The Bioscan AN INTERNATIONAL QUARTERLY JOURNAL OF LIFE SCIENCES 21(1) S (1) 909-920, 2026 www.thebioscan.com

DEVELOPMENT AND VALIDATION OF A STABILITY-INDICATING RP-HPLC METHOD FOR SIMULTANEOUS ESTIMATION OF NIRAPARIB AND ABIRATERONE IN PHARMACEUTICAL DOSAGE FORMS

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DOI: https://doi.org/10.63001/tbs.2026.v21.i01.S.I(1).pp909-920

KEYWORDS

RP-HPLC,
Niraparib,
Abiraterone,
Method validation,
Stability-indicating method,
ICH guidelines.

Received on:25-02-2026
Accepted on: 10-03-2026

Published on: 20-03-2026

Abstract

A novel, robust, and stability-indicating reverse-phase high-performance liquid chromatography (RP-HPLC) method was developed and validated for the simultaneous estimation of niraparib and abiraterone in pharmaceutical dosage forms. Chromatographic separation was achieved on an Inspire C18-EP column (4.6 × 250 mm, 5 µm) using a mobile phase consisting of KH_2PO_4 buffer (pH 3) and methanol in a ratio of 30:70 (v/v) at a flow rate of 1.0 ml/min. Detection was performed at 252 nm using a PDA detector. The developed method showed excellent system suitability with a resolution of 5.6 between the analytes, tailing factor <2, and theoretical plates >2000. The method was validated in accordance with ICH Q2(R1) guidelines for linearity, precision, accuracy, robustness, LOD, and LOQ. The calibration curves were linear with correlation coefficients greater than 0.999. The percentage recovery ranged between 98–101%, confirming the accuracy of the method. Precision studies showed %RSD values below 2%. Sensitivity was demonstrated with limits of detection and quantification of 0.10 µg/ml and 0.32 µg/ml respectively. Forced degradation studies under acidic, alkaline, oxidative, thermal, and photolytic conditions confirmed that the method is stability-indicating. The developed RP-HPLC method is simple, accurate, precise, and suitable for routine quality control and stability testing of formulations containing niraparib and abiraterone.

INTRODUCTION

Cancer remains one of the leading causes of mortality worldwide, and the development of targeted therapeutic agents has significantly improved treatment outcomes. Combination therapies targeting multiple biological pathways have become an effective strategy to improve therapeutic efficacy and delay drug resistance. Among these approaches, the combination of androgen-biosynthesis inhibitors and DNA-repair inhibitors has gained considerable attention in the management of advanced cancers.

Niraparib is a potent inhibitor of poly(ADP-ribose) polymerase (PARP) enzymes, particularly PARP-1 and PARP-2, which are involved in DNA repair mechanisms. Inhibition of PARP leads to accumulation of DNA damage and ultimately induces cancer cell death, especially in tumors with defective homologous recombination repair pathways such as BRCA-mutated cancers (Heo and Duggan, 2018). Niraparib is widely used as maintenance therapy for ovarian, fallopian tube, and primary peritoneal cancers.

Abiraterone acetate is a prodrug that is metabolized to abiraterone, a selective inhibitor of the CYP17 enzyme involved in androgen biosynthesis. By inhibiting androgen production in the testes, adrenal glands, and prostate tumor tissue, abiraterone reduces the growth of androgen-dependent prostate cancer cells (Attard et al., 2009). It is commonly used in the treatment of metastatic castration-resistant prostate cancer (mCRPC).

The combination of niraparib and abiraterone represents a promising therapeutic approach because it simultaneously targets DNA repair pathways and androgen signaling mechanisms. Such combination therapy has shown potential benefits in patients with metastatic castration-resistant prostate cancer.

Reliable analytical methods are essential for quality control and stability assessment of pharmaceutical formulations containing these drugs. High-performance liquid chromatography (HPLC) is one of the most widely used analytical techniques for the quantitative analysis of drugs in pharmaceutical dosage forms due to its high sensitivity, accuracy, and reproducibility.

