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Two spectrophotometric methods for determination of ornidazole in tablets

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ABSTRACT

Two simple, precise and accurate colorimetric methods have been developed and validated for determination of Ornidazole in bulk and tablet formulation. These methods involves formation of complex diazonium salt of reduced Ornidazole with Metacresol reagent and Resorcinol reagent which shows absorption maxima (λmax) at 425 nm and 435 nm respectively. The linearity was observed in the concentration range of 5-40 µg/mL and 8-20 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 they were found to be accurate, precise and reproducible for determination of Ornidazole in bulk and solid dosage form.

Key words: Ornidazole, Colorimetry, Validation.

INTRODUCTION

Ornidazole[1,2] is an antimicrobial agent, chemically α-chloromethyl-2-methyl-5nitro-1H-imidazole-1-ethanol, with molecular formula. 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 colorimetric methods for estimation of Ornidazole in bulk and tablet formulation. The method was 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.

MATERIAL AND METHODS

Preparation of reduced Ornidazole solution

100 mg of Ornidazole was taken in a round bottom flask, 3 gm of the zinc granules, 10 ml of the hydrochloric acid and 25 ml of the distilled water was added, the mixture was refluxed at 80°C for 10 min. The solution mixture was filtered through Whattmann paper no. 42 and the volume was made upto 100 ml, so that the final concentration was 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 std and sample solution in 10 ml volumetric flask, 1 ml of 1 % sodium nitrite, 1 ml of ethanol, 1.5 ml of 1 % metacresol was added. The solution mixture was kept for 30 min at 5°C. The volume was made upto 10 ml with the distilled water. The absorbance was measured at 425 nm against the reagent blank.

Method B

Procedure

To 1 ml of std and sample solution in 10 ml volumetric flask, 1 ml of 3 % sodium nitrite, 0.1 ml of 0.1N NaOH and 0.3 ml of 0.1 % of resorcinol was added and the solution kept for 5 min at 5°C and the volume was made upto 10 ml with distilled water. The absorbance was measured at 435 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).

Linearity

The linearity of Ornidazole was found to be 5-40 µg/mL and 8-20 µg/mL and the linear regression value was found to be R^2 = 0.9979 and 0.9991 for method A and method B respectively.

Precision

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

Sensitivity

Absorbance of standard solutions of Ornidazole was measured at 425 nm and 435 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 425 nm and 435 nm for method A and method B was found to be 0.0236 $\mu gcm^{-3}AU$ and 0.1027 $\mu gcm^{-3}AU$ respectively.

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 analysis of Ornidazole in 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 and accurately weigh equivalent to 100 mg of Ornidazole was taken in a round bottom flask, 3 gm of the zinc granules, 10 ml of the hydrochloric acid and 25 ml of the distilled water was added, the flask was refluxed at 80°C for 10 min. The solution mixture was filtered through whattmann 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.

Method A

Procedure

To 1 ml sample solution in 10 ml volumetric flask, 1 ml of 1 % sodium nitrite, 1 ml of ethanol, 1.5 ml of 1 % metacresol was added. The solution mixture was kept for 30 min at 5°C. The volume was made upto 10 ml with the distilled water. The absorbance was measured at 425 nm against the reagent blank.

Method B

Procedure

To 1 ml of sample solution in 10 ml volumetric flask, 1 ml of 3 % sodium nitrite, 0.1 ml of 0.1N NaOH and 0.3 ml of 0.1 % of resorcinol was added and the solution kept for 5 min at 5°C and the volume was made upto 10 ml with distilled

water. The absorbance was measured at 435 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.

RESULTS AND DISCUSSION

Ornidazole is an antimicrobial agent. Two colorimetric methods were developed and validated for determination of Ornidazole in bulk and tablet formulation. They showed absorption maxima (ëmax) at 425nm and 435 nm and the linearity were observed in the concentration range of 5-40 µg/mL and 8-20 µg/mL for method A and method B respectively. The % recovery was found to be in range of 100-102 % 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.

Hence the developed method was found to be simple, accurate, precise and reproducible.

Table 1: Validation Parameters

S. No	Parameters		Method A	Method B
1.	Linearity	µg/mL	5-40	8-20
		Regression eq^{n}	y=0.0369x + 0.063	y=0.0254x – 0.0103
		R^2	0.9979	0.9991
2.	Precision	Method (SD)	0.0019	0.0011
		% RSD	0.4666	0.8689
		% RSDInter-day (SD)	0.16210.0007	0.44620.0006
		Intra-day (SD)	0.0054	0.0019
		% RSD	1.2615	1.5202
3.	Sandell's Sensitivity	At 425 nm	At 435 nm	
	µgcm^{-3} AU	0.0236	0.1028	
4.	Accuracy (% Recovery)	N = 3	100-102 %	100-101 %

Table 2: Recovery studies (Method A and Method B)

S. No.	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	0.9324	22.01	2.06	100.00
B	20	2	22	0.2179	22.01	2.01	100.00
A	20	4	24	1.0583	24.40	4.40	101.66
B	20	4	24	0.3691	24.06	4.06	100.20
A	20	8	28	0.0847	28.06	8.06	100.20
B	20	8	28	0.4009	28.15	8.75	100.50

*Average of three readings

Table 3: Analysis of tablet formulation

Label claim mg/tablet	Method	% Amount found	SD	% RSD
100 mg	A	100.08	0.0566	0.0565
100 mg	B	102.18	1.5415	1.5086

*Average of six determinations

CONCLUSION

The proposed methods for the determination of Ornidazole are simple, accurate, linear, precise and reproducible. Hence the methods can be used for routine analysis of Ornidazole in bulk and tablet formulation.

REFERENCES

1. Budavari S., Maryadele J. and Smith A. The Merck Index, 13th edition; Merk & Co., Inc. Ruchway: U.S.A. (Monograph No 6746), p 985 (1989).
2. CIMS-101, Sri Sudhindra Printing Press, 8th Cross, Malleshwaram: Bangalore-03, India. Apr - Jan 2008 [Update-2], p 367, 388.
3. Merdjan. H., Bonnat. C., Singlas. E., Diquite. B. Measurement of ornidazole by high performance liquid chromatography. *J Chromatogr*., **273**(2): 475-480 (1983).
4. Groppi. A., Papa. P., Montaqna. M., Carosi. G. Determination of ornidazole in human plasma and red blood cells using high performance liquid chromatography. *J Chromatogr.,* 380(2): 437-442 (1986).
5. Heizmann P., Geschke R., Zinapold K. Determination of ornidazole and its metabolites in biological fluids. *J Chromatogr.* **534**: 233-240 (1990).
6. Hassan Mohammed. Spectrophotometric quantitation of ornidazole and its formulations. *Pharmaceutical analysis.* **22**(2): 111-121 (1989).
7. Salman. A., Sumer. C. Spectrophotometric assay of ornidazole and nimorazole. *Scientia Pharmaceutica*. **64**(2): 145-149 (1996).
8. Ozkan S.A., Enturk Z., Biryol I. Determination of ornidazole in pharmaceutical dosage forms based on reduction at an activated glassy carbon electrode. *International Journal of Pharmaceutics.* 1997; 152(2): 137-144.
9. Beckette AH, Stenlake JB. Practical Pharmaceutical Chemistry. 4th ed. The Anthlone Press; London. 157, 281-307 (1988).