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Spectrophotometric Method for Determination of Ornidazole

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ABSTRACT : Two simple, precise and accurate spectrophotometric methods have been developed and validated for determination of Ornidazole in bulk and solid dosage form. These methods involve formation of Schiff bases of reduced Ornidazole with Paradimethylaminobenzaldehyde (PDAB) and Trimethoxybenzaldehyde (TMB) with absorption maxima (λmax) at 505 nm and 385 nm respectively. The linearity was observed in the concentration range of 16-40 µg/mL and 5-50 µg/mL for method A and method B respectively. The assay result was found to be in good agreement with label claim. The recovery studies were carried out at three different levels. The methods were validated statistically and by recovery studies and it were found to be accurate, precise and reproducible for determination of Ornidazole in bulk and solid dosage form.
KEY WORDS: Ornidazole, Spectrophotometric, Validation

INTRODUCTION

Ornidazole[1], chemically α-chloromethyl-2-methyl-5nitro-1H-imidazole-1-ethanol, with molecular formula $C_7H_{10}N_3O_3Cl$ is an antimicrobial agent[2]. It is not an official drug in any pharmacopoeia. Literature review revealed that few methods[3-8] are available for the determination of Ornidazole in bulk and solid dosage form.

The present study describes accurate, precise and reproducible two spectrophotometric methods for estimation of Ornidazole in bulk and solid dosage form. The methods were validated by using various parameters as per ICH guidelines.

EXPERIMENTAL

INSTRUMENTS AND MATERIALS:

Pure Ornidazole was obtained as gift sample from Sun Pharmaceutical Limited, Mumbai, India. The Shimadzu UV-Visible spectrophotometer 1601 was used with spectral bandwidth 3 nm and wavelength accuracy (with automatic wavelength correction) 0.5 nm. All the apparatus and instruments were calibrated and validated before starting the experimental work.

MATERIALS AND METHODS:

PREPARATION OF REDUCED ORNIDAZOLE SOLUTION: 100 mg of Ornidazole was accurately weighed and dissolved in 25 ml of the distilled water, 3 gm of the zinc granules, 10 ml of the hydrochloric acid was added and refluxed at 80°C for 10 min. The solution mixture was filtered through Whatmann paper No. 42 and the volume was made upto 100 ml, to get the final concentration of 1 mg/ml. 10 ml of this solution was further diluted to 100 ml to get 100 mcg/ml solution of reduced Ornidazole.

METHOD A

PROCEDURE: To 1 ml of standard solution in 10 ml volumetric flask, 1 ml of 1 % paradimethylaminobenzaldehyde was added. The solution mixture was kept for 15 min at 37°C. The volume was made upto 10 ml with the distilled water. The absorbance was measured at 505 nm against the reagent blank.

METHOD B

PROCEDURE: To 1 ml of standard solution in 10 ml volumetric flask, 0.6 ml of 0.1 N HCl and 1.2 ml of 5 % 2, 4, 6 Trimethoxybenzaldehyde solution was added. The solution mixture was heated at 80°C for 5 min, cooled and the volume was made upto 10 ml with distilled water. The absorbance was measured at 385 nm against the reagent blank.

VALIDATION OF ANALYTICAL METHOD:

Validation is the process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting its predetermined specifications and quality attributes. Both the methods

were validated by various parameters as per ICH guidelines (Table 1).

1. LINEARITY:

The linearity of Ornidazole was found to be 16-40 µg/mL and 5-50 µg/mL and linear regression value of R^2 = 0.9994 and 0.9988 for method A and method B respectively.

2. PRECISION:

The values of % RSD of all precision study were within the acceptable limits (less than 2 %). Hence the methods provide good precision and reproducibility.

3. SENSITIVITY:

Absorbance of standard solutions of Ornidazole was measured at 505 nm and 385 nm for method A and method B respectively. Sandell's sensitivity[9] for Ornidazole was calculated from formula.

$$(\mu gcm^{-3}\ AU) = \frac{\text{Conc. of drug } (\mu g 100\ mL^{-1})}{\text{Absorbance}} \times 0.001$$

The Sandell's sensitivity for Ornidazole at 505 nm and 385 nm for method A and method B was found to be 0.5104 µgcm^{-3}AU and 0.0544 µgcm^{-3}AU respectively.

4. ACCURACY:

Accuracy of the method was determined in the terms of % recovery of standard Ornidazole. Results of the recovery study were found to be within the acceptance criteria 100 ± 10 %, indicates sensitivity of the method towards detection of Ornidazole and non interference of excipients in the method (Table 2).

