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Estimation of Clindamycin and Miconazole Nitrate in Soft Gelatin Capsules By RP-HPLC

P. Amani[1], K. Chaithanya[2], N. Karnakar[3] and R. Narasimha Rao[4]
[1,2,3,4] **Venkateshwara Institute of Pharmaceutical Sciences, Charlapally, Nalgonda, Telangana.**

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***Corresponding Author Email:** amanipaduri0002@gmail.com

Abstract
New method was established for simultaneous estimation of Clindamycin and Miconazole nitrate by RP-HPLC method. The chromatographic conditions were successfully developed for the separation of Clindamycin and Miconazole nitrate by using ACE C18 column (4.6×150mm) 5µ, flow rate was 1.2 ml/min, mobile phase ratio was (70:30 v/v) methanol: Phosphate buffer pH 3 (pH was adjusted with orthophosphoric acid), detection wavelength was 240nm. The instrument used was WATERS HPLC Auto Sampler, Separation module 2690, photo diode array detector 996, Empower-software version-2. The retention times were found to be 2.344 mins and 3.284 mins. The % purity of Clindamycin and Miconazole nitrate was found to be 101.27% and 99.97% respectively. The system suitability parameters for Clindamycin and Miconazole nitrate such as theoretical plates and tailing factor were found to be 4668, 1.3 and 6089 and 1.2, the resolution was found to be 6.0. The analytical method was validated according to ICH guidelines (ICH, Q2 (R1)). The linearity study n Clindamycin and Miconazole nitrate was found in concentration range of 50µg-250µg and 5µg-50µg and correlation coefficient (r^2) was found to be 0.999 and 0.999, % recovery was found to be 99.56% and 99.48%, %RSD for repeatability was 0.2 and 0.2, % RSD for intermediate precision was 0.2 and 0.1respectively. The precision study was precise, robust, and repeatable. LOD value was 3.17 and 5.68, and LOQ value was 0.0172 and 0.2125 respectively. Hence the suggested RP-HPLC method can be used for routine analysis of Clindamycin and Miconazole nitrate in API and Pharmaceutical dosage form.

Keywords
ACE C18 column, Clindamycin and Miconazole nitrate, RP-HPLC

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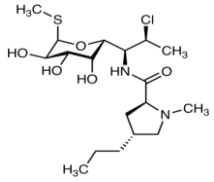
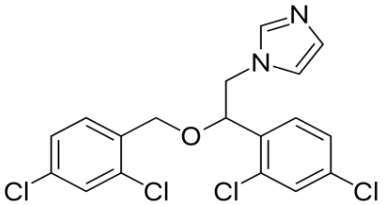
INTRODUCTION
Clindamycin is an antibiotic used for treatment of a number of bacterial infections, including bone or joint infections, pelvic inflammatory disease, strep throat, pneumonia, middle ear infections, and endocarditis.



Miconazole, sold under the brand name Monistat among others, is an antifungal medication used to treat ring worm, pityriasis versicolor, and yeast infections of the skin or vagina.



MATERIALS AND METHOD AND INSTRUMENTATION

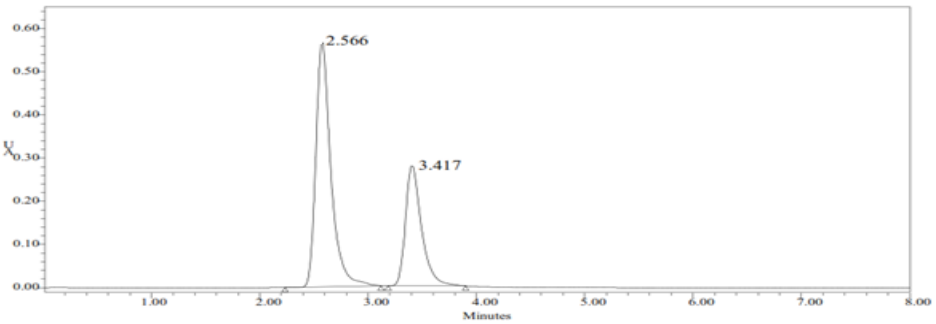
HPLC- Alliance, model No. Waters 2695, Empower 2, U.V double beam spectrometer UV 3000+ U.V win software Lab India Digital weighing balance (sensitivity 5mg) pH meter Sonicator Suction pump. Clindamycin and Miconazole nitrate, API, Ortho phosphoric acid, KH_2PO_4, K_2HPO_4, Acetonitrile, Methanol, Water.

