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An Innovative UV-Visible Spectrophotometric Approach for the Quantitative Assessment of Glibenclamide in API and Dosage Form

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Abstract:

- ***Background:***

Glibenclamide, a second-generation sulfonylurea, is widely used to treat type 2 diabetes. The precise measurement of this medication is essential to ensuring the quality and therapeutic efficacy of drug products.

- ***Objective:***

The current work is intended to develop and validate a simple and cost-effective UV-visible spectrophotometric approach for quantifying Glibenclamide in distilled water.

- ***Methods:***

To improve solubility, Glibenclamide was dissolved in a tiny proportion of methanol before being diluted with distilled water to make a standard solution. Absorbance was measured at a maximum wavelength of 324 nm. According to the ICH Q2(R1) criteria, the technique was validated by finding the limit of detection (LOD) and limit of quantification (LOQ), which examined critical aspects such as linearity, accuracy, precision, specificity, robustness, and sensitivity.

- ***Results:***

The novel approach showed a good linear response from 2-10 µg/mL, with a correlation value (R^2) 0.9991. The accuracy was proven by recovery outcomes ranging from 98.47% to 101.21%. Precision was proved by keeping %RSD assessments below 2%. The detection and quantification limits were 0.35µg/mL and 1.06µg/mL. Furthermore, the approach demonstrated significant specificity and remained steady despite minor changes in analytical conditions, illustrating its robustness.

- ***Conclusion:***

The validated UV-visible spectrophotometric technique for routine testing of Glibenclamide in distilled water proved trustworthy, consistent, and practical. It provides an efficient option for labs without access to sophisticated equipment like HPLC.

Keywords: *Glibenclamide, UV-Visible Spectrophotometry, Method Validation, ICH Q2 (R1).*

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I. INTRODUCTION

Glibenclamide, often known as glyburide, is an anti-diabetic drug used to treat type 2 diabetes. It is sulfonyl compound chemically, 5-chloro-N-[2-[4-cyclohexyl carbamoyl sulfamoyl) phenyl] ethyl]-2-methoxy benzamide (Fig 1). It is employed as pancreatic or extrapancreatic, enhancing insulin release from beta cells [1].

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It elevates insulin production from pancreatic β-cells and improves peripheral glucose utilisation, effectively regulating blood glucose levels in non-insulin-dependent diabetic patients. Glibenclamide's narrow therapeutic index requires precise and consistent measurement in pharmaceutical products and biological systems to ensure effectiveness and safety [2].

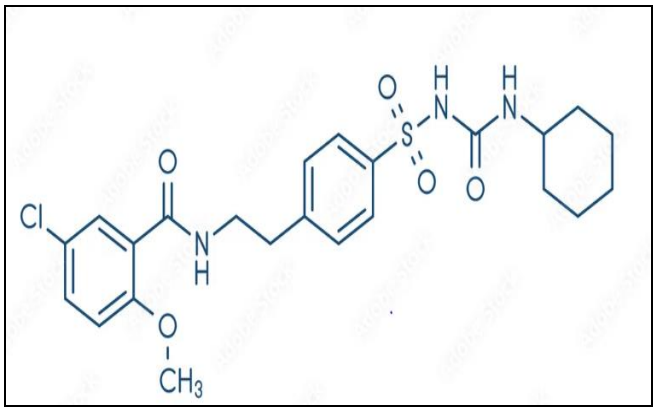

Fig 1 Chemical Structure of Glibenclamide

Numerous analytical approaches have been established for estimating Glibenclamide, including high-performance liquid chromatography (HPLC), gas chromatography (GC), and liquid chromatography-mass spectrometry (LC-MS) [3 & 4]. Although these approaches have excellent sensitivity and selectivity, they need expensive equipment, extensive sample preparation, and specialised operating experience, which might restrict their use in regular quality monitoring, especially in poor countries [5].

A practical alternative for Glibenclamide analysis is UV-visible spectrophotometry. It is highly regarded for its simplicity, cheap cost, quick execution, and little instrumentation needs [6]. Previous research has shown that it is effective for assessing Glibenclamide levels in bulk and formulated goods. Due to the compound's low water solubility often employs organic solvents such as methanol or ethanol as diluents [7].

In contrast, relying on organic solvents may not be appropriate in some applications, such as dissolution testing, environmental monitoring, or aqueous system analysis. Some research has tried circumventing solubility constraints using co-solvent systems or surfactants [9]. However, there is still a desire for a validated UV spectrophotometric approach specially designed for measuring Glibenclamide in pure water with minimum methanol.

This study aims to develop and verify a simple, accurate, and cost-effective UV-visible spectrophotometric technique for determining Glibenclamide in distilled water per the International Council for Harmonisation (ICH) Q2 (R1) criteria. The method will be tested using validation criteria such as linearity, accuracy, precision, specificity, robustness, limit of detection (LOD), and limit of quantification (LOQ). This research is significant for labs in resource-constrained areas where access to sophisticated chromatographic methods is restricted [8].

