EUROPEAN JOURNAL OF BIOMEDICAL AND PHARMACEUTICAL SCIENCES
http://www.ejbps.com

ISSN 2349-8870
Volume: 11
Issue: 6
154-158
Year: 2024

DEVELOPMENT AND VALIDATION OF UV SPECTROSCOPIC METHOD FOR ESTIMATION OF GLIPIZIDE IN TABLET DOSAGE FORM

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Article Received on 05/04/2024 **Article Revised on 26/04/2024** **Article Accepted on 16/05/2024**

ABSTRACT

Objective: To develop and validate simple, rapid, linear, accurate, precise and economical UV Spectroscopic method for estimation of Glipizide in tablet dosage form. **Methods:** The drug is freely soluble in 0.1N NaOH.[1] The drug was identified in terms of solubility studies and on the basis of melting point done on melting point apparatus of Equiptronics.[2] It showed absorption maxima were determined in 0.1N NaOH. The drug obeyed the Beer's law and showed good correlation of concentration with absorption which reflect in linearity.[3, 4, 5, 6] The UV spectroscopic method was developed for estimation of Glipizide in tablet dosage form and also validated as per ICH guidelines.[7] **Results:** The drug is freely soluble in 0.1N NaOH, slightly soluble in methylene chloride and practically insoluble in water and ethanol. So, the 0.1N NaOH is used as a diluent in method. The melting point of Glipizide was found to be 208-209˚C (uncorrected). It showed absorption maxima 272 nm in 0.1N NaOH. On the basis of absorption spectrum the working concentration was set on 20 µg/ml (PPM). The linearity was observed between 10-30 µg/ml (PPM). The results of analysis were validated by recovery studies. The recovery was found to be 98.7, 101 and 99.2% for three levels respectively. The % RSD for precision was found to be 0.62%. **Conclusion:** A simple, rapid, linear, accurate, precise and economical UV Spectroscopic method has been developed for estimation of Glipizide in tablet dosage form. The method could be considered for the determination of Glipizide in quality control laboratories.

KEYWORDS: Glipizide, UV Spectrophotometer, Melting Point, Assay Method, Validation, Accuracy, Linearity, Ruggedness, Precision.

INTRODUCTION

Glipizide, N-[2(4{[(Cyclohexyl carbamoyl)amino]sulfonyl}phenyl)ethyl]-5-methyl pyrazine-2-carboxamide.[1] It was first introduced in 1984 and is available in various countries including Canada and the U.S. According to the 2018 Clinical Practice Guidelines by Diabetes Canada, sulfonylurea drugs are considered a second-line glucose-lowering therapy following metformin. Mechanism of action of Glipizide is to stimulate insulin release from the pancreatic beta cells; for this reason, they are only effective when the patient has some residual pancreatic beta cell activity.[2] The sulfonylureas act by closing the ATP-sensitive potassium (KATP) channels in the cell membrane of the pancreatic beta cells and therefore cause: membrane depolarisation, calcium influx and insulin release.[3] Compared to other members of the sulfonylurea drug group, glipizide displays rapid absorption and onset of action with the shortest half-life and duration of action, reducing the risk for long-lasting hypoglycemia that is often observed with blood glucose-lowering agents. Glipizide was first approved by the FDA in 1994 and is available in extended-release tablets under the brand name Glucotrol®, as well as in combination with metformin under the brand name Metaglip®.[4,5]

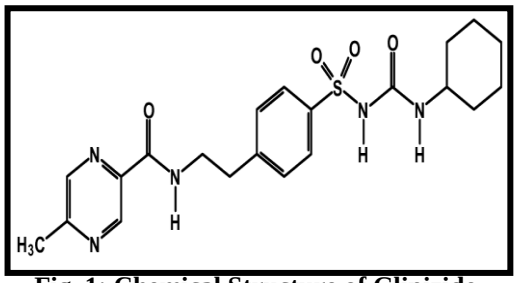

Fig. 1: Chemical Structure of Glipizide.

From literature review it's found that one method was reported on spectrophotometry for simultaneous estimation of Glipizide their combined dosage form.[6]

Also the method was reported on HPTLC[7] on Glipizide. Lot of work was done on HPLC method development for Glipizide drugs.[8-12] But very few methods were reported on estimation of Glipizide in tablet dosage form for UV spectroscopic method. This indicates that so far no UV method exists for the estimation and determination of Glipizide in tablet dosage forms.

MATERIALS AND METHODS

- **Instruments**

Shimadzu double beam UV-visible spectrophotometer 1700 Ultra with matched pair Quartz cells corresponding to 1 cm path length and spectral bandwidth of 1 nm, Bath sonicator and citizen weighing balance.
Melting point apparatus of Equiptronics were used.

