, indicating developed method is precise.

3.2. Limit of detection (LOD) and limit of quantification (LOQ)

Limit of detection (LOD) and limit of quantification (LOQ) decide about the sensitivity of the method. LOD is the lowest detectable concentration of the analyte by the method while LOQ is the minimum quantifiable concentration. LOD and LOQ were calculated by Eqs. LOD = $\frac{3.3\delta}{s}$ (1) and LOQ = $\frac{10\delta}{s}$ (2), respectively, where δ is the standard deviation of blank and s is slope of calibration [12].

The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.31 µg/ml and 0.92 µg/ml respectively (Table 1) indicating proposed UV method is highly sensitive.

3.3. Analysis in tablets formulations, accuracy and reproducibility

The proposed method has successfully estimated the amount of gliclazide in the range of 98.56-99.14% in all tested formulations (brands). The accuracy and specificity of the proposed method was checked by recovery experiments (Table 2). The percentage recovery values for Glizid and Gylis were found to be 98.68% and 100.12% respectively (Table 2).

Table 2 Results of analysis of formulations and recovery studies

Formulations (Brand)	Label Claim (mg)	% Estimated*	%RSD	% Recovery*	%RSD
Glizid	80	99.82	0.93	98.68	1.23
Gylis	40	99.24	0.95	100.12	0.88

** Indicates mean of six determinations (n=6); RSD: Relative standard deviation.*

The high recoveries with low % RSD values indicated that the method had a good accuracy and specificity, as there was no interference from the excipients present in formulations.

Precision is the degree of repeatability of an analytical method under normal operational conditions. The precision of the method was determined by repeatability (intraday) and intermediate precision (inter-day) and reported as RSD % for a statistically significant number of replicate measurements. Intra-day precision and accuracy were determined by analyzing replicate samples of different concentrations, prepared on same day. Inter-day variability was evaluated by analyzing two concentrations on three different days. The % RSD values reported in Table 3 shows an acceptable intra-day and inter-day variation of gliclazide for the proposed method indicating accuracy and reproducibility of the assays.

Table 3 % RSD values for repeatability, intra- day, inter-day variation and ruggedness

Formulations (Brands)	Parameters				
	Repeatability	Precision		Ruggedness	
		Intra-day	Inter-day	Analyst 1	Analyst 2
Glizid	1.13	0.87	1.52	0.58	1.32
Gylis	0.79	0.97	0.85	0.29	0.51

RSD: Relative standard deviation; (n = 6).

3.4. Ruggedness and robustness

The ruggedness of developed method was checked by analyzing gliclazide by different analysts at similar operational and environmental conditions. The % RSD values were found to be less than 2% (Table 3).

Robustness of the proposed method was determined by estimating a drug at slightly different wavelength from the selected wavelength. No significant difference was found in the absorbance of samples. Therefore, the proposed method was considered as robust.

CONCLUSION

The developed method was found to be sensitive, accurate, precise, reproducible and linear over the concentration range studied. The proposed method can be used for the routine quality control analysis of gliclazide in bulk drugs and pharmaceutical dosage forms. .

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