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QUANTIFICATION OF SILODOSIN IN PHARMACEUTICALS EMPLOYING MULTIVARIATE CALIBRATION TECHNIQUE BY UV SPECTROPHOTOMETRIC AND EVALUATION OF GREENNESS PROFILE

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ABSTRACT

According to the current study, a novel methodology should be developed and tested in order to ascertain the bulk and formulation concentrations of silodosin. This analytical method can be described as sensitive, exact, fast. Utilising a multivariate calibration process and a UV spectrophotometer, the analytical approach was conducted. We utilized the established relationship between concentration and absorbance at five distinct wavelengths, including the absorption maxima at 269 nm, to construct this multivariate calibration approach. The linear regression equations were employed in conjunction with other mathematical and statistical methods to validate the process in accordance with the ICH Q2 (R1) requirements. The instructions' validation parameters for linearity, precision, and other factors have all been met. This technology is dependable, sensitive, and economical when used to routine drug and formulation examinations. The analytical, Agree metrics, Eco scale and Green Analytical Procedure Index were used to calculate the technique greenness values.

Keywords: Silodosin, Multivariate calibration technique, UV spectrophotometric, Pharmaceutical formulations, ICH guidelines, Validation

INTRODUCTION

The drug silodosin is used to treat benign prostatic hyperplasia symptomatically. It functions as an antagonist of α1adrenoceptors with a high

degree of uroselectivity, or prostate-specific selectivity. The FDA gave it its approval in August 2008.1-(3Hydroxypropyl)-5- [(2R)-2-({2 - [2 (2,2,2trifluoroethoxy) phenoxy] ethyl} amino) propyl] -2,3-dihydro-1H-indole-7-carboxamide is the chemical name for silodosin. Symptoms of benign prostatic hyperplasia in the lower urinary tract are addressed with this medication **[1]**. Males are susceptible to benign prostatic hyperplasia (BHP), a chronic condition that induces age-dependent symptoms in the lower urinary tract. First-line medical therapy for BPH involves the use of effective medications called α1-adrenergic receptor blockers (ABs). Reducing the contraction of the prostate smooth muscle and improving selectivity for the dominant subtype of the α1A-adrenergic receptor in prostate tissues have been demonstrated by SLD **[2]**.

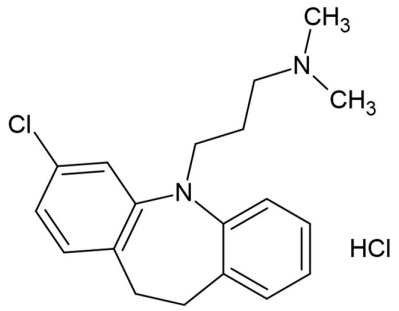

Figure 1: Structure of Silodosin hydrochloride

The objective of this work is to create stability indicating methods that are straightforward, specific, accurate, and exact so that the amount of SLD in pharmaceutical formulations and in pure form may be determined without interfering with the probable degradation products. The benefits of UV spectrophotometry are reproducibility, low cost, ease of application, and reduced time commitment. The current work successfully analyses SLD in pharmaceutical preparations and laboratory-prepared mixtures of SLD with its degradation products using a variety of UV spectrophotometric techniques **[3]**. The reported methods included UV–visible spectrophotometric methods **[4, 5]**, spectrofluorimetric methods **[6, 7]**, HPLC methods **[7, 8]**, UPLC method **[9]**, (LC–MS/MS) **[10]**, and HPTLC method **[11]**. In the presence of its degradation products, there were limited methods available for determining SLD **[7, 9]**. Therefore, the development of the UV spectrophotometric MVC for the determination of silodosin is the focus of the current technique. Applied analytical technique offers powerful, rapid, sensitivity, and low-cost quantitative analysis of investing admixtures under optimized conditions.

The following equations may be generated for each chosen wavelength when the absorbance of a sample(x) is measured at various wavelengths (λ), namely at 248, 80, 269, 84, and 86nm.

$A_{\lambda 259} = p \times C_X + k1$ …….. (1)

$A_{\lambda 264} = q \times C_X + k2$…… (2)

$A_{\lambda 269} = r \times C_X + k3$ …….. (3)

$A_{\lambda 274} = s \times C_X + k4$ …… (4)

$A_{\lambda 279} = t \times C_X + k5$ …… (5)

Whereas,

- A_λ= Absorbance of the sample;
- p, q, r, s, t, = Slope of the straight regression functions of a sample;
- k_1, k_2, k_3, k_4, k_5 = Intercept of the straight regression;
- C_X = Concentration of the sample

The five equations mentioned before can be arranged as follows:

$A_T = p \times C_X + q \times C_X + r \times C_X + s \times C_X + t \times C_X + K_T$…… (6)

The aforementioned equation may be reduced even more to

$A_T = C_X (p+q+r+s+t) + K_T$…… (7)

Whereas,

- A_T = Sum of the absorbances acquired
- K_T = Sum of intercepts of regression equation

To determine the amount of analyte X in a solution, use the formula.

$$Cx = \frac{A_T - K_T}{(p+Aq+r+s+\ )} \quad \text{……(8)}$$

Strategies for evaluating greenness

According to "The Globally Harmonized System of Classification and Labeling of Chemicals (GHS)," the eco analytical scale **[12]** assigns penalty points depending on the number of signal words coupled with the pictograms. Each reagent is considered in the analytical eco-scale method, together with its type, Quantity, waste, possible, energy depletion, and occupational exposure. Penalty points are subtracted from the original score of 100 points.

