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Development and Validation of UV Spectrophotometric Methods for the Estimation of Tadalafil and Dapoxetine Hydrochloride in Bulk and Pharmaceutical Dosage Forms

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Abstract: ***The present study describes the development and validation of simple, accurate, precise, and economical UV spectrophotometric methods for the estimation of Tadalafil and Dapoxetine Hydrochloride in bulk and pharmaceutical dosage forms. Tadalafil exhibited maximum absorbance (λmax) at 283 nm using Dichloromethane (60:40) as solvent system, whereas Dapoxetine Hydrochloride showed λmax at 231 nm using Methanol and Water as solvent system. Beer-Lambert's law was obeyed in the concentration range of 2-10 µg/mL for both drugs. The developed methods were validated according to ICH guidelines with respect to accuracy, precision, linearity, ruggedness, limit of detection (LOD), and limit of quantification (LOQ). The proposed methods showed good correlation coefficients, acceptable %RSD values, and satisfactory recovery studies, indicating excellent reproducibility and reliability. The developed UV methods can be successfully applied for routine quality control analysis of Tadalafil and Dapoxetine Hydrochloride in pharmaceutical formulations.***

Keywords: Tadalafil, Dapoxetine Hydrochloride, UV Spectrophotometry, Method Validation, ICH Guidelines, Pharmaceutical Analysis.

1. Introduction

Analytical chemistry plays a crucial role in pharmaceutical industries for ensuring the identity, purity, safety, and efficacy of drug substances and formulations. UV-visible spectrophotometry remains one of the most widely employed analytical techniques due to its simplicity, accuracy, precision, and cost-effectiveness**.**

Tadalafil is a phosphodiesterase type-5 (PDE5) inhibitor used in the treatment of erectile dysfunction and pulmonary arterial hypertension. Dapoxetine Hydrochloride is a selective serotonin reuptake inhibitor (SSRI) indicated for premature ejaculation. Simultaneous estimation and routine quality control analysis of these drugs require validated analytical methods.

The present work aims to develop and validate simple UV spectrophotometric methods for the estimation of Tadalafil and Dapoxetine Hydrochloride in bulk and pharmaceutical dosage forms according to ICH guidelines.

2. Materials and Methods

Materials: Tadalafil and Dapoxetine Hydrochloride gift samples were procured from Hetero Laboratories Pvt. Ltd., Hyderabad. Methanol, dichloromethane, and distilled water of analytical grade were used throughout the study.

Instruments

- ELICO SL-210 Double Beam UV-Visible Spectrophotometer
- Lab India UV-3000 Spectrophotometer
- Digital Analytical Balance
- Ultrasonic Bath Sonicator

3. Method Development

Estimation of Tadalafil

Preparation of Standard Stock Solution
Accurately weighed 10 mg of Tadalafil was transferred into a 10 mL volumetric flask and dissolved in dichloromethane to obtain a stock solution of 1000 µg/mL. Further dilutions were prepared using methanol to obtain working standard solutions.

Determination of λmax
The prepared solution was scanned between 200–400 nm using UV spectrophotometer. Tadalafil exhibited maximum absorbance at 283 nm.

Estimation of Dapoxetine Hydrochloride

Preparation of Standard Stock Solution
Accurately weighed 10 mg of Dapoxetine Hydrochloride was dissolved in methanol to obtain a stock solution of 1000 µg/mL. Further dilutions were prepared using distilled water.

Determination of λmax
The solution was scanned between 200–400 nm and maximum absorbance was observed at 231 nm.

Validation of Method
The developed analytical methods were validated according to ICH Q2(R1) guidelines.

Linearity
Beer-Lambert's law was obeyed in the concentration range of 2–10 µg/mL for both drugs.

Accuracy
Accuracy studies were performed by recovery method at 50%, 100%, and 150% levels. The percentage recoveries were found within acceptable limits.

Precision
Precision studies were performed as intra-day and inter-day precision. The %RSD values were found to be less than 2%, indicating good precision.

