



UV Spectrophotometric Method for the Estimation of Tadalafil in Bulk and Tablet Dosage form

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Abstract: A simple, sensitive, precise and highly accurate UV spectrophotometric method has been developed for the determination of tadalafil in bulk and tablet dosage form. Solution of tadalafil in methanol shows maximum absorbance at 284 nm. Beer's law was obeyed in the concentration range of 2-20 mcg mL⁻¹ with 1.65x10⁴ mol⁻¹ cm⁻¹, the slope, intercept, correlation coefficient, detection and quantitation limits were also calculated. The proposed method has been applied successfully for the analysis of the drug in pure and in its tablets dosage forms. Result of percentage recovery and placebo interference shows that the method was not affected by the presence of common excipients. The method was validated by determining its sensitivity, accuracy and precision which proves suitability of the developed method for the routine estimation of tadalafil in bulk and solid dosage form.

Keywords: UV Spectrophotometry, Tadalafil, Beer's law, Tablet dosage form.

Introduction

Tadalafil, chemically pyrazino [1',2':1,6] pyrido [3,4-b] indole-1,4-dione,6-(1,3-benzodioxol-5-yl)-2,3,6,7,12,12 *a*-hexahydro-2-methyl-, (6R,12aR)-(Figure 1), is an impotence agent. It is indicated for the treatment of erectile dysfunction^{1,2}. It is a selective inhibitor of cyclic guanosine monophosphate (cGMP)-specific phosphodiesterase type 5 (PDE5)³. In previous studies, only one assay has been reported for the determination of tadalafil in human serum and urine by LC-MS-MS⁴. Tadalafil has also been quantified in pharmaceutical preparations, human serum and biological fluids by HPLC⁵ with UV detection. Determination of tadalafil by densitometric (TLC)⁶ and colorimetric⁷ methods has also been reported. Although the ultraviolet spectrophotometric method is the instrumental method of choice commonly used in industrial laboratories because of their simplicity, selectivity and sensitivity. As of our knowledge no report has been mentioned in the literature for the determination of tadalafil by

UV method. The aim of the present work was to develop simple, rapid, accurate and sensitive UV spectrophotometric method for the determination of tadalafil in bulk and tablet formulation. UV analysis of tadalafil was performed in methanol. The spectrum was recorded from 200 nm to 400 nm. The quantitative analysis was carried out at 284 nm. The method was validated and applied for the determination of tadalafil in tablet dosage form.

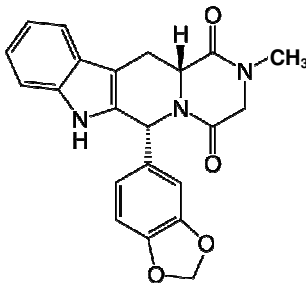


Figure 1. Chemical structure of tadalafil.

Experimental

Pharmaceutical grade of tadalafil was procured from Orchid chemicals and Pharmaceuticals Ltd., India. All the chemicals were of analytical reagent grade of Merck (Germany) unless otherwise specified. Methanol was used to prepare all solutions. Freshly prepared solutions were always employed. Different brands of tablets of tadalafil were supplied from local pharmacy.

Instrumentation

The spectrophotometric measurements were carried out using Elico UV/Visible double beam spectrophotometer SL-164 with 1 cm matched quartz cells.

Tadalafil stock solution

Standard stock solution was prepared by dissolving 50 mg of tadalafil in 50 mL of methanol to get concentration of 1000 $\mu\text{g/mL}$ solution. It was further diluted to get working standard solution of 100 $\mu\text{g/mL}$.

Method development

Aliquot of working standard solution was further diluted with methanol to get concentration of 10 $\mu\text{g mL}^{-1}$ and it was scanned between 200–400 nm which shows the maximum absorbance at 284 nm (Figure 2). The same λ_{max} was used for the further measurement of the drug.