PROCEDURE FOR THE ANALYSIS OF TABLET FORMULATION:

BRAND NAME: Orni, Ornizin, Dazolic. Giro, Ornida

MANUFACTURER: Sun Pharmaceutical Limited, Mumbai, India.

PREPARATION OF REDUCED ORNIDAZOLE SAMPLE SOLUTION: 20 tablets were weighed, powdered; the powder equivalent to 100 mg of Ornidazole was dissolved in 25 ml distilled water, 3 gm of the zinc granules, 10 ml of the hydrochloric acid was added and refluxed at 80^{o}C for 10 min. The solution mixture was filtered through Whatmann paper No. 42 and the volume was made upto 100 ml. 10 ml of this solution was further diluted to 100 ml to get 100 mcg/ml solution of reduced Ornidazole.

Table 1: Validation Parameters for Ornidazole

SI. No.	Parameters		Method A	Method B
1.	Linearity	Range (µg/mL)	16-40	5-50
		Regression eq^{n}	y=0.0199x + 0.0471	y=0.0254x – 0.0103
		Correlation coefficient (R^2)	0.9994	0.9988
2.	Precision	Method (SD)	0.002817	0.004294
		% RSD	1.200282	1.30254
		Inter-day (SD)	0.000943	0.000340
		% RSD	0.12809	0.102175
		Intra-day (SD)	0.003113	0.003226
		% RSD	1.313047	1.360726
3.	Sandell's Sensitivity (µgcm^{-3} AU)		0.5104	0.0544
4.	Accuracy (% RSD) (n=3)		0.9537	0.9314

Table 2: Recovery studies of Ornidazole for Method A and Method B

Method	Conc. of standard drug (µg/ml) (A)	Conc. of marketed sample (µg/ml) (B)	Total drug Conc. (µg/ml) (A+B)	Absorbance*	Total Conc. of Ornidazole from standard curve (µg/ml)	Amount of sample (µg/ml)	% Recovery
A	20	2	22	0.4011	22.47	2.47	93.00
	20	4	24	0.5111	24.39	4.39	100.60
	20	8	28	0.6001	28.10	8.10	100.30
B	40	10	50	1.2320	50.45	10.45	100.90
	40	20	60	1.1110	60.99	20.90	104.50
	40	30	70	0.5611	71.02	31.20	101.70

*Average of three readings.

METHOD A

PROCEDURE: To 1 ml of sample solution in 10 ml volumetric flask, 1 ml of 1 % paradimethylaminobenzaldehyde was added. The solution mixture was kept for 15 min at 37^{o}C. The volume was made upto 10 ml with the distilled water. The absorbance was measured at 505 nm against the reagent blank.

METHOD B

PROCEDURE: To 1 ml of sample solution in 10 ml volumetric flask, 0.6 ml of 0.1 N HCl and 1.2 ml of 5 % 2, 4, 6 Trimethoxybenzaldehyde solution was added. The solution mixture was heated at 80^{o}C for 5 min, cooled and the volume was made upto 10 ml with distilled water. The absorbance was measured at 385 nm against the reagent blank.

RECOVERY STUDIES:

To check the accuracy of the proposed method, recovery studies were carried out at three different levels. The standard bulk drug was added at 3 different levels to the preanalyzed sample solution and then reanalyzed.

RESULT AND DISCUSSION

Ornidazole is an antimicrobial agent. Two spectrophotometric methods were developed and validated for determination of Ornidazole in bulk and solid dosage form. They showed absorption maxima (λmax) at 505 nm and 385 nm and linearity was observed in the concentration range of 16-40 µg/mL and 5-50 µg/mL for method A and method B respectively. The percentage label claim was found to be in good agreement. The % recovery was found to be in the range of 93-101 % and 100-101 % for method A and method B respectively, indicating no interference by excipients in the methods. The % RSD less than 2 indicated that the method was accurate and precise. The method was found to sensitive with respect to Sandell's sensitivity.

The proposed methods for the determination of Ornidazole were simple, accurate, linear, precise and reproducible. Hence the methods can be used for routine analysis of Ornidazole in bulk and solid dosage form.

Table 3: Analysis of tablet formulation

Label claim mg/tablet	Method	% Amount found	SD	% RSD
100 mg	A	100.061	0.0431	0.0400
100 mg	B	100.002	0.0014	0.0014

*Average of six determinations

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