Trial -5 (optimized method)

Chromatographic conditions

Column (4.6×150 mm) 5.0 µm	:	ACE C18
Column temperature	:	Ambient
Wavelength	:	240 nm
Mobile phase ratio Methanol: Phosphate buffer	:	70:30
Flow rate	:	1.2 ml/min
Auto sampler temperature	:	Ambient
Injection volume	:	10µl
Run time minutes	:	10.0

Fig. no.1 Chromatogram showing trial-5 injection



Preparation of the Clindamycin and Miconazole nitrate standard and sample solution

Sample solution preparation:

10 mg of Clindamycin and 1 mg Miconazole nitrate tablet powder were accurately weighed and transferred into a 10 ml clean dry volumetric flask, add about 2ml of diluent and sonicate to dissolve it completely and making volume up to the mark with the same solvent (Stock solution). Further pipette 10ml of the above stock solution into a 100ml volumetric flask and was diluted up to the mark with diluent.

Standard solution preparation

10 mg Clindamycin and 1 mg Miconazole nitrate working standard was accurately weighed and transferred into a 10ml clean dry volumetric flask and add about 2ml of diluent and sonicate to dissolve it completely and make volume up to the mark with the same solvent (Stock solution). Further pipette out 1ml of the above stock solution into a 10ml volumetric flask and was diluted up to the mark with diluent.

METHOD VALIDATION

- Linearity
- Accuracy
- Precision
- Intermediate Precision
- Limit of Detection
- Limit of Quantification
- Robustness
- System suitability testing

RESULTS AND DISCUSSIONS

Linearity

Table.No.1. Linearity Results for Clindamycin

S.No	Linearity Level	Concentration	Area
1	I	50 ppm	471543
2	II	100 ppm	656277
3	III	150 ppm	794999
4	IV	200 ppm	946124
5	V	250 ppm	1002139
Correlation Coefficient			0.999

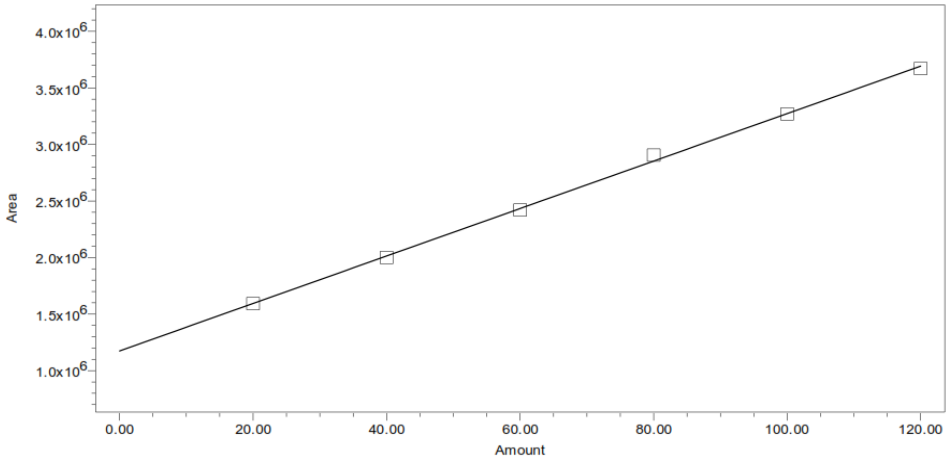
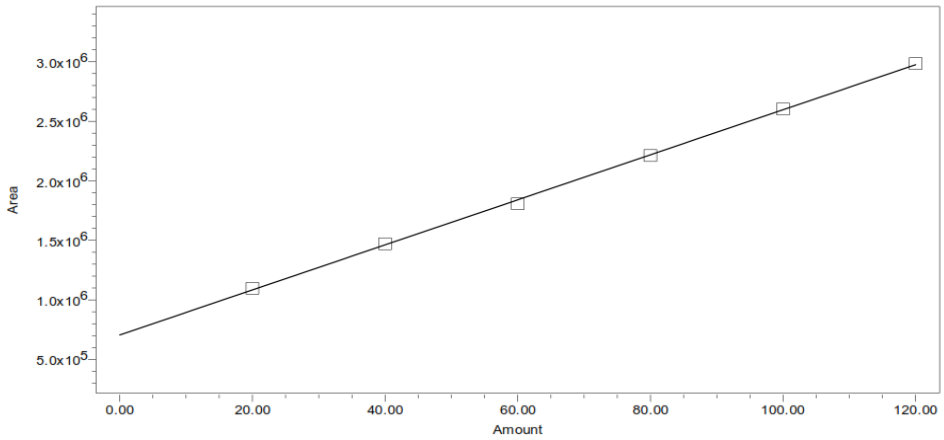


Fig.No.2. showing Calibration graph Clindamycin

Table.No.2. Linearity Results for miconazole nitrate

S. No	Linearity Level	Concentration	Area
1	I	5ppm	56472
2	II	10 ppm	73841
3	III	15ppm	92655
4	IV	20ppm	111541
5	V	25ppm	130567
Correlation Coefficient			0.999