Analytical eco-scale = 100 - total penalty points …… (9)

An additional illustration is the Green Analytical Procedure Index (GAPI) **[13]**, which has five pentagons and has a unique color scheme. The colour coding of the pictogram for each phase of an analytical procedure uses three levels of assessment Green, red, and yellow are the three colors that GAPI employs to represent the respective degrees of minimal, moderate, and significant environmental damage linked to the analytical technique. In 2018, J. Potka-Wasylka provided succinct, accurate, informative reporting on GAPI(20). AGREE metrics, **[14]** to quantify the greenness profile during the second evaluation procedure, specialized software is used. The software generates a circular clockwise figure with oriented with numerals on the edges, ranging from 1 to 12. The 12 green analytical chemistry

philosophies are depicted in these imagesOn a scale of 1 to 12, the outputs of these 12 principles are rated from 0 to 1 based on the data provided and their weight. On this overall scale, red denotes a value of zero, dark green a value of one or a number which is near one, and yellow a value in between. The twelve principles are combined with the essence to produce a score that determines the extent of greenness.

MATERIALS AND METHODS:

Chemicals and solvents employed:

- Ethanol
- SILODAL 8® TABLETS – (Label claim – 8 mg), made by Sun Pharma Laboratories

  Pvt. Ltd., The medication formulations that were marketed were procured on a regional basis.

Solubility:

- Readily dissolving in ethanol, methanol, and dimethyl formamide.

Instrumentation:

- Electronic balance (SHIMADZU AY-220H).
- Soniclean sonicator (model 160T, Thebarton-Australia).
- UV-Vis double beam Spectrophotometer (Lab India UV-3092).

METHOD DEVELOPMENT:

Selection of solvent:

During the analysis, ethanol was used as the solvent to dissolve the drug, and it was shown to be highly soluble.

Preparation of the standard solution

100 mg of the drug component were diluted in 100 mL of ethanol to create the silodosin standard stock solution. We adjusted the concentration of this solution (7-13g/mL) and utilized it for further investigation.

Preparation of sample solution:

Accurately quantifying 40mg of silodosin, it was subsequently placed into a 100ml volumetric flask. After adding 100ml of ethanol, the mixture was sonicated for 10 minutes. The resulting volume was 100 ml (400 µg mL^{-1}). The resulting mixture was then diluted with the solvent to generate concentrations between 7 and 13 µgmL^{-1}

λ_{max} determination and selection of wavelength for multivariate calibration:

The ethanol solution, which did not contain any substance, was analyzed by comparing it to the standard solutions of silodosin. This analysis was done by scanning the solutions over a range of wavelengths from 200 to 400 nm.

Ethanol has a maximal absorption at 269 nm.

The wavelength of the MVC method was accordingly located between these absorption peaks, at 259, 264, 269, 274, 279nm.

METHOD VALIDATION

The proposed method's linearity, accuracy, and precision were verified in accordance with ICH recommendations **[15]**.

Linearity

After diluting the stock solution with ethanol, quantities of silodosin ranging from 7 to 13 µg mL-1 were used to examine its linearity and spectrum area. The absorbance of linear solutions at the specified wavelength was measured and studied for the MVC method.

Limit of Quantification and Detection

The Limits of Detection (LOD) and Limits of Quantification (LOQ) for silodosin were determined using the slope of the calibration curve and the standard deviation of responses for a specific wavelength. The following calculations were employed.

$$\text{LOD} = \frac{3.3 \text{ x standard deviation}}{\text{Slope}} \quad \text{..... (10)}$$

$$\text{LOQ} = \frac{10 \text{ x standard deviation}}{\text{Slope}} \quad \text{..... (11)}$$

Precision

The repeatability of the precision was evaluated using intraday and interday precision. A standard silodosin solution with a concentration of 10 µgmL^{-1} was used to evaluate various degrees of accuracy. Six solutions were examined at five different wavelengths to assess repeatability. The absorbance of prepared solutions was tested three times at different time intervals on the same day to assess intervariation. Three more days of using the absorbance were employed to account for intravariation.

Accuracy

The silodosin methodology's precision was assessed at 80, 100, and 120 percent of the concentrations of the previously examined sample solutions, while the recovery values percentages were estimated.

Assay

Weigh and grind Ten tablets. Add 8 ml of ethanol to a quantity of tablet powder that is approximately 50 mg of silodosin, and sonicate for 10 minutes. Weigh the powder accurately. Add an adequate amount of ethanol to achieve a total of 50 milliliters. To achieve a concentration of 10 µg mL-1 of silodosin, the solution obtained above is filtered and diluted with ethanol. The concentration of silodosin is determined by measuring the absorbance of the resulting solution at 269 nm.

RESULTS AND DISCUSSION

Silodosin standard solution was originally scanned between 200 and 400 nm. The peak wavelength of the silodosin spectrum is 269 nm. The UV spectrum of silodosin standards and samples was measured using ethanol as a reference and a wavelength of 269nm for MVC. The standard silodosin spectrum at a dose of 10 µg mL-1is depicted in **Figure 2**.

Linearity

The linearity values for the devised technique for silodosin were found within the concentration range of 70 to 130 percent for 10g mL-m1 (7 to 13 µgmL-1), as required by the ICH Q2 R1 guidelines. In

Figure 3, the linearity spectrum of silodosin is depicted. By estimating the absorbance of reference solutions that had been diluted at five distinct wavelengths (259, 264, 269, 274, 279), the calibration curve was produced. The results that were observed are presented in tabular format in **Table 1**. It was determined that all of the standard curves were linear within the selected concentration range. The calibration graphs and regression analysis are illustrated in **Figure 4-8 and Table 2**, respectively.