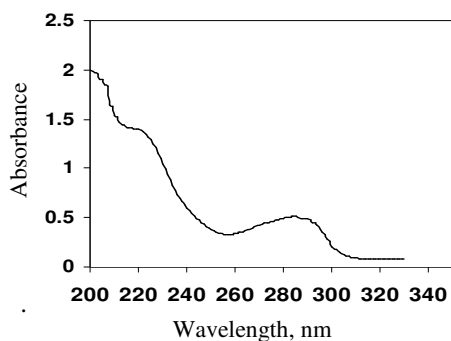


Figure 2. UV spectra of tadalafil.

Procedure for calibration curve

Aliquots of working standard solution were further diluted with methanol to get concentration of 2, 4, 8, 12, 16 and 20 $\mu\text{g mL}^{-1}$. Finally, the prepared standards were measured after standing for 5.0 min at λ_{max} as recorded in Table 1, in each case against a solvent methanol as blank. A calibration graph of the absorbance *versus* the concentration of the drug was plotted (Figure 3).

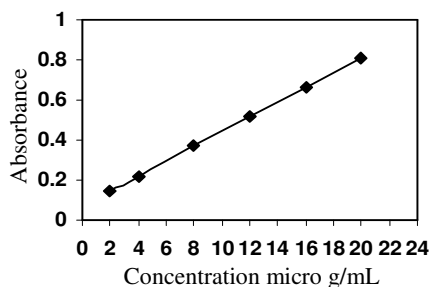


Figure 3. Standard plot of tadalafil.

Procedure for dosage forms

For analysis of commercial formulations, twenty tablets containing tadalafil were taken and powdered. Tablet powder equivalent to 50 mg of tadalafil was transferred to 50 mL volumetric flask and dissolved in methanol. Then the solution was sonicated for 15 min and filtered through Whatman filter paper No. 41 and it was further diluted to get the required concentration. The absorbance of the prepared sample solution was measured against methanol as a blank at 284 nm. A standard additions technique was also used to confirm the accuracy and precisions

Results and Discussion

The optical characteristics such as Beer's law limit, molar absorptivity, Sandell's sensitivity, correlation coefficient, slope and intercept, % Relative Standard Deviation (RSD), % range of error (0.05 and 0.01 confidence limits) were calculated and are summarized in Table 1. To ensure the reproducibility and accuracy of the method, recovery studies were carried out by adding a known quantity of pure drug with preanalyzed sample and contents were reanalyzed by the proposed method. From the amount of drug found, percentage recovery was calculated. The results of analysis and recovery studies are given in Table 2.

Table 1. Optical characteristics and precision of the proposed method.

Parameters	Results
λ_{max} , nm	284
Beer's law limit, $\mu\text{g/mL}$	2-20
Molar absorptivity, $\text{L mole}^{-1} \text{cm}^{-1}$	1.65×10^4
Sandell's sensitivity ($\mu\text{g cm}^{-2} / 0.001$ absorbance unit)	0.023
Regression equation ($Y = a + bC$)	
Slope (b)	0.0372
Intercept (a)	0.0075
Correlation coefficient (r)	0.0075
Relative standard deviation (%)*	0.28
% Range of error (Confidence limits)	
0.05 level	0.2341
0.01 level	0.3462

* Average of eight determinations.

Table 2. Assay of tadalafil in tablet dosage form.

Brand	Labeled amount mg/tablet	Estimated amount	Spike level, %	Amount of drug added,mg	Amount of drug found,mg	Percentage Recovery \pm RSD*
TADIL	10	9.98	80	4	3.98	99.5 \pm 0.44
			100	8	7.97	99.6 \pm 0.29
			120	10	10.02	100.2 \pm 0.37

*Mean of six determinations.

Conclusion

In this study a simple, rapid, sensitive, accurate and precise UV spectrophotometric method for the determination of tadalafil in bulk and pharmaceutical formulation has been developed and validated. It was found that the common excipients present in the formulation did not interfere with the proposed method and can be used for the routine quality control analysis of tadalafil in bulk as well as in tablet formulations.

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