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RESEARCH ARTICLE

Method Development and Validation for Estimation of Ornidazole in Dosage Form by Differential UV-Spectrophotometry Method

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ABSTRACT

Ornidazole is an antiamoebic drug. The drug is commercially available as tablets for oral administration. In the present work two simple, economical, precise and accurate UV spectrophotometric methods have been developed for the estimation of Ornidazole in bulk and pharmaceutical formulation. Differential spectrophotometric method in which λ_{max}=322 nm was selected for analysis of ornidazole. Linearity was observed in the concentration range 8-20µg/ml (r^2=0.9992). The methods were validated with respect to linearity, precision and accuracy studies. Recovery studies for Differential spectrophotometry was found to be 100.04%. The methods were found to be simple, precise and accurate and can be employed for routine quality control analysis of ornidazole in bulk as well as from its dosage form.

KEYWORDS

Ornidazole, Differential Spectrophotometry

INTRODUCTION

Ornidazole is chemically 1-chloro-3-(2-methyl-5-nitro-1*H*-imidazol-1-yl) propan-2-ol. It is the drug of choice for the treatment of protozoan infections. It is converted to reduction products that interact with DNA to cause destruction of helical DNA structure and strand leading to a protein synthesis inhibition and cell death in susceptible organisms.[1-4] Numerous UV, HPLC and HPTLC methods have been reported for estimation of these drugs alone as well as in combination with other drugs in pharmaceutical dosage forms. The present work aims to develop a simple, precise, accurate and validated Differential spectroscopic method for the estimation of Ornidazole in bulk and in tablet dosage form.

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MATERIALS AND METHOD

Instrumentation

UV 1700 Pharmaspec, Shimadzu Corporation, with 1cm matched cell was used to carry out the UV detection. The samples were weighed on electronic analytical balance (Reptech).

Chemical and Reagent

Ornidazole and 0.1N NaOH were obtained from the Department of Pharmaceutical Science's store (Rajkot, India). Orni (500mg) was procured from the market (Zydus health care).

Experimental Condition

Solvents: 0.1N NaOH, Distilled Water.

Preparation of Solutions

Preparation of Standard Stock Solution

Weigh accurately 50mg of ornidazole and dissolve in 50ml of 0.1N NaOH (1000µg/ml).

Weigh accurately 50mg of ornidazole and dissolve in 50ml of 0.1N HCl (1000µg/ml).

Preparation of Working Standard Solution

The solution was further diluted with 0.1N Noah and 0.1N HCl in separate to get the concentration of 100µg/ml. Different aliquots were taken from their working standards and diluted with 0.1N NaOH and 0.1 N HCl in separate to prepare a series of concentration from 8-20µg/ml solution .Spectrum was recorded by placing ornidazole in 0.1N Noah in sample cell and 0.1N HCl in reference cell. Absorbance was calculated at λ_{max} = 322 and to find out the absorbance. The calibration curve was prepared by plotting absorbance vs. concentration.

Preparation of Sample Solution

Powder of Tablet formulation equivalent to 50 mg ornidazole was transferred to a 50ml 0.1N NaOH and 0.1N HCl in separate (1000µg/ml). The solution was further diluted with 0.1N NaOH and 0.1N HCl separately to get the concentration of 100µg/ml. Then take suitable aliquots and prepare a concentration 10µg/ml solution in 0.1N NaOH and 0.1N HCl in separately.

Method Validation

The method was validated in terms of linearity, range, accuracy, and precision, limit of detection (LOD) and limit of quantitation (LOQ).

Linearity and Range

The linearity was evaluated by analyzing different concentrations of the standard solutions of ornidazole. These were scanned in the wavelength range 200-400 nm. Beer's law was obeyed in the concentration range 8-20µg/ml for both methods. The correlation coefficient was found to be 0.9992.

Precision

The reproducibility of the proposed methods was determined by performing standard solution (8µg/ml) at different time intervals on same day (n=6, Intraday precision) and on three different days (Inter-day precision).

Accuracy

To ascertain the accuracy of proposed methods, recovery studies were carried out by standard addition method at three different levels (80%, 100% and 120%). Percent recovery for ornidazole by both methods, was found in the range of 99% - 101%.

Robustness

Robustness of the method was performed by changing the Wavelength. The data clearly shows that the proposed method is robust at small but deliberate change.

Limit of Detection (LOD) and Limit of Quantitation (LOQ)

The LOD and LOQ were separately determined basis of standard calibration curve. The residual standard deviation of the regression line or the standard deviation of y-intercepts of regression lines was used to calculate LOD and LOQ. Following formulae were used; LOD= 3.3×D/S and LOQ= 10×D/S, where, D is the standard deviation of the y-intercepts of regression line and S is the slope of the calibration curve.

RESULTS AND DISCUSSION

For Differential spectrophotometric method λ_{max}=322 was selected for the analysis. Linear regression data showed a good relationship over a concentration range of 8-20µg/ml for ornidazole. The correlation coefficients (r^2) was found to be 0.9992 for method. The limit of detection and limit of quantification was found to be 3.50µg/ml and 10.63µg/ml in table 4. The values indicate that the method is sensitive. The intra-day and inter-day precisions were assessed by analyzing standard solutions. The lower values of % RSD indicate that the method is precise. Result shown table 2. To study accuracy of the developed method, recovery study was carried out using standard addition method at three different levels. The average % recoveries for both method were found to be 99%.-101%. The results revealed that there was no interference of excipients. The results of

accuracy are shown in Table 3. Change in the process parameters of method does not affect the acceptance result. So method is robust. Result shown in table 5.