Limit of Detection and Limit of Quantification

The linearity slope was employed for calculating the LOD and LOQ for silodosin, and many sample studies have supported this method. The average of all the absorbance was used to compute the LOD for silodosin, which was found to be 0.1597 µg mL^{-1}. The limit of quantification (LOQ) for silodosin was determined to be 0.1595 µg mL-1 by averaging the absorbances.

Precision

The system precision spectrum for silodosin is displayed in **Figure 9**. **Figure 10** depicts the silodosin interday precision spectra. **Figure 11** for silodosin depicts the intraday precision spectra. For silodosin, the percentage RSD of the system's intraday and interday precision was calculated. It was discovered to be less than 2%, demonstrating the precision of the approach method. Comparing the results acquired from other accuracy approaches, the suggested method shows good precision.

Accuracy

The overlay spectra for silodosin was displayed in **Figure 12**, were verified for accuracy at 80, 100, and 120%. The silodosin values are displayed in **Table 3**, and it was determined that the results were within acceptable bounds.

Analyze of commercialized formulation:

By employing the proposed spectrophotometric methodology, the formulation of the tablet was assessed for the quantity of silodosin. The UV absorption spectra of a commercially available medicine were analyzed through a sequence of three experiments. Throughout the extraction and filtration process, the pharmaceutical formulation demonstrated consistently high and exceptional analytical recovery values..The **Table 4** presents the findings.

Evaluation of Greenness Profile

The outcomes of the suggested approaches' greenness profile were assessed. Analytical scale findings are displayed in **Table 6**, whereas agree and GAPI metrics data are provided in **Figures 14 and 15.**

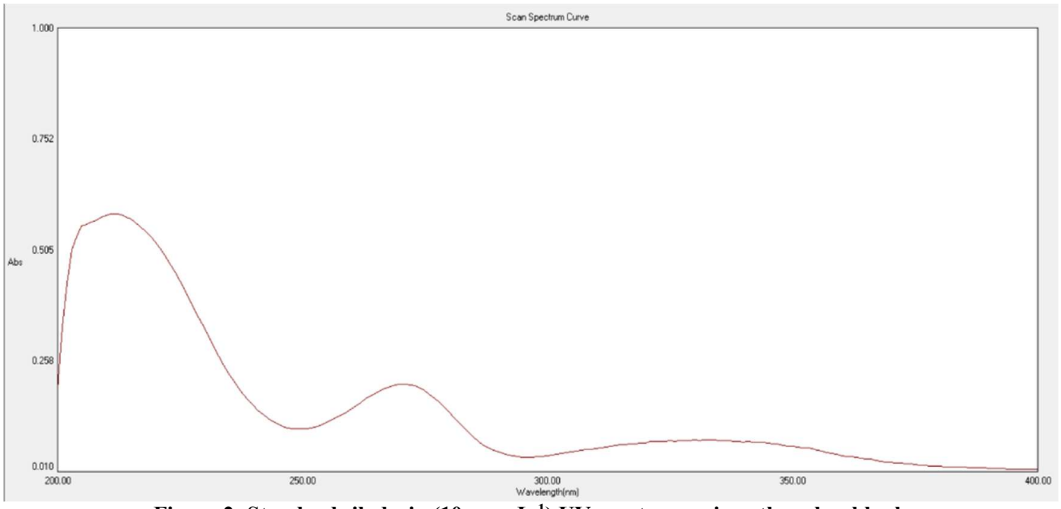

Figure 2: Standard silodosin (10 µg mL^{-1}) UV spectrum using ethanol as blank

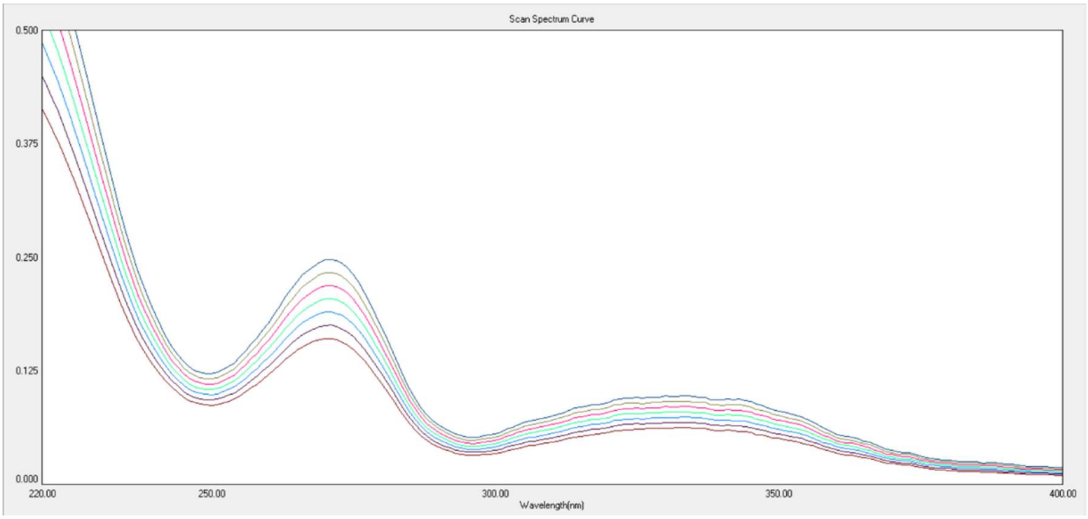

Figure 3: The spectrum of linearity for silodosin (7-13 µg mL-1) was determined by utilizing ethanol as a baseline