II. MATERIALS AND METHODS

➢ *Materials*

Glibenclamide pure drug was obtained from Shreeji Pharma International as a gift sample. Distilled water was available in the research lab from the distillation unit (Vaagdevi Pharmacy College, Bollikunta, Warangal, and TG). All other chemicals and solvents used were of analytical grade.

➢ *Methods*

- *Preparation of Standard Solution*

In order to produce a standard stock solution of Glibenclamide at 100µg/mL, 10 mg of pure Glibenclamide was carefully weighed and placed into a 100 mL volumetric flask. About 10 mL of methanol was added to assist in dissolving, and the mixture was sonicated for 10 minutes until completely dissolved. The solution was diluted to the desired amount with distilled water and well mixed. To generate working standard solutions with concentrations ranging from 2 to 10µg/mL, aliquots were transferred into 10 mL volumetric flasks and diluted with distilled water. These working standards created the calibration curve for Glibenclamide [9].

- *Preparation of Sample Solution (Tablet Formulation)*

Around twenty Glibenclamide tablets were weighed and pulverised to a fine powder. A piece of the powder comprising 10 mg of Glibenclamide was precisely placed into a 100 mL volumetric flask. To help extract the active component, about 10 mL of methanol was added, and the mixture was sonicated for 15 minutes. The solution was then filtered using a 0.25µm filter. Dilute the filtrate to 100 mL with distilled water to create a stock solution with a theoretical 100µg/mL concentration. Aliquots from this stock were diluted with distilled water to reach concentrations within the calibration range of 2-10 µg/mL for analytical purposes [8].

- *Determination of Maximum Absorbance*

The UV absorption spectra of the reference solution were scanned between 200 and 400 nm, and the wavelength with the highest absorbance, 324 nm, was chosen to ensure subsequent experiments.

- *Calibration Curve (Linearity Assessment)*

The concentrations ranging from 2 to 10 µg/mL were produced, and their absorbance was measured at 324 nm. A calibration curve was then created by graphing absorbance vs concentration.

- *Method Validation (As per ICH Q2(R1))*

- *Accuracy:*

Recovery levels were measured at 80%, 100%, and 120% of the target concentration.

- *Precision:*

At a six µg/mL concentration, changes were evaluated intra-day and inter-day to assess repeatability.

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- *LOD and LOQ:*

The limits of detection (LOD) and quantification (LOQ) were determined using the formulas LOD = 3.3σ/S and LOQ = 10σ/S, where σ represents the standard deviation of the response, and S is the slope of the calibration curve.

- *Specificity:*

The blanks of distilled water and methanol were investigated for any possible interference in absorbance.

- *Robustness:*

Researchers studied the influence of purposeful variations in wavelength (±2 nm) and temperature [9].

III. RESULTS AND DISCUSSION

➢ *Maximum Absorbance Wavelength*

Glibenclamide exhibited maximum absorbance at 324 nm, which was used as the detection wavelength for all quantitative experiments, as shown in Fig 2.

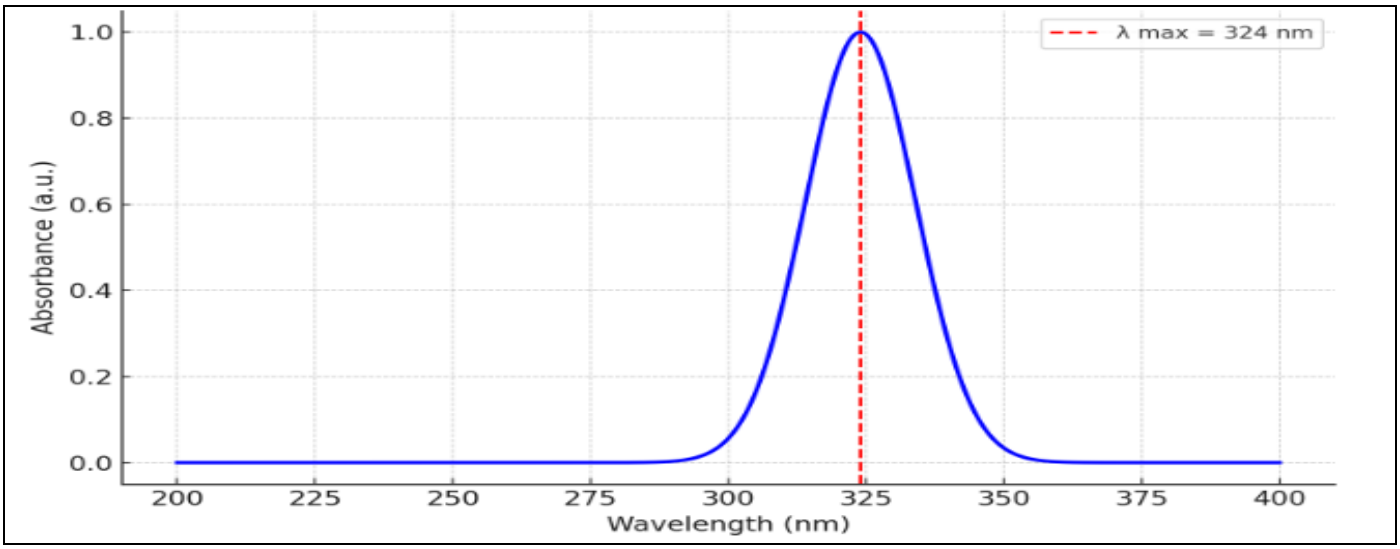

Fig 2 Absorption Spectrum Showing a Peak at 324 nm

➢ *Linearity*

The method demonstrated strong linearity across the 2–10 µg/mL concentration range, with a correlation coefficient (R^2) of 0.9991. This linear response is detailed in Table 1 and further illustrated in Fig 3, confirming the method's reliability within the specified range.