Fig.No.3. Showing calibration graph for Miconazole nitrate



Accuracy

Table.No.3. Showing accuracy results for Clindamycin

%Concentration (At specification level)	Average area	Amount added (mg)	Amount found (mg)	% Recovery	Mean recovery
50%	656659	5	4.96	99.91%	
100%	1304258	10	9.98	99.18%	99.56%
150%	1854608	15	15.02	99.60%	

Table.No.4. Showing accuracy results for Miconazole nitrate

%Concentration (At specification level)	Average area	Amount added (mg)	Amount found (mg)	% Recovery	Mean recovery
50%	65312	0.5	0.99	99.53%	99.47%
100%	124509	1.0	1.05	99.38%	
150%	178517	1.5	1.495	99.52%	

Precision

Table.No.5. Showing% RSD results for Clindamycin

	Peak name	RT	Area	Height
1	Clindamycin	2.343	1302729	248455
2	Clindamycin	2.344	1309759	248699
3	Clindamycin	2.344	1302947	249526
4	Clindamycin	2.345	1303977	246695
5	Clindamycin	2.345	1303236	250012
MEAN			1304529.8	
STD.DEV.			2961.1	
%RSD			0.2	

Table.No.6. Showing %RSD results for Miconazole nitrate

	Peak name	RT	Area	Height
1	Miconazole nitrate	3.285	124263	19458
2	Miconazole nitrate	3.287	124487	19634
3	Miconazole nitrate	3.287	124175	19600
4	Miconazole nitrate	3.288	124894	19327
5	Miconazole nitrate	3.288	124495	19540
Mean			124462.7	
Std.dev.			278.6	
%RSD			0.2	

Intermediate precision/Ruggedness

Table.No.7. Showing results for intermediate precision of Clindamycin

	Peak name	RT	AREA	HEIGHT
1	Clindamycin	2.342	1305937	247870
2	Clindamycin	2.343	1306476	246764
3	Clindamycin	2.344	1304520	247140
4	Clindamycin	2.344	1300148	247280
5	Clindamycin	2.345	1308271	250012
Mean			1305070.2	
Std.Dev.			3061.8	
%RSD			0.2	

Table.No.8. Showing results for intermediate precision of Miconazole nitrate

	Peak name	Rt	Area	Height
1	Miconazole nitrate	3.278	122962	19165
2	Miconazole nitrate	3.281	122487	19115
3	Miconazole nitrate	3.281	122632	19073
4	Miconazole nitrate	3.281	122626	19003
5	Miconazole nitrate	3.283	122702	19123
Mean			122681.8	
Std.dev.			174.8	
%RSD			0.1	

Detection limit

Table.No.9. Showing results for Limit of Detection

Drug name	Standard deviation(σ)	Slope(s)	LOD(µg)
Clindamycin	382625.50	572175863	3.17
Miconazole nitrate	5862.40	467579210	0.0172

Quantitation limit

Table.No.10. Showing results for Limit of Quantitation

Drug name	Standard deviation(σ)	Slope(s)	LOQ(µg)
Clindamycin	381727.80	583265980	5.80
Miconazole nitrate	5681.30	469828490	0.212

Robustness

Table.No.11. Showing system suitability results for Clindamycin

S. No	Flow rate (ml/min)	System suitability results	
		USP Plate Count	USP Tailing
1	0.8	5339	1.4
2	**1**	**4668**	**1.3**
3	1.2	5216	1.4

Table.No.12. Showing system suitability results for Miconazole nitrate

S. No	Flow rate (ml/min)	System suitability results	
		USP Plate Count	USP Tailing
1	0.8	7036	1.3
2	**1**	**6089**	**1.2**
3	1.2	6998	1. 3

Table.No.13. Showing system suitability results for Clindamycin

S. No	Change in organic composition in the mobile phase	System suitability results	
		USP Plate Count	USP Tailing
1	5 % less	6232	1.4
2	***Actual**	**4668**	**1.3**
3	5 % more	6387	1.4

Table.No.14. Showing system suitability results for Miconazole nitrate

S. No	Change in organic composition in the mobile phase	System suitability results	
		USP Plate Count	USP Tailing
1	5 % less	5437	1.3
2	***Actual**	**6089**	**1.2**
3	5 % more	4817	1.2

SUMMARY AND CONCLUSION

Analytical method by UV spectrophotometry method provides precise, simple, accurate and rapid analytical method for the estimation of Clindamycin and Miconazole nitrate. The present analytical method was validated as per ICH Q2 (R1) guideline, and it was able to meet to specific acceptance criteria. The present developed analytical method can be used for its intended purpose.

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