Table 1: UV calibration data at five designated wavelengths in a multivariate pattern

Concentration (µgmL^{-1})	259nm	264nm	269nm	274nm	279nm
7	0.124	0.142	0.153	0.142	0.116
8	0.141	0.162	0.176	0.164	0.132
9	0.157	0.183	0.199	0.186	0.148
10	0.174	0.203	0.220	0.207	0.163
11	0.190	0.224	0.244	0.231	0.179
12	0.207	0.244	0.266	0.254	0.195
13	0.223	0.265	0.289	0.276	0.211

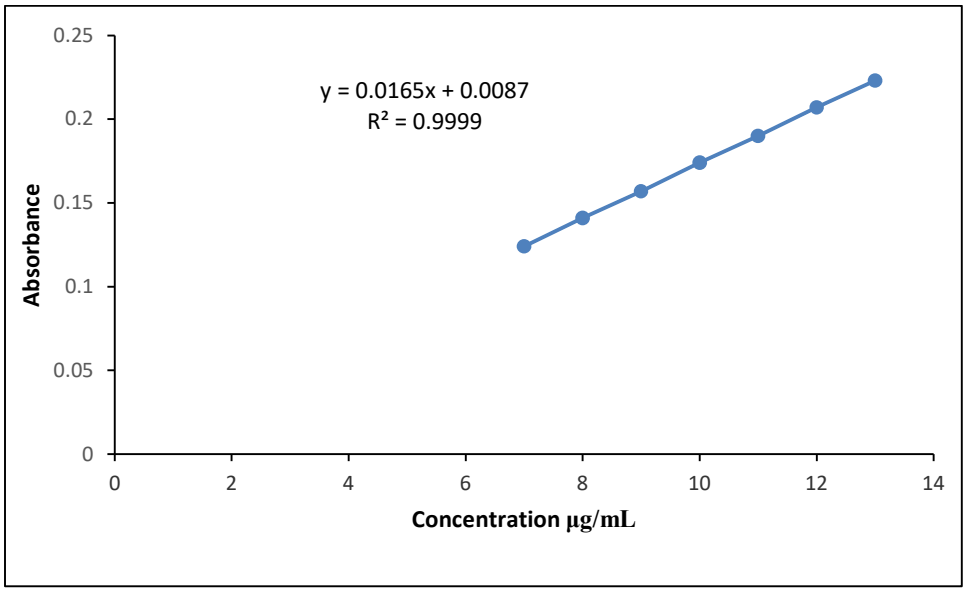