According to International Conference on Harmonization (ICH) guidelines, analytical methods used for drug analysis should be stability-indicating and capable of distinguishing active pharmaceutical ingredients from degradation products, impurities, and excipients. Several analytical methods have been reported for the individual estimation of niraparib and abiraterone, including HPLC, LC-MS/MS, and UV spectrophotometric methods. However, only a limited number of methods are available for their simultaneous estimation in combined formulations.

Therefore, the present study aimed to develop and validate a simple, sensitive, and stability-indicating RP-HPLC method for the simultaneous estimation of niraparib and abiraterone in pharmaceutical dosage forms according to ICH Q2(R1) guidelines.

MATERIALS AND METHODS

Instruments

The chromatographic analysis was performed using a Waters Alliance HPLC system equipped with a 2695 separation module and PDA detector using Empower-3 software. A Labindia UV-3000+ spectrophotometer was used for wavelength scanning. A digital pH meter (Orion Star A111) and analytical balance (Scaletec SAB-203L) were used for sample preparation.

Chemicals and Reagents

Working standards of niraparib and abiraterone were supplied by MSN Laboratories. Potassium dihydrogen phosphate (KH_2PO_4), hydrochloric acid, sodium hydroxide, hydrogen peroxide, methanol, and acetonitrile (HPLC grade) were obtained from standard chemical suppliers.

Chromatographic Conditions

Separation was achieved on an Inspire C18-EP column (4.6 × 250 mm, 5 µm). The mobile phase consisted of KH_2PO_4 buffer (pH 3) and methanol in a ratio of 30:70 (v/v). The flow rate was maintained at 1.0 ml/min with an injection volume of 10 µl. Detection was carried out at 252 nm, and the total run time was 8 minutes.

Preparation of Standard Solution

Accurately weighed quantities of 20 mg of abiraterone and 4 mg of niraparib were transferred into a 20 ml volumetric flask and dissolved in diluent. Appropriate dilutions were prepared to obtain the working standard solutions.

Preparation of Sample Solution

Tablet powder equivalent to 20 mg of abiraterone and 4 mg of niraparib was accurately weighed and transferred to a volumetric flask containing diluent. The solution was sonicated and filtered through a 0.45 µm membrane filter before injection into the HPLC system.

Method Validation

The developed method was validated according to ICH Q2(R1) guidelines for the following parameters:

Linearity

Linearity was evaluated at five concentration levels ranging from 30–150 µg/ml for abiraterone and 6–30 µg/ml for niraparib.

Precision

Precision was evaluated by injecting six replicate samples and calculating %RSD.

Accuracy

Accuracy was assessed by recovery studies at 50%, 100%, and 150% concentration levels.

Limit of Detection and Limit of Quantification

LOD and LOQ were determined based on signal-to-noise ratios of 3:1 and 10:1 respectively.

Robustness

Robustness was assessed by deliberate variations in flow rate and mobile phase composition.

Forced Degradation Studies

Forced degradation studies were conducted under acidic, alkaline, oxidative, thermal, and photolytic conditions to evaluate the stability-indicating nature of the method.

