

DEVELOPMENT AND VALIDATION OF A STABILITY-INDICATING UV SPECTROSCOPIC METHOD FOR PALBOCICLIB WITH FORCED DEGRADATION STUDIES

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ABSTRACT

Palbociclib is a selective cyclin-dependent kinase (CDK) 4/6 inhibitor indicated for the treatment of hormone receptor-positive, HER2-negative advanced or metastatic breast cancer. The present study aimed to develop and validate a simple, accurate, precise, and stability-indicating UV-Visible spectrophotometric method for the quantitative estimation of Palbociclib in capsule dosage form according to International Council for Harmonisation (ICH) guidelines. Forced degradation studies were also performed to evaluate the stability profile of the drug under different stress conditions. Palbociclib standard and capsule formulations were analyzed using a Shimadzu UV-Visible spectrophotometer with 0.1 N hydrochloric acid as the solvent. The absorption spectrum was scanned between 200 and 400 nm, and the maximum absorbance (λ_{max}) was observed at 258 nm. Calibration standards ranging from 5–30 $\mu\text{g/mL}$ were prepared, and the analytical method was validated for linearity, accuracy, precision, limit of detection (LOD), and limit of quantification (LOQ). Stability-indicating capability was evaluated through acid, alkaline, oxidative, thermal, and photolytic degradation studies. The developed method exhibited excellent linearity over the concentration range of 5–30 $\mu\text{g/mL}$ with a regression equation of $y = 0.020x + 0.001$ and a correlation coefficient close to 0.999. It is suitable for routine quantitative estimation of Palbociclib in capsule dosage forms and can be effectively employed in quality control laboratories for routine pharmaceutical analysis and stability evaluation.

Keywords: Palbociclib, UV-Visible spectroscopy, Method validation, Stability-indicating method, Forced degradation, ICH guidelines.

INTRODUCTION

Solubility refers to a substance's ability to dissolve in another substance, creating a homogeneous solution. This property depends on factors like the type of solvent used, temperature, and pressure. When the solubility limit is reached, further addition of solute does not increase its concentration in the solution. Solubility equilibrium occurs when dissolution and precipitation processes balance. Sometimes, solutions can become supersaturated, which is a meta stable state¹⁻². Solubility is not the same as the

ability to dissolve, as it can also involve chemical reactions. Solubility is commonly expressed in units of concentration, and its methods of expression vary. Solubility plays a crucial role in drug development, especially for oral delivery. Solubility is the phenomenon of dissolution of solid in liquid phase to give a homogenous system and is one of the important parameter to achieve desired concentration of drug in systemic circulation for pharmacological response. Poorly water-soluble drugs after oral administration often require high doses in order to reach therapeutic plasma concentrations. The bioavailability of an orally administered drug depends on its solubility in aqueous media over different pH ranges. The insufficient dissolution rate of the drug is the limiting factor in the oral bioavailability of poorly water soluble compounds. Various techniques are used for the improvement of the aqueous solubility, dissolution rate, and bioavailability of poorly water soluble drugs include micronization, chemical modification, pH adjustment, solid dispersion, complexation, cosolvency, micellar solubilization, hydrotrophy etc. Palbociclib is an orally active cyclin-dependent kinase 4 and 6 inhibitor used in the treatment of hormone receptor-positive breast cancer. It acts by inhibiting cell cycle progression and suppressing proliferation of malignant cells. Analytical methods play a significant role in quality control and stability assessment of pharmaceutical compounds ².

Among the available analytical techniques, UV spectroscopic methods are widely preferred due to their simplicity, rapidity, accuracy, and cost-effectiveness. Stability studies and forced degradation studies are essential to establish the intrinsic stability characteristics of the drug and to develop stability-indicating analytical methods ³.

The present work aims to develop and validate a simple UV spectroscopic method for estimation of Palbociclib using 0.1N HCl as solvent and to perform forced degradation studies according to ICH guidelines.

MATERIALS AND METHODS

Materials

Palbociclib reference standard was obtained from a Hetero Healthcare Limited. Hydrochloric acid and distilled water used were of analytical grade.