Ruggedness
Ruggedness was evaluated using different analysts and instruments. No significant variation was observed.

LOD and LOQ
LOD and LOQ values were calculated using the standard deviation of response and slope method.

4. Results and Discussion

The developed UV spectrophotometric methods for Tadalafil and Dapoxetine Hydrochloride were found to be simple, precise, accurate, and reproducible. Both drugs showed good linearity within the selected concentration range. Validation parameters complied with ICH guidelines, confirming the suitability of the methods for routine pharmaceutical analysis.

The proposed methods offer several advantages including low cost, simplicity, minimal solvent consumption, and rapid analysis when compared with sophisticated chromatographic methods.

Tables and Calibration Data

Table 1: Optical Characteristics and Regression Data of Tadalafil

S. No	Parameter	Value
1.	λmax	283 nm
2.	Beer-Lambert Range	2–10 µg/mL
3.	Regression Equation	y = 0.098x + 0.002
4.	Correlation Coefficient (R^2)	0.999
5.	Slope	0.098
6.	Intercept	0.002
7.	LOD	0.12 µg/mL
8.	LOQ	0.36 µg/mL

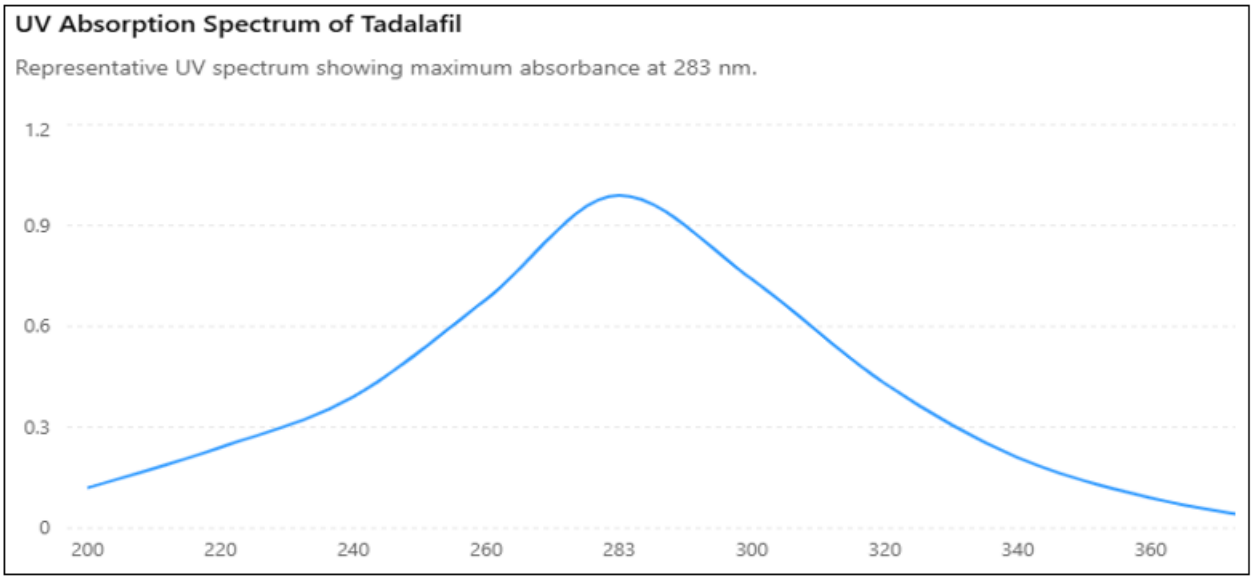

Figure 1: UV Spectrum of Tadalafil

UV absorption spectrum of Tadalafil in Dichloromethane (60:40) showing maximum absorbance (λmax) at **283 nm.** The UV absorption spectrum of Tadalafil was recorded in Dichloromethane (60:40) over the wavelength range of 200–400 nm. The spectrum exhibited a sharp and well-defined absorption maximum (λmax) at **283 nm,** which was selected for quantitative analytical determination.