RESULTS & DISCUSSION

CHROMATOGRAPHIC CONDITIONS

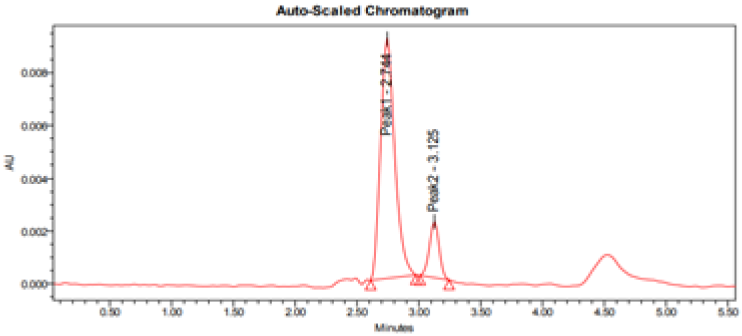

Figure 1

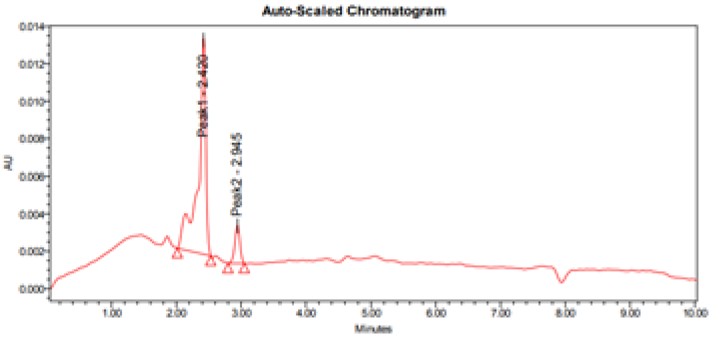

Figure 2

System Suitability:

Optimized Chromatographic Conditions:

Instrument used : High performance liquid chromatography equipped with Auto Sampler and PDA detector
Temperature : Ambient
Column : Inspire C18-EP (4.6 x 250mm, 5µm)
Mobile phase : 30% KH_2PO_4 pH 3:70% Methanol(30:70ml)
Flow rate : 1.0ml/min
Wavelength : 252 nm
Injection volume : 10µl
Run time : 8min.

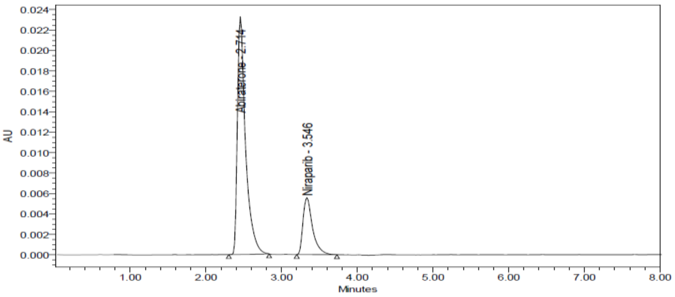

Figure 3: Chromatogram for system suitability

Table 1: Results of system suitability parameters

S.No	Name	RT(min)	Area (µVsec)	Height (µV)	USP tailing	Resolution	USP plate count
1	Abiraterone	2.714	755741	205321	1.10	5.60	5814
2	Niraparib	3.546	385417	40174	1.20		3624

VALIDATION PARAMETERS:

ASSAY: Standard and sample solution injected as described under experimental work. Thecorresponding chromatograms and results are shown below.