Figure 4: Calibration curve at 259nm

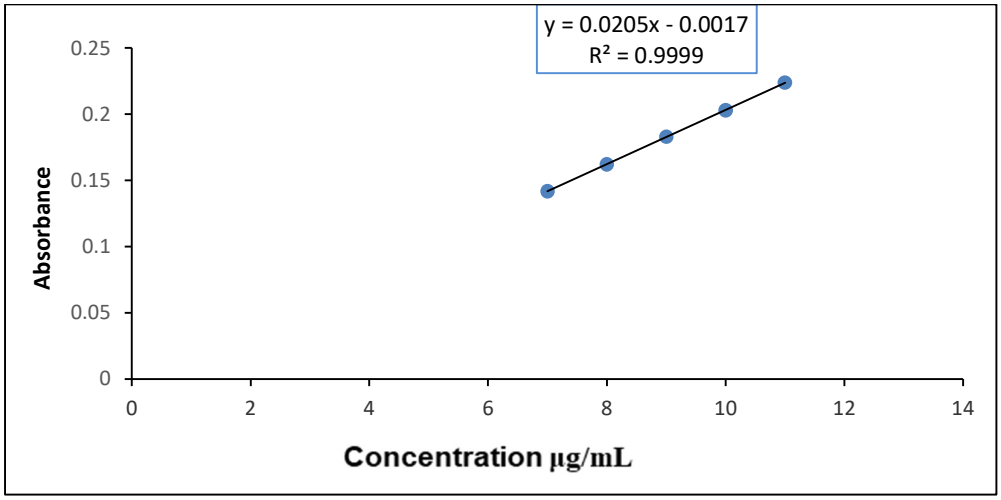

Figure 5: Calibration curve at 264nm

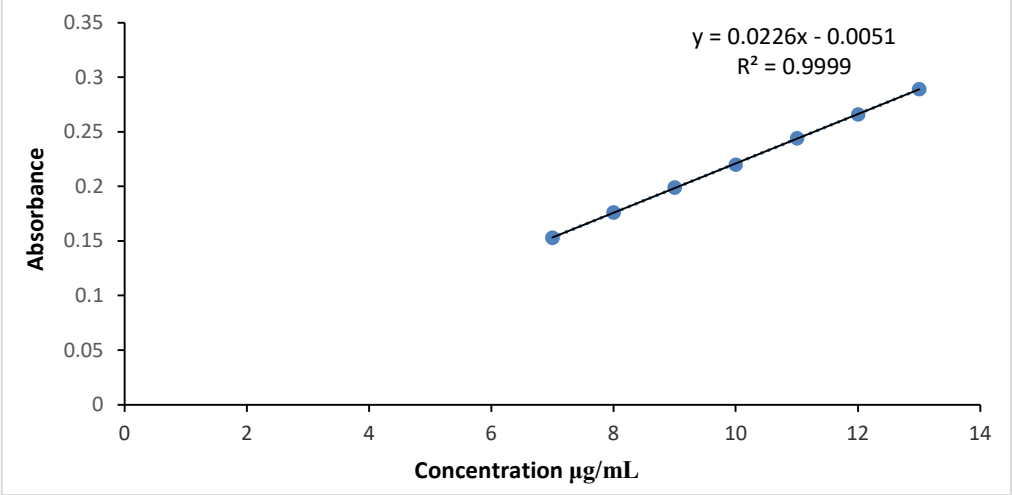

Figure 6: Calibration curve at 269nm

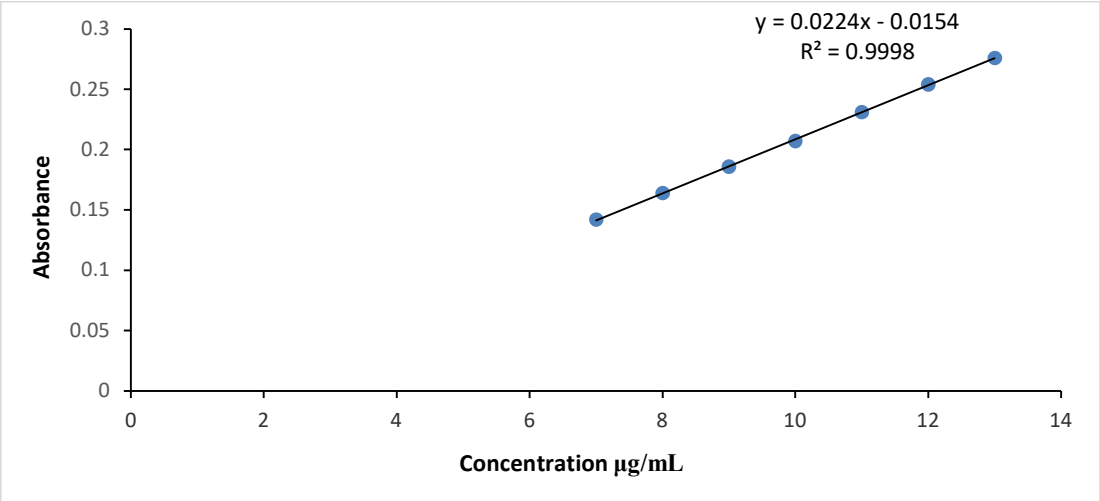

Figure 7: Calibration curve at 274nm

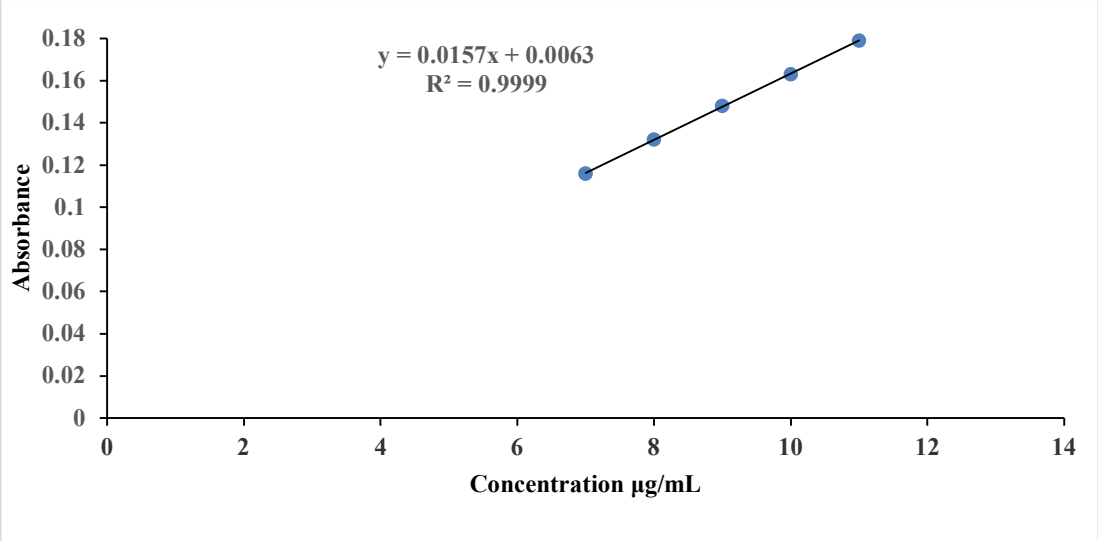

Figure 8: Calibration curve at 279nm

Table 2: Statistics parameters at the chosen wavelengths are shown by linearity data.

