International Journal for Multidisciplinary Research (IJFMR)
E-ISSN: 2582-2160 ● Website: www.ijfmr.com ● Email: editor@ijfmr.com

Development and Validation of a Simple Rp-HPLC Method for the Estimation of Palbociclib in Bulk and Pharmaceutical Dosage Form

Syeda Kulsum[1], Ayesha Naz[2], Pittala Divya[3], Amena Samreen[4], Summaiya Tazbeen[5], Maheen Tazbeen[6], Syeda Samreen Farhana[7]

[1]Professor, Department Of Pharmaceutical Analysis, Mrm College Of Pharmacy, Ibrahimpatnam, Hyderabad.
[2]Associate Professor, Department Of Pharmaceutics, Mrm College Of Pharmacy, Ibrahimpatnam, Hyderabad.
[3,4,5,6,7]MRM College Of Pharmacy, Ibrahimpatnam, Hyderabad

ABSTRACT

A simple, accurate, and precise reverse-phase high-performance liquid chromatographic (RP-HPLC) method was developed and validated for the estimation of Palbociclib in bulk and tablet dosage form. Chromatographic separation was achieved using a Waters HPLC system (Alliance 2695) equipped with a PDA detector and Symmetry ODS C18 column (4.6 mm × 150 mm, 5 µm). The mobile phase consisted of acetonitrile and methanol (60:40 % v/v) at a flow rate of 1.0 mL/min. Detection was carried out at 240 nm with an injection volume of 10 µl. The method was validated as per ICH Q2(R1) guidelines for linearity, accuracy, precision, and robustness. The method showed good linearity in the selected concentration range with acceptable correlation coefficient. The developed method was found to be simple, rapid, sensitive, and reproducible. Hence, the method can be successfully applied for routine analysis of Palbociclib in bulk and pharmaceutical dosage forms.

Keywords: Palbociclib, RP-HPLC, Validation, ICH Q2(R1), Pharmaceutical Analysis

1. INTRODUCTION

Palbociclib is a selective cyclin-dependent kinase (CDK 4/6) inhibitor widely used in the treatment of hormone receptor-positive and HER2-negative breast cancer. It functions by inhibiting cell cycle progression from the G1 phase to the S phase, thereby suppressing tumor cell proliferation. Due to its therapeutic importance, the development of reliable analytical methods for its quantification in pharmaceutical dosage forms is essential.

High-performance liquid chromatography (HPLC) is one of the most widely used analytical techniques for the estimation of drugs due to its high sensitivity, accuracy, and reproducibility. Although a few analytical methods have been reported for Palbociclib, there is still a need for a simple, rapid, and cost-effective RP-HPLC method suitable for routine analysis.[1,2]

The present study aims to develop and validate a robust RP-HPLC method for the estimation of Palbociclib in bulk and tablet dosage forms in accordance with ICH Q2(R1) guidelines.[3]

2. MATERIALS AND METHODS

2.1 Materials

Palbociclib pure drug was obtained as a gift sample and the marketed tablet formulation was procured from a local pharmacy. All solvents used, including acetonitrile and methanol, were of HPLC grade, and water was purified prior to use.

2.2 Instrumentation

The analysis was carried out using a High Performance Liquid Chromatography (HPLC) system (**Waters Alliance 2695 HPLC System**) equipped with a quaternary pump and autosampler. Detection was performed using a photodiode array (PDA) detector with a deuterium lamp as the ultraviolet light source. Chromatographic separation was achieved on a **Waters Symmetry ODS C18 Column 4.6 × 150 mm, 5 µm**, which was found suitable for the analysis.[4,5]

2.3 Chromatographic Conditions

Column	:	Symmetry ODS C18 (4.6 x 150mm, 5m)
Column temperature	:	Ambient
Wavelength	:	240 nm
Mobile phase ratio	:	ACN: Methanol (60:40% v/v)
Flow rate	:	1.0ml/min
Injection volume	:	10 µ

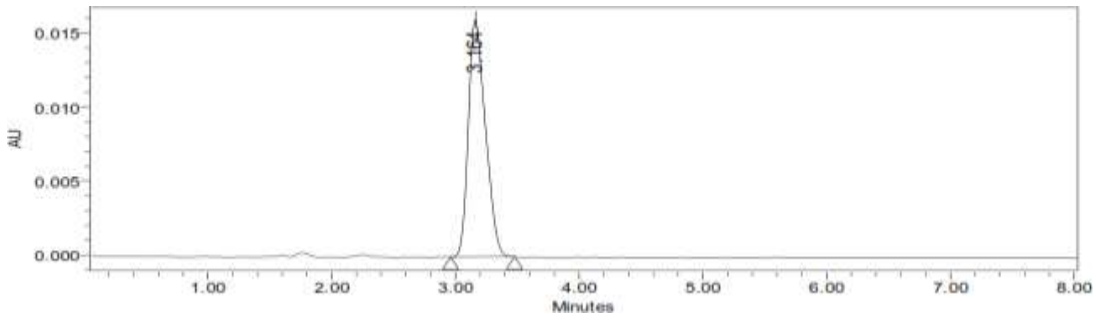
2.4: Preparation of Standard Solution:

Accurately weigh and transfer 10 mg of Palbociclib API and transfer it into 10 ml clean dry volumetric flask, add about 10 ml of diluent and sonicate to dissolve it completely and make volume up to the mark with the same solvent. (Stock solution). Further pipette 0.3ml of Palbociclib from stock solution into a 10ml volumetric flask and dilute up to the mark with diluent.[6,7]

2.5 Preparation of sample solution

Accurately weigh 20 tablets crush in mortar and pestle and transfer equivalent to 10 mg of Palbociclib, sample into a 10ml clean dry volumetric flask add about 7ml of diluent and sonicate to dissolve it completely and make volume up to the mark with the same solvent. (Stock solution) Further pipette 0.3ml of above stock solution into a 10ml volumetric flask and dilute up to the mark with diluent.

CHROMATOGRAM OF PALBOCICLIB



3. RESULTS AND DISCUSSION

3.1 Method Development and Optimization

Various mobile phase compositions were evaluated to achieve optimal chromatographic conditions. The combination of acetonitrile and methanol (60:40 % v/v) provided a sharp and symmetrical peak with

acceptable system suitability parameters. The selected wavelength of 240nm ensured adequate sensitivity for detection.

3.2 Method Validation

The method was validated in accordance with ICH Q2(R1) guidelines.

3.2.1 Linearity

The calibration curve demonstrated a linear relationship between concentration and peak area over the range of 10–50 µg/ml. The results are presented in Table 1. The correlation coefficient (r^2) was found to be 0.999, indicating excellent linearity.

Table 1: DATA FOR LINEARITY

Concentration g/ml	Average Peak Area
10	78683
20	146545
30	213584
40	279895
50	346568

3.2.2 Accuracy

Accuracy was assessed by recovery studies at three different levels (50%, 100%, and 150%). The results, summarized in Table 2, indicate that the method is accurate within acceptable limits.

Table 2: THE ACCURACY RESULTS FOR PALBOCICLIB

%Concentration	Area	Amount Added (ppm)	Amount Found (ppm)	%Recovery	Mean Recovery
50%	109283.3	15	15.060	100.40%	
100%	212732	30	30.124	100.413%	
150%	316263.3	45	45.201	100.446%	100.42%

3.2.3 Precision

Precision of the method was evaluated by repeatability studies. The %RSD value was found to be less than 2%, indicating good precision. The %RSD was calculated to be 0.12, confirming the precision of the method.

3.2.4 System Suitability parameters

System suitability parameters were evaluated to ensure the adequacy of the chromatographic system.

Table3: SYSTEM SUITABILITY PARAMETERS

SNO	Parameter	Palbociclib	Acceptance criteria
1	Retention Time (min)	4.85	-
2	Theoretical Plates (N)	5235	>2000

3	Tailing Factor	1.10	< 2
4	%RSD	0.12	< 2%

3.2.5 LOD and LOQ

The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.5 µg/mL and 1.5 µg/mL, respectively, indicating the sensitivity of the method.

TABLE 4: LOD and LOQ

SNO.	Parameter	Palbociclib
1	LOD (ng/ml)	0.5
2	LOQ (ng/ml)	1.5

3.2.6 Robustness

The robustness of the method was evaluated by making small deliberate changes in chromatographic conditions. The results indicated that the method remained unaffected, demonstrating its robustness.

4. CONCLUSION

A simple, sensitive, accurate, and reproducible RP-HPLC method has been successfully developed and validated for the estimation of Palbociclib in bulk and pharmaceutical dosage forms. The method complies with ICH Q2(R1) guidelines and demonstrates excellent performance characteristics.

Due to its simplicity, rapidity, and cost-effectiveness, the method can be effectively applied for routine analysis in pharmaceutical industries and quality control laboratories.

6. ACKNOWLEDGEMENTS

Authors are thankful to Sun Pharma Ltd for providing the sample of Palbociclib API and also thankful to the Management of MRM college of pharmacy for providing laboratory facilities to carry out this research.

REFERENCES

1. Vogel's Textbook of Quantitative Chemical Analysis. Mendham J, Denney RC, Barnes JD, Thomas MJK. 6th ed. Pearson Education; 2000.
2. Beckett and Stenlake Practical Pharmaceutical Chemistry. Beckett AH, Stenlake JB. 4th ed. CBS Publishers; 2002.
3. Remington The Science and Practice of Pharmacy. 21st ed. Lippincott Williams & Wilkins; 2006.
4. Sweetman SC (Ed.). Martindale The Complete Drug Reference. 36th ed. Pharmaceutical Press; 2009.
5. Skoog DA, Holler FJ, Crouch SR. Principles of Instrumental Analysis. 6th ed. Cengage Learning; 2007.
6. Sharma BK. Instrumental Methods of Chemical Analysis. Goel Publishing House; 2014.
7. International Council for Harmonisation. ICH Q2(R1): Validation of Analytical Procedures: Text and Methodology. Geneva; 2005.
8. Snyder and Kirkland Practical HPLC Method Development. Snyder LR, Kirkland JJ, Dolan JW. 2nd ed. Wiley; 2012.