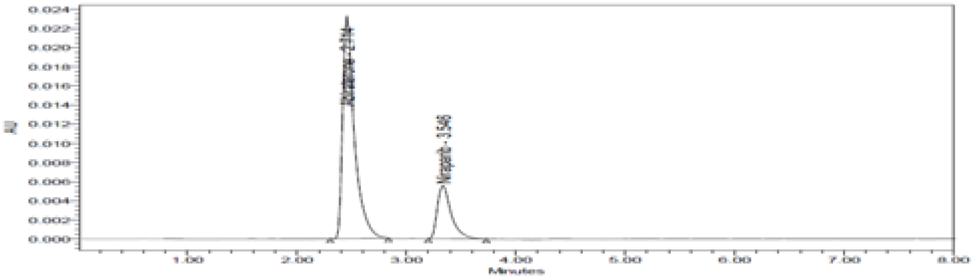

Figure 4: Chromatogram for Standard

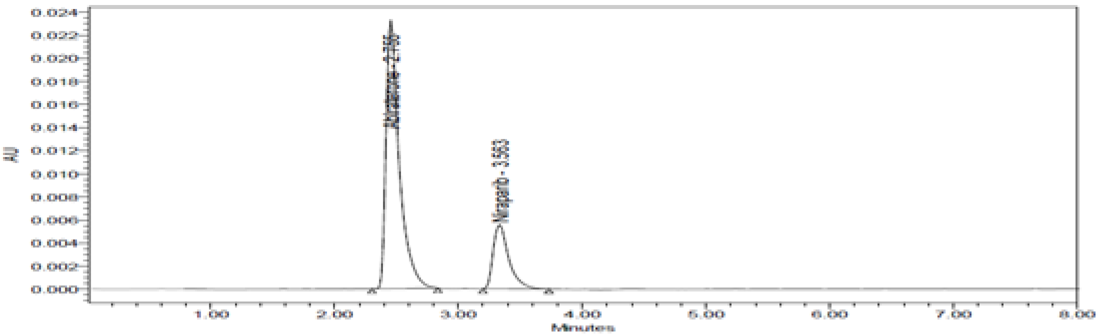

Figure 5: Chromatogram for Sample

Table 11: Results of Assay for Abiraterone and Niraparib

	Label Claim (mg)	% Assay
Abiraterone	500mg	100.1%
Niraparib	100mg	99.7%

LINEARITY

The linearity range was found to lie from 10µg/ml to 50µg/ml of Abiraterone and 2µg/ml to 10µg/ml Niraparib and chromatograms are shown below.

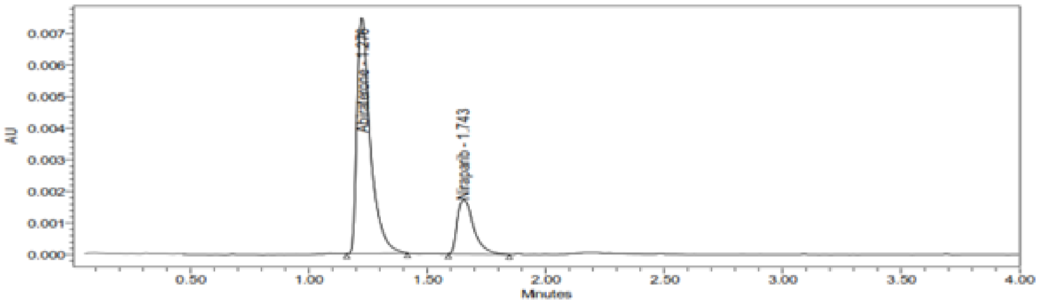

Figure 6: Chromatogram for linearity-1

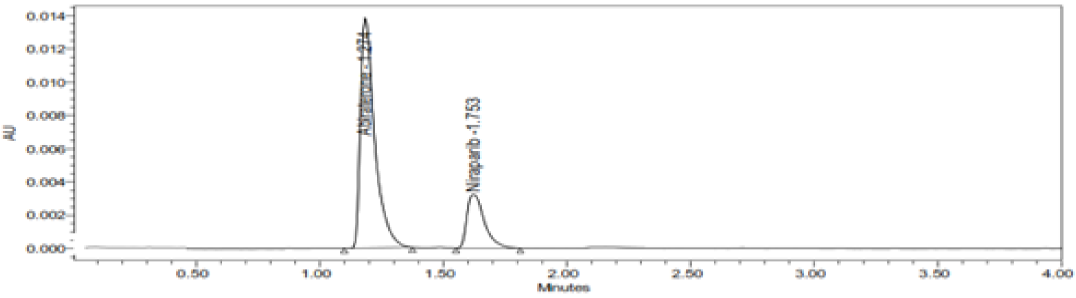

Figure 7: Chromatogram for linearity-2

Table 3: Area of different concentration of Abiraterone and Niraparib

S.N	Concentration (µg/ml)	Area
1	30	251881
2	60	480091
3	90	755582
4	120	1007512
5	150	1259397

Precision:

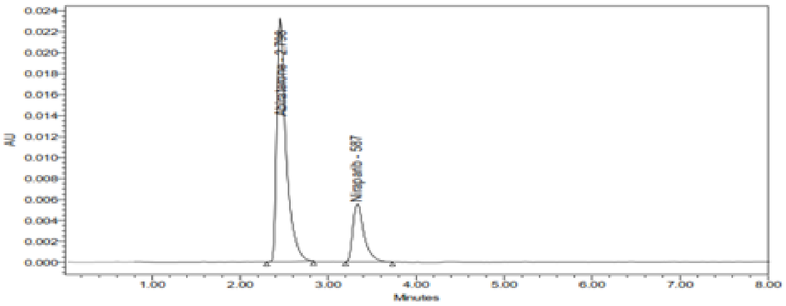

Figure 8: Chromatogram for Precision

Table 14: Results of Precision for Abiraterone and Niraparib

Injection	Areas of Abiraterone	Areas of Niraparib
Injection-1	746724	384715
Injection-2	740651	386647
Injection-3	745463	382251
Injection-4	740157	383167
Injection-5	746515	385201
Injection-6	744196	381974
Average	743951	383992.5
Standard Deviation	2894.624	1833.983
%RSD	0.4	0.5

INTERMEDIATE PRECISION (ruggedness)

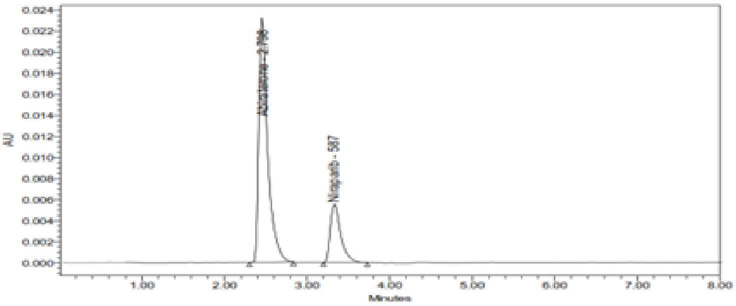

Figure 9: Chromatogram for ID Precision

ACCURACY:

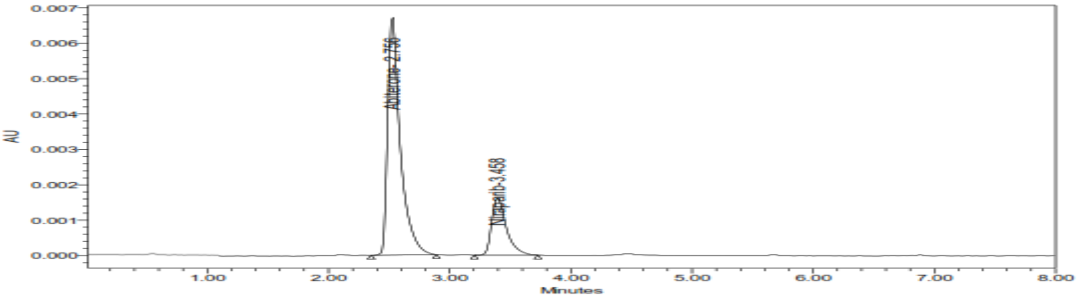

Figure 10: Chromatogram for Accuracy 50%

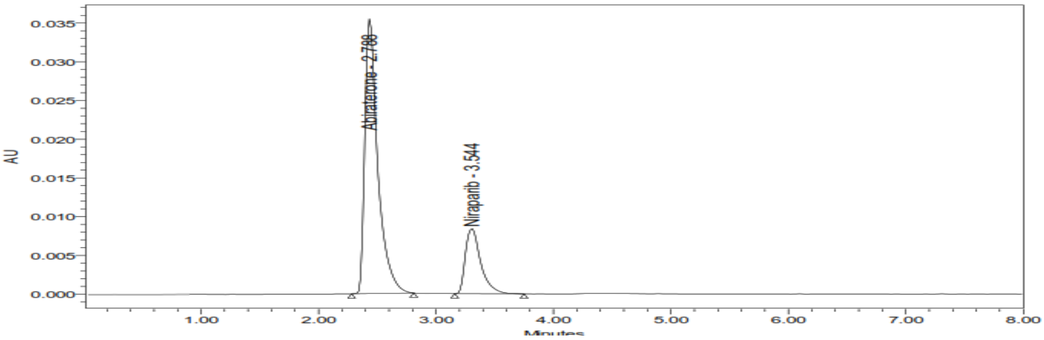

Figure 11: Chromatogram for Accuracy 150%

Table 5: Accuracy (recovery) data for Abiraterone and

%Concentration Abiraterone (at specification Level)	**Area***	**Amount Added (mg)**	**Amount Found (mg)**	**% Recovery**	**Mean Recovery**
50%	375417	10	9.92	99.2	
100%	745182	20	19.68	98.4	