Wavelength(nm)	Regression equation	Slope	Intercept	R^2	LOD (µg mL^{-1})	LOQ (µg mL^{-1})
259	y = 0.0165x + 0.0087	0.0165	0.0087	0.9999	0.0585	0.1774
264	y = 0.0205x - 0.0017	0.0205	-0.0017	0.9999	0.4712	0.1428
269	y = 0.0226x - 0.0051	0.0226	-0.0051	0.9999	0.0846	0.2563
274	y = 0.0224x - 0.0154	0.0224	- 0.0154	0.9998	0.1230	0.0349
279	y = 0.0157x + 0.0063	0.0157	0.0063	0.9999	0.0615	0.1864

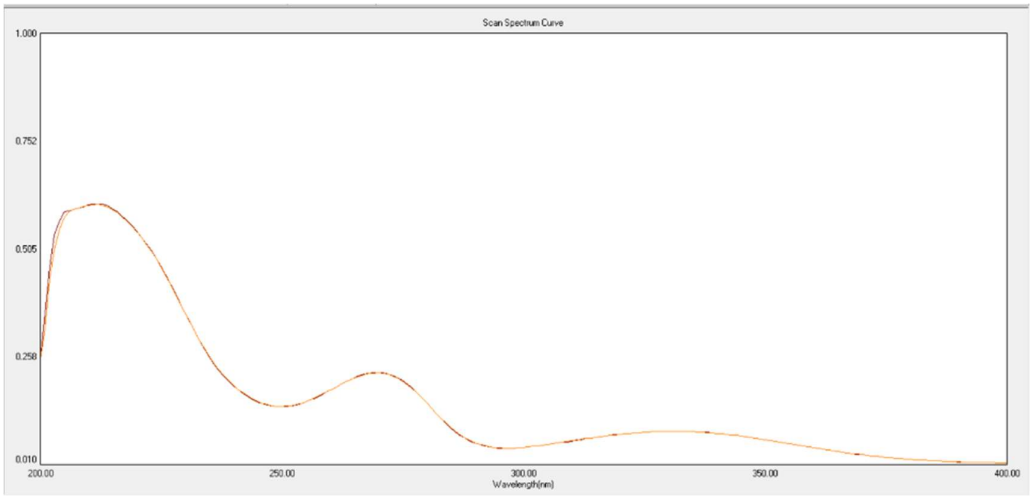

Figure 9: Spectra of silodosin with system precision overlay silodosin

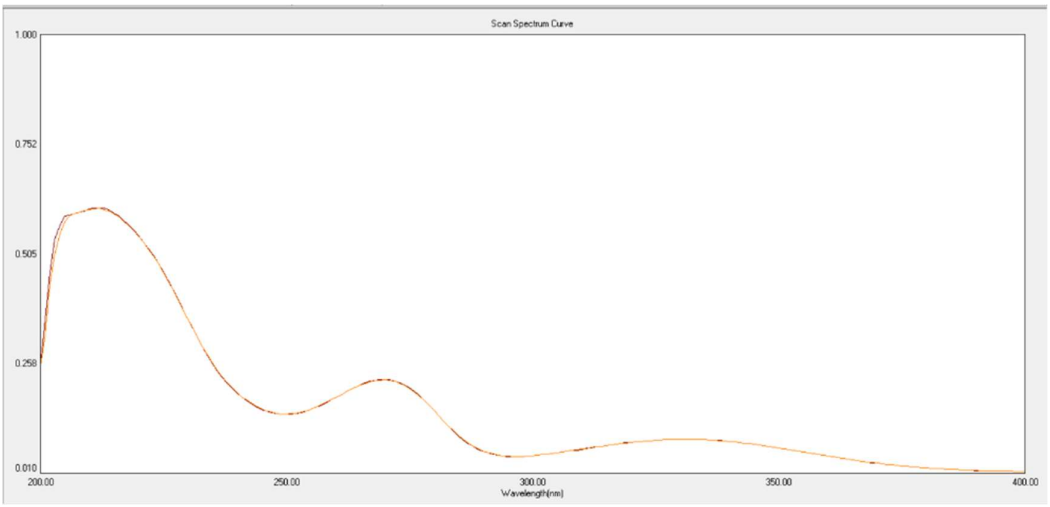

Figure 10: Interday precision overlay spectra of silodosin

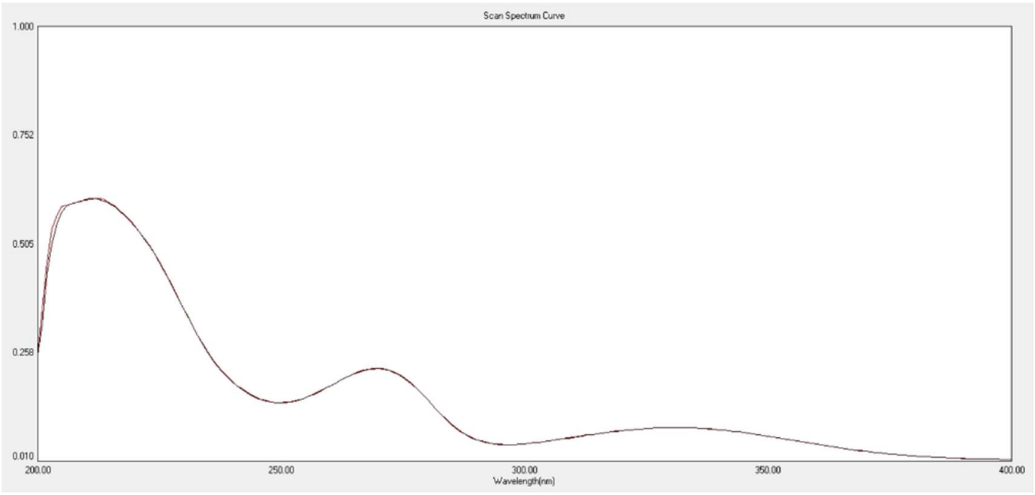

Figure 11: Intraday precision overlay spectra of Silodosin

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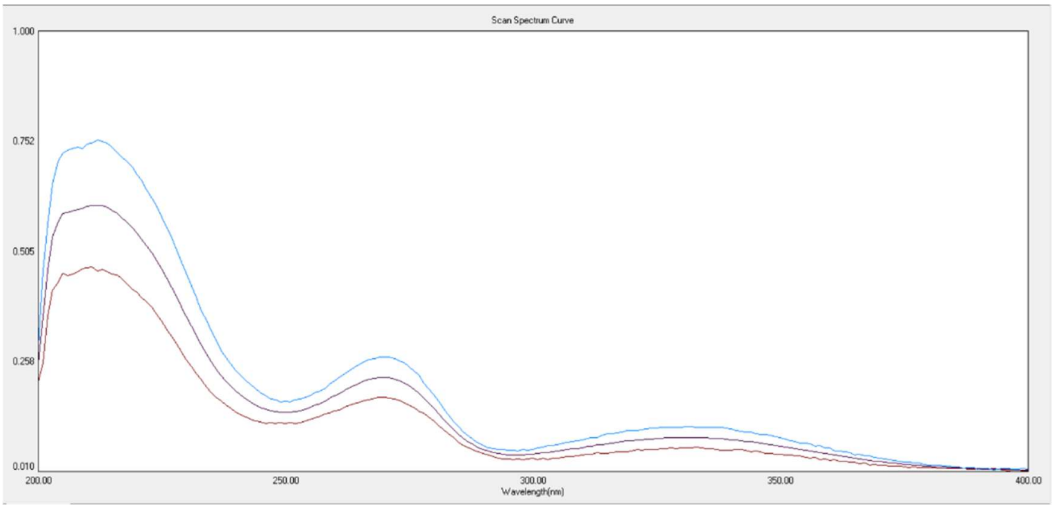

Figure 12: The overlay spectra of silodosin's accuracy in raising by 80, 100, and 120 percent

Table 3: Recovery Studies