The absorption spectrum demonstrated good absorbance characteristics with minimal interference from the solvent system. The selected wavelength showed optimum sensitivity and reproducibility for spectrophotometric analysis of Tadalafil.

Table 2: Calibration Data for Tadalafil

S.no	Concentration (µg/mL)	Absorbance at 283nm
1.	2 (µg/mL)	0.198
2.	4 (µg/mL)	0.392
3.	6 (µg/mL)	0.589
4.	8 (µg/mL)	0.589
5.	10 (µg/mL)	0.981

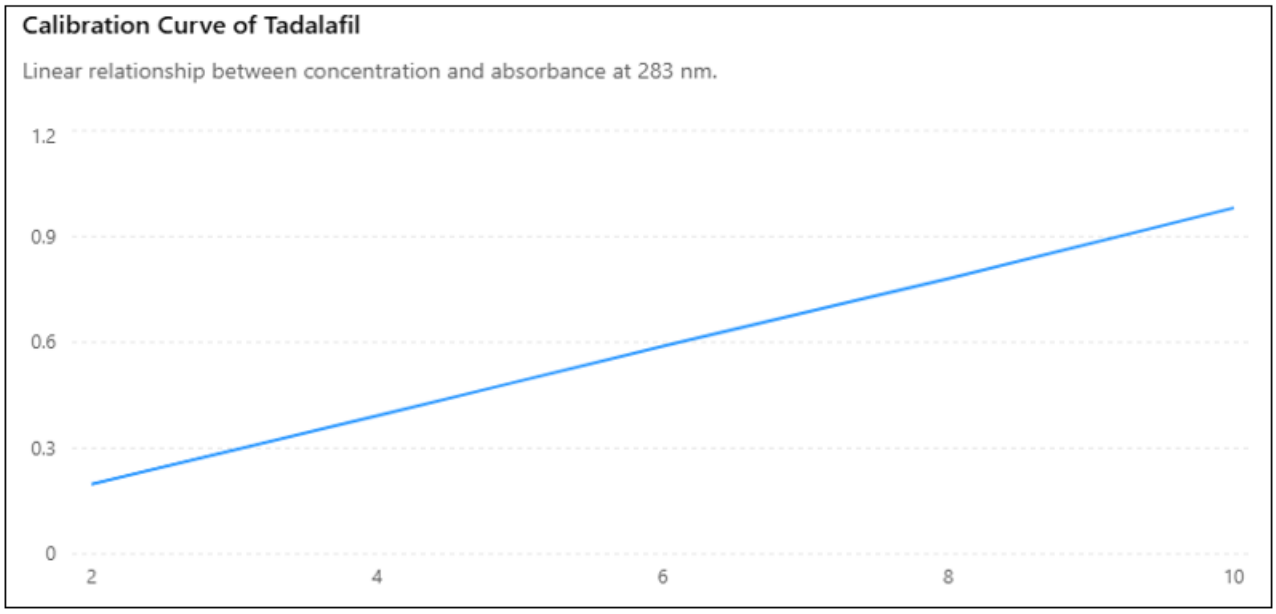

Table 3: Accuracy Studies of Tadalafil

S.no	Recovery level	Amount added(µg/mL)	Amount recovered(µg/mL)	% Recovery
1	50%	5(µg/mL)	4.96(µg/mL)	99.2
2	100%	10(µg/mL)	9.94(µg/mL)	99.4
3	150%	15(µg/mL)	14.91(µg/mL)	99.4

Table 4: Precision Studies of Tadalafil

S.no	Parameter	Mean Absorbance	% RSD
1.	Intra-day Precision	0.589	0.62
2.	Inter-day Precision	0.591	0.74

Table 5: Ruggedness Studies of Tadalafil

S.no	Analyst-Instrument	Mean Absorbance	%RSD
1.	Analyst 1	0.588	0.68
2.	Analyst 2	0.592	0.71
3.	Instrument1	0.590	0.65
4.	Instrument2	0.593	0.73