Instrumentation

A double beam UV-Visible spectrophotometer equipped with matched quartz cells of 1 cm path length was used for analysis.

Preparation of 0.1N HCl

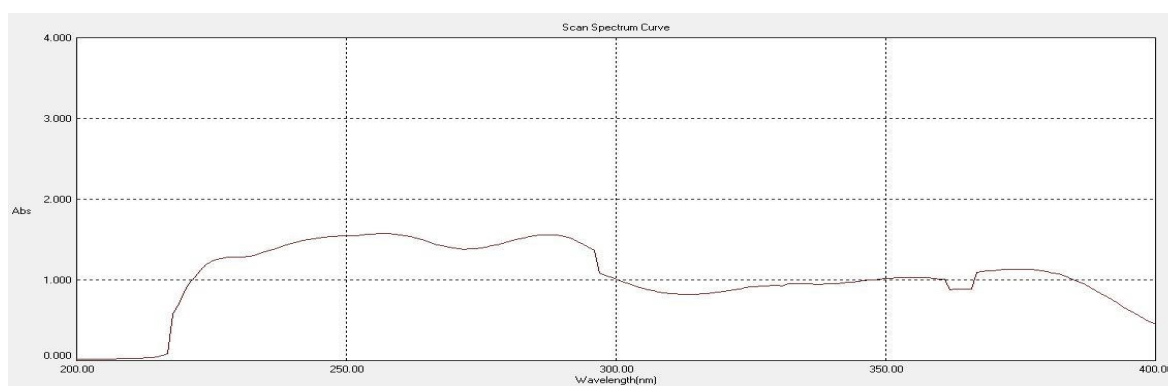
Approximately 8.5 ml of concentrated hydrochloric acid was diluted to 1000 mL with distilled water to obtain 0.1N HCl.

Determination of λ_{max}

The standard solution was scanned in the wavelength range of 200–400 nm using a UV spectrophotometer. The wavelength corresponding to maximum absorbance (λ_{max}) was found to be 258 nm.

Preparation of Standard Solution

Accurately weighed 10 mg of Palbociclib was transferred into a 100 mL volumetric flask and dissolved in 0.1N HCl to obtain a stock solution of 100 $\mu\text{g/mL}$.



UV Absorption Spectrum of Standard API Of Palbociclib Solution in 0.1 N Hydrochloric Acid

Preparation of Working Standard Solutions

Aliquots of the standard stock solution were transferred into a series of 10 mL volumetric flasks and diluted to volume with 0.1 N HCl to obtain concentrations of 5–30 $\mu\text{g/mL}$.

TABLE 1: PREPARATION OF WORKING STANDARD SOLUTION OF PALBOCICLIB

Volume taken(ml)	Final Volume (ml)	Concentration($\mu\text{g/mL}$)
0.5	10	5
1.0	10	10
1.5	10	15
2.0	10	20
2.5	10	25
3.0	10	30

3. Method Validation

Validation was carried out according to ICH guidelines.

3.1 Linearity

The absorbance of the working standard solutions was measured at 258 nm against 0.1 N HCl as blank. A calibration curve was plotted between concentration and absorbance. The method obeyed linearity in the range of 5–30 µg/ml ⁴.

TABLE 2: LINEARITY DATA OF PALBOCICLIB AT 258nm

S.NO.	DRUG CONCENTRATION	ABSORBANCE (258nm)
1	5	0.381
2	10	0.703
3	15	1.095
4	20	1.439
5	25	1.810
6	30	1.906

Slope= 0.020 Intercept= 0.001 R²= 0.9

3.2 Accuracy

The accuracy is calculated by % recovery studies within known amount of sample solution. The determination of content is performed at three levels 80%,100%,120%

TABLE 3: ACCURACY STUDY FOR PALBOCICLIB

ACCURACY	AMOUNT TAKEN	AMOUNT RECOVERED	PERCENTAGE RECOVERY
80	8µg	7.92 µg	99.0%
100	10 µg	10.05µg	100.5%
120	12 µg	11.95µg	99.6%

Precision

Precision of proposed method is determined by intraday precision and inter day procedure expressed in terms of % RSD intraday precision. It was determined by recording 50%,100% and 120% of test concentration on same day but at different times in triplets. Studies showed %RSD values less than 2%, indicating good repeatability ⁵.