wavelength (nm)	Amount present (µg mL^{-1})	Amount added (µg mL^{-1})	Absorbance	Amount recovered (µg mL^{-1})	% Recovery
259	5	3	0.141	7.92	99.00
		5	0.174	9.96	99.60
		7	0.207	11.98	99.83
264	5	3	0.162	7.94	99.25
		5	0.203	9.98	99.80
		7	0.244	11.96	99.67
269	5	3	0.176	7.96	99.50
		5	0.22	9.95	99.50
		7	0.266	11.97	99.75
274	5	3	0.164	7.99	99.88
		5	0.207	9.96	99.60
		7	0.254	11.91	99.25
279	5	3	0.132	7.91	98.88
		5	0.163	9.89	98.90
		7	0.195	11.92	99.33

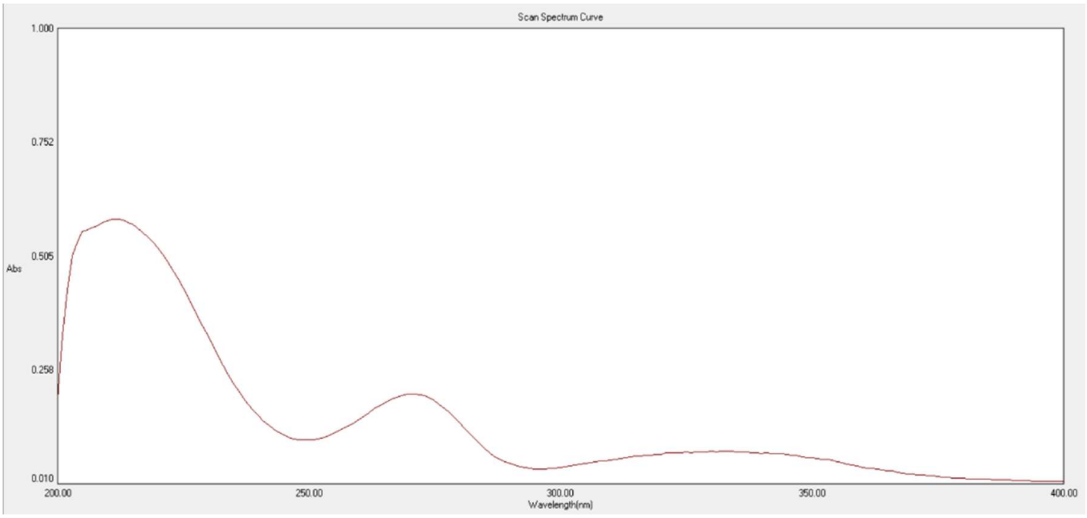

Figure 13: standard silodosin (10µg mL^{-1}) UV spectrum using ethanol as a blank

Table 4: Assay of SILODOSIN

Label claim (mg)	Amount estimated (mg)	% Assay
8	7.98	99.75
8	8.02	100.25
8	7.97	99.63
Average	7.99	99.88
SD		0.3307
% RSD		0.3311

Table 6: The proposed method's summary of the Eco scale penalty points

Description	Penalty points	Total Penalty Points	Score
Occupational hazard	0	4	96
Waste	0		
Ethanol	4		
Instrument	0		