- **Materials**

Glipizide was obtained as a gift sample. Glipizide tablets were procured from local pharmacy. 0.1N NaOH was used throughout the experiment as a diluent. Freshly prepared solutions were employed.

Method development

A. Determination of ƛ max (20 PPM)[13, 14]

100 mg weighed amount of Glipizide was dissolved into 100 ml of volumetric flask with 01.N NaOH. Pipette out 2 ml and added in 100 ml of volumetric flask dissolved and diluted up to the mark with 0.1N NaOH. This solution was subjected to scanning between 200-400 nm and absorption maximum was determined.

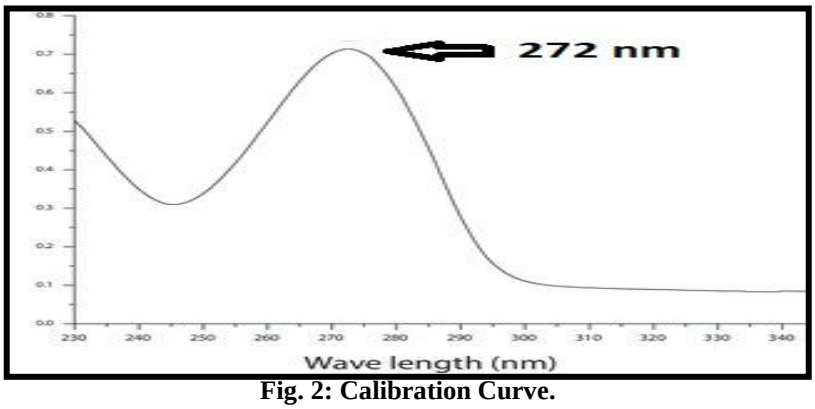

Fig. 2: Calibration Curve.

B. Preparation of Working concentration[14]

Preparation of Standard stock solution

Standard stock was prepared by dissolving 100 mg of Glipizide in 100 ml of 0.1N NaOH to get concentration of 1000 µg/ml (PPM).

Preparation of Standard solution

Pipette out 2 ml from standard stock solution and diluted up to 100 ml with 0.1N NaOH to get concentration of 20 µg/ml (PPM).

C. Preparation of Working concentration

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D. Procedure for UV reading

Blank Solution: (For Auto zero)

Fill the cuvette with 0.1N NaOH. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

Standard Solution

Fill the cuvette with standard solution. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

Sample Solution

Fill the cuvette with sample solution. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

E. Procedure for sample preparations

For analysis of commercial formulations; twenty tablets are taken weighed it and powdered. The powder equivalent to 100 mg of Glipizide was accurately weighed and transferred into the 100 ml of volumetric flask, added 60 ml 0.1N NaOH, the solution was sonicated for 20 min. After sonication cool the flask and diluted upto 100 ml with 0.1N NaOH. Filtered the solution through whatmann filter paper. Pipette out 2 ml of the above solution and diluted up to 100 ml with 0.1N NaOH. The absorbance was measured at 272 nm. The absorbance was recorded.

Table 1: Absorbance of Dosage Form.

USV Pharmaceutical Limited (10 mg)		
Sr. no.	**Sample**	**Absorbance**
1	Blank	0.0001
2	Standard	0.7294
3	Sample	0.7214

Table 2: Dosage Form Specifications.

Type	Company	M.D.	E.D.	Batch No.	Average weight (g)	Assay (%)
1	USV Pharma LTD GLYNASE® XL (10mg)	05/2023	07/2026	SH 489	0.084	98.90

F. Method of validation[15-18]

The proposed method was developed by using linearity, accuracy, precision and ruggedness as per ICH guidelines, 1996.

Linearity

The linearity of the proposed assay was studied in the concentration range 10 - 30 PPM at 272nm. The calibration data showed a linear relationship between concentrations.

Table 3: Linearity Studies.

Sr. no.	Sample Concentration	Absorbance
1	10 PPM	0.3501
2	15 PPM	0.5494
3	20 PPM	0.7201
4	25 PPM	0.9112
5	30 PPM	1.0810
Correlation coefficient		**0.9993 ~ 0.999**

Accuracy

To ensure the accuracy of the method, recovery study was performed by preparing 3 sample solutions of 80, 100 and 120% of working concentration and adding a known amount of active drug to each sample solution and dissolved in 100ml of volumetric flask with 0.1N NaOH and measuring the absorbance at 272nm.

Table 4: Accuracy Studies.

SPECTROPHOTOMETRIC METHOD			
Accuracy (%)	**Qty weighed (mg)**	**Qty found (mg)**	**Recovery (98-102%)**
80	0.8	0.81	100.92
100	1	1.02	101.86
120	1.2	1.18	98.55

Precision

The precision of the method was demonstrated by inter-day and intra-day variation studies. Five sample solutions were made and the % RSD was calculated.