LOD and LOQ

Limit of detection and limit of quantification were calculated using standard deviation and slope method.

$$\text{LOD}=3.3\sigma \quad \text{LOQ}=10\sigma S \quad \text{Where:}$$

- σ = standard deviation
- S = slope of calibration curve

TABLE 4: LOD AND LOQ FOR PALBOCICLIB

SD	SLOPE
0.002	0.02

Forced Degradation Studies ^{6,7}

Acidic Degradation

The sample solution was treated with 1 mL of 0.1 N HCl and kept for 1 hour. It was then neutralized, diluted, and analysed.

Alkaline Degradation

The sample solution was treated with 1 mL of 0.1 N NaOH and kept for 1 hour, followed by neutralization and analysis.

Oxidative Degradation

The sample was treated with 3% hydrogen peroxide and kept for 30 minutes before analysis

Thermal Degradation

The sample was heated at 60°C for 1 hour.

Photolytic Degradation

The sample was exposed to sunlight or UV light for a specified period and then analyzed.

TABLE 5: FORCED DEGRADATION STUDY OF PALBOCICLIB

CONDITIONS	ABSORBANCE	% DEGRADATION
Control	0.410	0%
Acid	0.350	14.6%
Base	0.330	19.5%
Oxidation	0.300	26.8%
Thermal	0.370	9.7%
Photolytic	0.360	12.2%

RESULTS AND DISCUSSION

Results:

The developed UV spectrophotometric method for estimation of Palbociclib in capsule dosage form showed reliable and reproducible results.

Palbociclib exhibited maximum absorbance (λ_{max}) at 258 nm in 0.1 N HCl, which was selected as the analytical wavelength. The calibration curve constructed in the concentration range of 5–30 $\mu\text{g/mL}$ showed a linear relationship between absorbance and concentration, confirming adherence to Beer–Lambert law.

The regression equation was found to be: $y = 0.020x + 0.001$, with a correlation coefficient (r^2) of 0.999, indicating excellent linearity.

The accuracy of the method was confirmed by recovery studies, with percentage recovery values ranging from 99.0% to 100.5%, which lies within acceptable limits (98–102%). Precision studies showed %RSD values less than 2%, indicating good repeatability.

The LOD and LOQ values were found to be 0.33 $\mu\text{g/mL}$ and 1.0 $\mu\text{g/mL}$, respectively, demonstrating that the method is sensitive enough for detection and quantification of the drug.

Forced degradation studies revealed that Palbociclib undergoes degradation under various stress conditions. Maximum degradation was observed under oxidative conditions, followed by alkaline and

acidic conditions. Thermal and photolytic degradation showed comparatively lesser degradation.

Discussion:

The selection of 0.1 N HCl as solvent provided good solubility and stability of the drug, resulting in clear and consistent absorbance readings. The linearity observed within the concentration range of 5–30 µg/mL confirms the applicability of the method for quantitative analysis. The high correlation coefficient ($r^2 = 0.999$) indicates a strong linear relationship, validating the method's reliability. Accuracy and precision results comply with ICH guidelines, indicating that the method produces consistent and reproducible results without significant error. The low values of LOD and LOQ demonstrate the sensitivity of the method, making it suitable even for low concentration analysis. Forced degradation studies confirm that the method is stability-indicating, as it can effectively detect changes in drug concentration in the presence of degradation products.

CONCLUSION

A simple, precise, accurate, and stability-indicating UV spectroscopic method was successfully developed and validated for estimation of Palbociclib using 0.1N HCl as solvent. The method complied with ICH validation requirements and was found suitable for routine pharmaceutical analysis and stability studies.

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