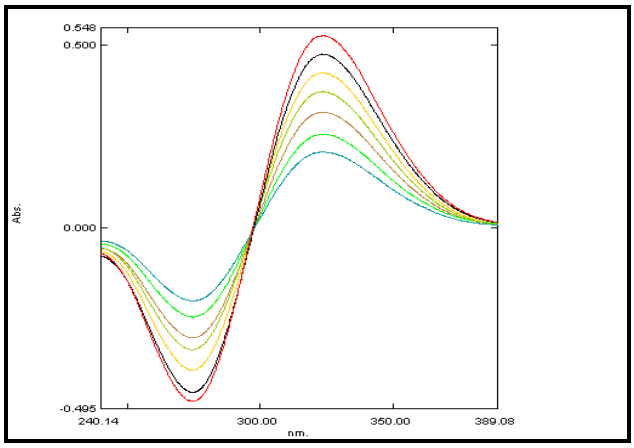

Figure: 1 Differential Spectra of Ornidazoless

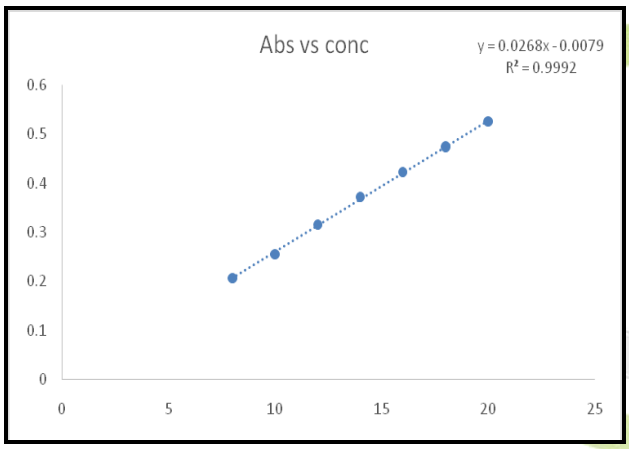

Figure-2: Linearity of Ornidazole

Method Validation Parameter

Table 1: Optical Characteristics of Ornidazol

Parameters	Value
Selected wavelength	319nm (λ_{max})
Beer's law limit (µg/ ml)	8-20
Correlation coefficient (r^2)	0.9992
Regression equation (Y = mx + c)	Y=0.0268x - 0.0079
Slope(m)	0.0268
Intercept (c)	0.0079

Table 2: Precision Study of Ornidazole

Methods	Conc. (µg/ml)	S.D	%RSD
Intraday precision			
Differential Method	8	0.002741	1.0393
Interday precision			
Differential Method	8	0.002759	0.9616

Table 4: LOD and LOQ study

Parameters	Differential Method (µg/ml)
LOD	3.50
LOQ	10.63

CONCLUSION

Differential spectrophotometric method was developed and validated as per ICH guidelines. The standard deviation and % RSD calculated for the proposed method are within limits, indicating high degree of precision of the method. The results of the recovery studies performed indicate the methods to be accurate. Hence, it can be concluded that the developed spectrophotometric methods are accurate, precise and can be employed successfully for the estimation of ornidazole in bulk and formulation. The proposed method was found to be simple, economical, rapid, precise and accurate for the determination of ornidazole. Thus, it can be easily and conveniently adopted for routine quality control analysis.

ACKNOWLEDGEMENTS

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Table 3: Recovery study of Ornidazole

Pre - analysed tablet solution µg/ml	Std. Drug added µg/ml	Recovery level	%Recovery	S.D	% RSD
8	6	80	100.13	0.001	0.27
8	8	100	100.06	0.0015	0.36
8	10	120	100.07	0.002	0.42

Table 5: Robustness Study

Drug	Parameter	Normal condition	Altered condition 1	Altered condition 2
Ornidazole	Wavelength	320nm	322nm	324nm
	S.D	0.001	0.000577	0.001
	%RSD	0.398	0.227	0.396

REFERENCES

1. Tripathi KD, Antimicrobial drugs: Essentials of medical pharmacology. New Delhi, ND, JP Brothers medical publishers (p) ltd., 2010, 706.
2. Rang HP, Dale MM, Ritter JM, Flower RJ, The molecular basis of chemotherapy, pharmacology, sixth ed. John F. Kennedy Boulevard, Philadelphia, PA, 2004, 651.
3. John HB, John MB, Organic and medicinal pharmaceutical chemistry, eleventh ed. Lippincott Williams & Wilkins, Philadelphia, PA, 2004.
4. Mazumder R, Nath LK, Giri TK, Choudhury PK, Kar AK, Sarkar MK, "Spectrophotometric Method Developmentand Determination of Ornidazole in Bulk and Tablet Dosage Form", International Journal of PharmTech Research, 2011, 3(1), 153-156**.**
5. Natraj KS, Suvarna Y, Prashanti G, Saikumar SV, "UV Spectroscopic Method Development and Validation Of Simultaneous Estimation of Ciprofloxacine and Ornidazole in Tablet Dosage Form", International Research Journal of Pharmacy, 2013, 4(7), 178-181.
6. ICH, Q2 (R1) Harmonized Tripartile Guideline, Validation of Analytical Procedures: Text and Methodology, International Conference on Harmonization, Geneva: Switzerland, 2005.
7. ICH Streering Committee, "ICH Q2B Validation of Analytical Procedures: methodology. London (CPMP/ICH/281/95): European Agency for the Evaluation of Medicinal Products", International Commission on Harmonization, Switzerland, 1996.
8. The United State Pharmacopeia, USP28-NF23, Rockville, MD. United State Pharmacopeial Convention. Inc., 2005.