Table 5: Precision studies.

Sr. No.	Sample Solution	Absorbance
1	Sample Solution 1	0.7304
2	Sample Solution 2	0.7254
3	Sample Solution 3	0.7311
4	Sample Solution 4	0.7326
5	Sample Solution 5	0.7212
MEAN		**0.7281**
SD		**0.0047**
% RSD		**0.6494 ~ 0.65**

Ruggedness

Ruggedness is a measure of the reproducibility of a test result under normal, expected operating condition from instrument to instrument and from analyst to analyst.

Table 6: Results for Ruggedness Studies.

Sr. No.	Analyst	Results	Mean	% Assay	% RSD
1	Analyst 1	0.7219	0.7223	99.02	0.4100 ~ 0.41
		0.7226			
2	Analyst 2	0.7245	0.7265	99.60	
		0.7284			

RESULTS

1. Solubility of Glipizide

Solubility test was passed as per criteria.

Table 7: Results for solubility studies.

Sr. no.	Title	Result
1	0.1N NaOH	Freely Soluble
2	Methylene Chloride	Slightly soluble
3	Water and Ethanol	Practically insoluble

2. Melting point of Glipizide

The melting point of Glipizide was found to be 208-209˚C (uncorrected).

3. Results for linearity for assay method of Glipizide

The linearity of method was determined at concentration level ranging from 10 to 30 µg/ml (PPM). The correlation coefficient value was found to be **(R^2) 0.9993 ~ 0.999.**

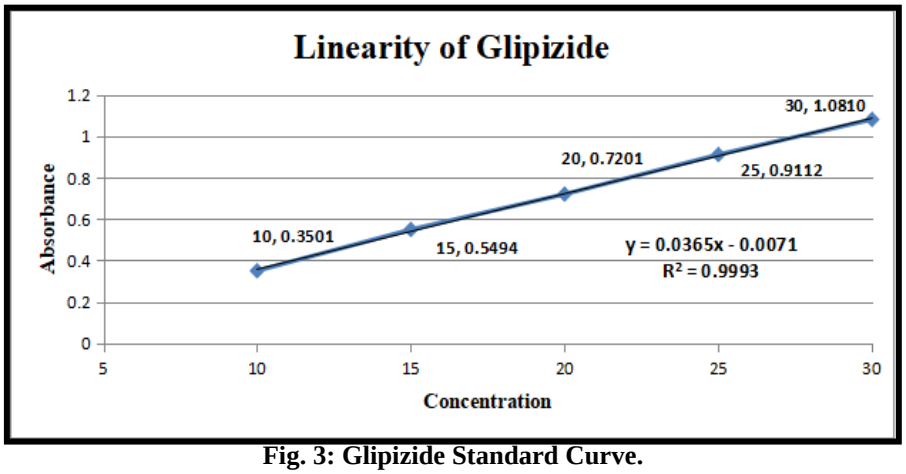

Fig. 3: Glipizide Standard Curve.

4. Results for accuracy for assay method of Glipizide

The accuracy of the method was determined by recovery experiments. The recovery studies were carried out and the percentage recovery were calculated and represented in Table - 4. The high percentage of recovery indicates that the proposed method is highly accurate. Accuracy results were found within acceptance criteria that are within 98-102%.

5. Results for precision for assay method of Glipizide

The % RSD for different sample of precision was found to be 0.65 and it is within acceptance criteria represented in Table - 5.

6. Results for ruggedness for assay method of Glipizide

The % RSD for different sample of ruggedness was found to be 0.41 and it is within acceptance criteria represented in Table - 6.

CONCLUSION

A method for the estimation of Glipizide in tablet form has been developed. From the spectrum of Glipizide, it was found that the maximum absorbance was 272 nm in 0.1N NaOH. A good linear relationship was observed in the concentration range of 10-30 µg/ml (PPM). The high percentage recovery indicates high accuracy of the method. This demonstrates that the developed spectroscopic method is simple, linear, accurate, rugged and precise for the estimation of Glipizide in solid dosage forms. Hence, the method could be considered for the determination of Glipizide in quality control laboratories.

ABBREVARTIONS

1. PPM - Parts per Million
2. nm - Nanometer
3. HPLC - High Performance Liquid Chromatography
4. UV - Ultra violet
5. KATP - ATP-sensitive potassium
6. FDA - Food and Drug Administration
7. NaOH - Sodium Hydroxide
8. ICH - International Council for Harmonization
9. RSD - Relative Standard Deviation
10. SD - Standard Deviation
11. Qty - Quantity
12. C - Celsius
13. M.D. - Manufacturing Date
14. E.D. - Expiry Date

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