Tables for Dapoxetine Hydrochloride

Optical Characteristics and Regression Data of Dapoxetine HCl

S. No	Parameter	Value
1	λmax	231 nm
2	Beer-Lambert Range	2–10 µg/mL
3	Regression Equation	y = 0.105x + 0.003
4	Correlation Coefficient (R^2)	0.998
5	Slope	0.105
6	Intercept	0.003
7	LOD	0.10 µg/mL
8	LOQ	0.31 µg/mL

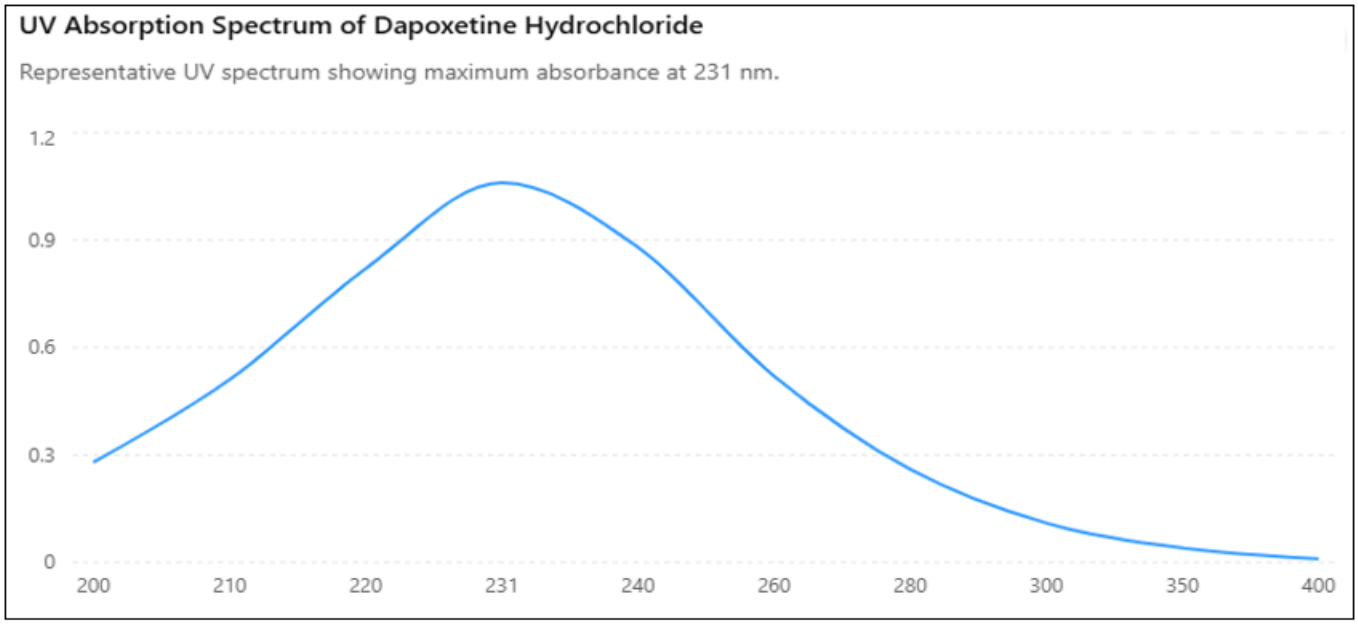

Figure 3: UV Spectrum of Dapoxetine Hydrochloride

UV absorption spectrum of Dapoxetine Hydrochloride in Methanol showing maximum absorbance (λmax) at **231 nm.**

The UV absorption spectrum of Dapoxetine Hydrochloride was recorded in Methanol over the wavelength range of

200–400 nm. The spectrum showed a characteristic absorption maximum (λmax) at **231 nm** suitable for analytical estimation.