150%	1145712	30	30.3	100.9	99.5

LIMIT OF DETECTION FOR ABIRATERONE AND NIRAPARIB

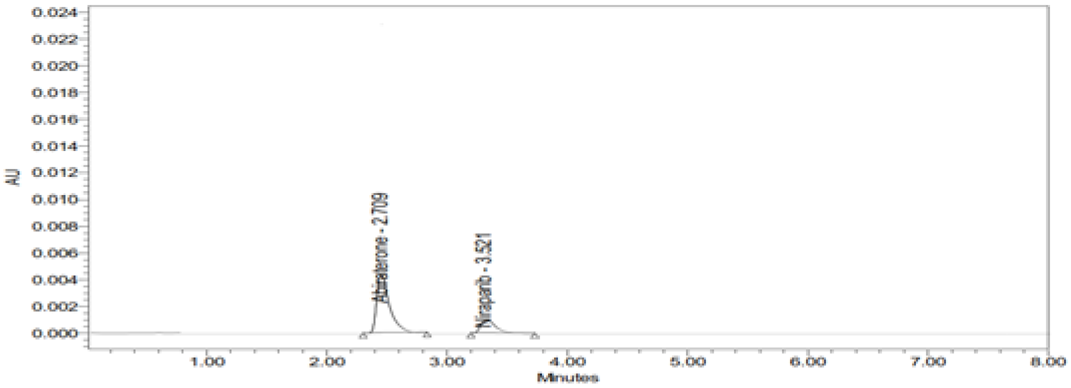

Figure 12: Chromatogram of Abiraterone and Niraparib showing LOD

Table 6: Results of LOD

Drug name	Baseline noise (µV)	Signal obtained (µV)	S/N ratio	Conc.
Abiraterone	75	217	2.89	0.10µg/ml
Niraparib	75	221	2.95	0.10µg/ml

Limit of Quantification for Abiraterone and Niraparib

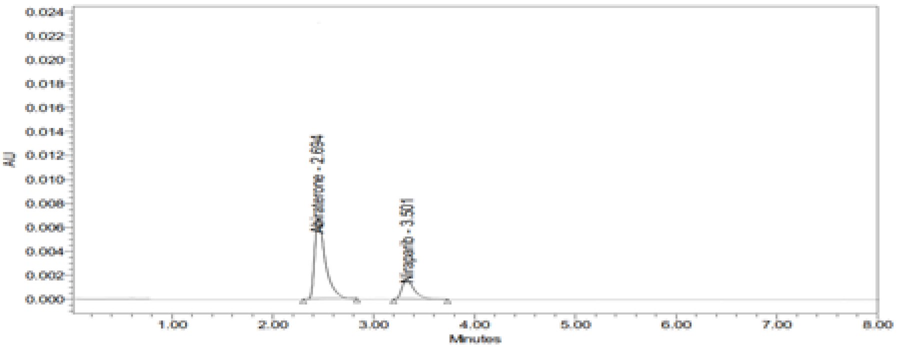

Figure 13: Chromatogram of Abiraterone and Niraparib showing LOQ

Table 7: Results of LOQ

Drug name	Baseline noise(μV)	Signal obtained(μV)	S/N ratio	CONC.
Abiraterone	75	738	9.84	0.32μg/ml
Niraparib	75	747	9.96	0.33μg/ml

ROBUSTNESS:

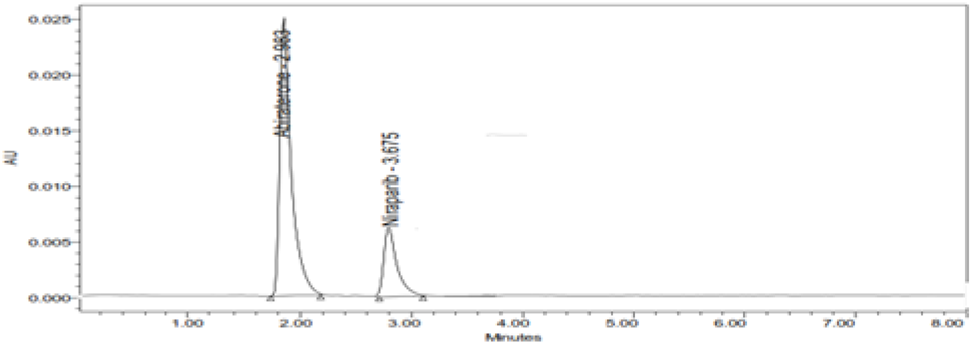

Figure 13: Chromatogram showing less flow

DEGRADATION STUDIES

Acid degradation:

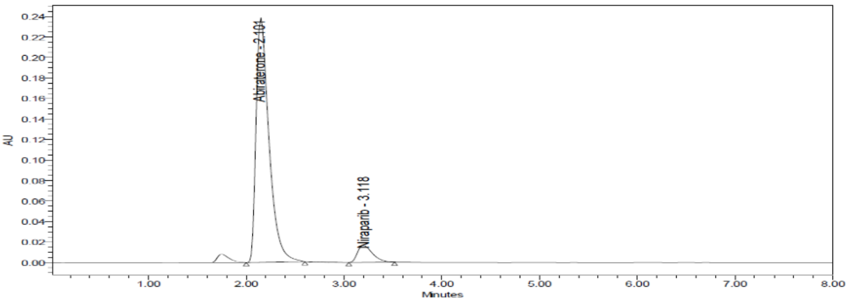

Fig.14: Chromatogram showing Acid

DISCUSSION

The developed RP-HPLC method successfully separated niraparib and abiraterone with retention times of approximately 3.546 min and 2.714 min, respectively. System suitability parameters such as resolution, tailing factor, and theoretical plates were within acceptable limits.

The calibration curves showed excellent linearity with correlation coefficients of 0.9996 for abiraterone and 0.9997 for niraparib. Precision studies showed %RSD values below 2%, indicating good repeatability of the method.

Accuracy studies showed recovery values between 98% and 101%, confirming the accuracy of the developed method. The limits of detection were found to be 0.10 µg/ml, and limits of quantification were 0.32 µg/ml for both analytes, demonstrating good sensitivity.

Robustness testing showed no significant changes in chromatographic parameters when small variations in flow rate and mobile phase composition were introduced.

Forced degradation studies demonstrated that both drugs were susceptible to degradation under acidic, basic, oxidative, thermal, and photolytic conditions, with maximum degradation observed under oxidative conditions. However, degradation products did not interfere with the analyte peaks, confirming the stability-indicating capability of the method.

CONCLUSION

A simple, precise, accurate, and stability-indicating RP-HPLC method was successfully developed and validated for the simultaneous estimation of niraparib and abiraterone in pharmaceutical dosage forms. The method showed excellent system suitability, linearity, accuracy, precision, and robustness as per ICH guidelines. Forced degradation studies confirmed that the method can effectively separate the drugs from their degradation products. Therefore, the developed method can be effectively applied for routine quality control analysis and stability studies of pharmaceutical formulations containing niraparib and abiraterone.

ACKNOWLEDGMENT

The authors thank the, Samskruti College of Pharmacy for technical assistance and support.

CONFLICT OF INTERESTS

The authors declare no conflict-of-interest

ETHICS APPROVAL

Not applicable

FUNDING

This study received no specific funding from public, commercial, or not-for-profit funding agencies.

AI TOOL DECLARATION

The authors declares that no AI and related tools are used to write the scientific content of this manuscript.

DATA AVAILABILITY

Data will be available on request

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