Table 1 Data of Linearity

Concentration (µg/mL)	Mean absorbance ± SD
2	0.152 ± 0.002
4	0.303 ± 0.004
6	0.450 ± 0.003
8	0.602 ± 0.005
10	0.750 ± 0.006

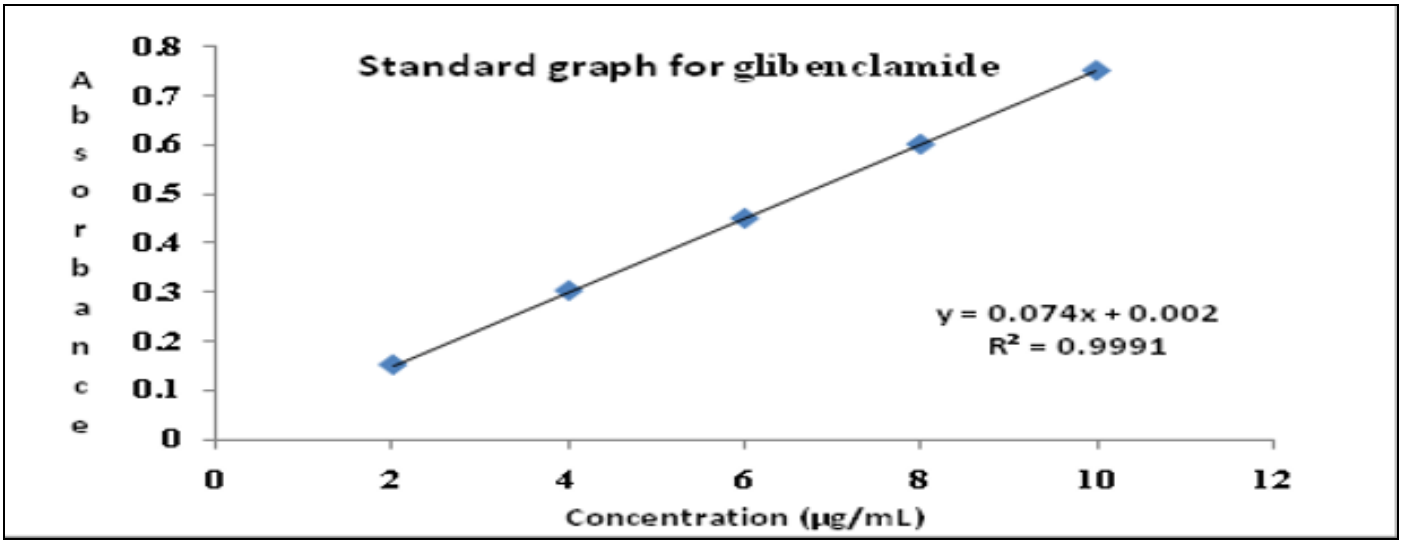

Fig 2 Standard Graph for Glibenclamide

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➢ *Accuracy*

The method's accuracy was confirmed by recovery trials, which yielded per cent recoveries ranging from 98.47% to 101.21% (Table 2).

Table 2 Results of Accuracy

Level	Amount Added (µg/mL)	% Recovery ± SD
80%	4.8	98.47 ± 0.92
100%	6.0	99.83 ± 0.64
120%	7.2	101.21 ± 0.71

➢ *Precision*

Intra-day and inter-day studies showed %RSD values of less than 2%, suggesting high repeatability, as shown in Table 3.

Table 3 Results of Precision

Parameter	Concentration (µg/mL)	%RSD
Intra-day	6.0	1.22
Inter-day	6.0	1.47

➢ *LOD and LOQ*

The calculated LOD and LOQ were 0.35 µg/mL and 1.06 µg/mL, respectively, reflecting the method's high sensitivity.

➢ *Specificity*

In the vicinity of 324 nm, no absorbance interference was recorded from the blank solutions, indicating that the approach is selective for detecting Glibenclamide.

➢ *Robustness*

The approach demonstrated resilience, as it remained steady and consistent despite minor fluctuations in wavelength and temperature.

IV. CONCLUSION

A UV-visible spectrophotometric approach was successfully developed and validated for quantifying Glibenclamide in distilled water. This strategy displayed high accuracy, precision, and consistency throughout the concentration range tested. Following the ICH Q2 (R1) validation requirements, it appears to be a reliable instrument for routine quality evaluation, especially in contexts where sophisticated analytical procedures such as HPLC are inaccessible.

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