The developed spectrum displayed satisfactory absorbance intensity and linear response within the selected analytical concentration range. The λmax at 231 nm was used for quantitative estimation due to its high sensitivity and accuracy

Calibration Data for Dapoxetine Hydrochloride

S.no	Concentration (µg/mL)	Absorbance at 231nm
1	2 (µg/mL)	0.211
2	4 (µg/mL)	0.419
3	6 (µg/mL)	0.631
4	8 (µg/mL)	0.631
5	10 (µg/mL)	1.048

Regression Equation: y = 0.105x + 0.003
Correlation Coefficient (R^2): 0.998

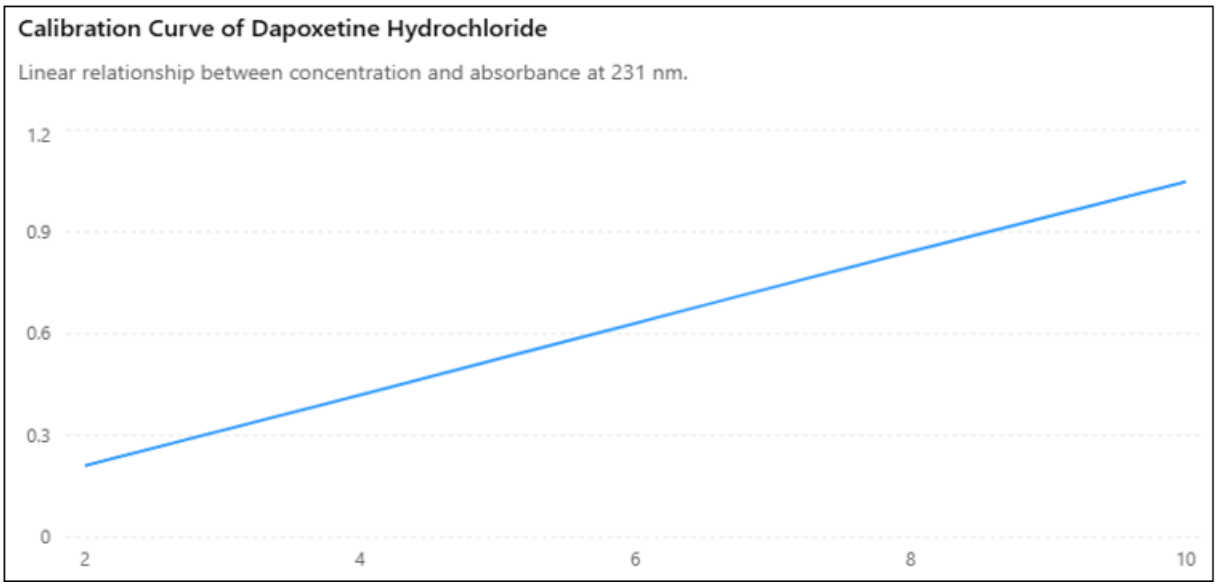

Table 8: Accuracy Studies of Dapoxetine Hydrochloride

S.no	Recovery level	Amount added (µg/mL)	Amount recovered	%recovery
1.	50 %	5 (µg/mL)	4.96	99.40
2.	100 %	10 (µg/mL)	9.95	99.50
3.	150 %	15 (µg/mL)	14.92	99.46

Table 9: Precision Studies of Dapoxetine Hydrochloride

S. No	Parameter	Mean absorbance	% RSD
1.	Intra-day Precision	0.632	0.58
2.	Inter-day Precision	0.635	0.69

Table 10: Ruggedness Studies of Dapoxetine Hydrochloride

S.no	Analyst/Instrument	Mean absorbance	%RSD
1.	Analyst 1	0.630	0.61
2.	Analyst 2	0.636	0.72
3.	Instrument 1	0.633	0.64
4.	Instrument 2	0.637	0.70

5. Conclusion

The developed UV spectrophotometric methods exhibited excellent linearity within the concentration range of 2–10 µg/mL for both Tadalafil and Dapoxetine Hydrochloride. The correlation coefficient values were found close to 1, indicating good linear relationships between concentration and absorbance.

The recovery studies confirmed the accuracy of the methods with percentage recoveries ranging between 99–100%. Precision studies demonstrated low %RSD values, confirming good repeatability and reproducibility of the developed methods.

The developed methods were validated successfully according to ICH guidelines and found suitable for routine pharmaceutical quality control analysis.

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