Figure 15: Agree with the metrics output of the proposed method

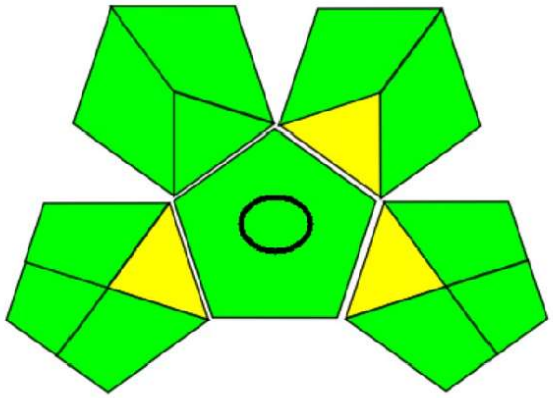

Figure 14: The proposed method's GAPI Pictogram

CONCLUSION

The recently developed spectrophotometric method for measuring silodosin was validated by evaluating a number of validation criteria, and it was discovered to be within allowable ranges in compliance with ICH guidelines It was demonstrated that the measurement of silodosin in its tablet formulation was sensitive, accurate, precise, and reproducible using the proposed method. The proposed methodology is more precise than the current UV spectrophotometric approaches and incorporates a method with straightforward mathematical components, we strongly recommend utilising it for routine research on silodosin in pharmaceutical formulations.

STATEMENT OF ETHICS

There are no animal or human participants used in this study's trials.

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DISPUTE OF INTEREST

There are no financial interests that could be at odds with this content.

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REFERENCES

[1] Aneesh T.P, A.Rajasekaran Method Development And Validation For The Estimation Of Sildosin In Bulk And Pharmaceutical Dosage Forms Using Uv-Vis Spectrophotometry Asian Journal of Pharmaceutical and Clinical Research

[2] Form Mehmet Emrah Yaman, Tugrul Cagri Akman Optimization of HPLC-FLD Conditions Using Analytical Quality by Design Approach for Quantification of Silodosin in Pharmaceutical Dosage Journal of Science and Technology DOI: 10.18185/erzifbed.955967

[3] Shereen A. Boltia, Mohammed Abdelkawy, Taghreed A. Mohammedc, Nahla N. Mostafa Validated stability-indicating spectrophotometric methods for the determination of Silodosin in the presence of its degradation products, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 202 (2018) 131–145 https://doi.org/10.1016/j.saa.2018.04.046

[4] K.K. Jahan, S.M. Malipatil, Development and validation of new spectrophotometric methods for the quantitative estimation of silodosin in bulk drug and pharmaceutical

formulations, Int. J. Pharm. Res. Anal. 4 (2014) 65–69.

[5] C.H. Mounika, N. Umadevi, I. Sudheerbabu, New visible spectrophotometric methods for the estimation of silodosin in pharmaceutical formulations, Int. J. Res. Pharm. Chem. 3 (2013) 595–597.

[6] S.J.R. PrachiBhamre, Spectrofluorimetric method for the determination of silodosin in bulk and pharmaceutical dosage form, Indo Am. J. Pharm. Res. 4 (2014) 5106–5110.

[7] E. Er, N. Erk, An effective and sensitive stability- indicating chromatographic approach based on HPLC for silodosin assay, J. Anal. Sci. Technol. (2016)https://doi. org/10.1186/s40543-016-0100-y.

[8] V.M. Goud, A.S. Rao, S.P. Ranjan, S.D. Shalini, S. Sowmya, B. Bhoga, Method development and validation of RP-HPLC method for assay of silodosin in pharmaceutical dos age form, Int. J. Pharm. Sci. 3 (2013) 194–196.

[9] J.V. Shaik, S. Saladi, S.S. Sait, Development of stability-indicating UHPLC method for the quantitative determination of silodosin and its related substances, J. Chromatogr. Sci. 52 (2014) 646–653, https://doi.org/10.1093/chromsci/bmt094.

[10] X. Zhao, Y. Liu, J. Xu, D. Zhang, Y. Zhou, J. Gu, Y. Cui, Determination of silodosin in human plasma by liquid chromatography-tandem mass spectrometry, J. Chromatogr. B Anal. Technol. Biomed. Life Sci. 877 (2009) 3724–3728, https://doi. org/10.1016/j.jchromb.2009.08.025.

[11] P.S. Sayana, R.S. Iyer, A. Shibi, S. Harischandran, Development and validation of HPTLC method for quantification of silodosin in bulk and pharmaceutical dosage form, Pharm. Innov. 1 (2012) 60–65http://www.thepharmajournal.com/ vol1Issue10/11.1.html.

[12] Gałuszka A, Migaszewski ZM, Konieczka P, Namieśnik J. Analytical Eco-Scale for assessing the greenness of analytical procedures. TrAC Trends Anal Chem [Internet]. 2012 Jul;37:61–72. Available from: https://linkinghub.elsevier.com/retrieve/pii/S016599361200158

[13] Płotka-Wasylka J. A new tool for the evaluation of the analytical procedure: Green Analytical Procedure Index. Talanta [Internet]. 2018 May;181:204–9.

Available from: https://linkinghub.elsevier.com/retrieve/pii/S0039914018300195

[14] Pena-Pereira F, Wojnowski W, Tobiszewski M. AGREE—Analytical GREEnness Metric Approach and Software. Anal Chem [Internet]. 2020 Jul 21;92(14):10076–82. Available from: https://pubs.acs.org/doi/10.1021/acs.analchem.0c01887

[15] Borman P, Elder D. Q2(R1) Validation of Analytical Procedures. In: ICH Quality Guidelines [Internet]. Hoboken, NJ, USA: John Wiley & Sons, Inc.; 2017. p. 127–66. Available from: https://onlinelibrary.wiley.com/doi/10.1002/9781118971147.ch5