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Research Article

VALIDATED RP-HPLC METHOD AS A TOOL FOR THE ESTIMATION OF TINIDAZOLE IN PHARMACEUTICAL DOSAGE FORMS

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ABSTRACT

A simple, specific, accurate, precise and sensitive Reverse Phase High Performance Liquid Chromatographic method has been developed for the quantitation of Tinidazole in both pure and pharmaceutical dosage forms. . Chromatographic separation achieved isocratically on a ODS2 reverse phase column [Use Symmetry C18, 250 X 4.6mm, 5µ] utilizing a mobile phase of Methanol. The flow rate was 0.5 ml/min and the effluents were monitored at 310 nm. The retention time was 4.610min. The linearity was in the range of 5-25µg / ml. This method was validated for linearity, precision and accuracy. Statistical analysis proves that the method is reproducible and selective for the estimation of the said drug.

Keywords: RP-HPLC, Tinidazole, Validation, Mobile phase.

INTRODUCTION

Tinidazole is a nitroimidazole antitrichomonal agent to both adults and pediatric patients older than three years of age, effective against Trichomonas vaginalis, Entamoeba histolytica and Giardia lamblia infections.Tinidazole is designated chemically as a 1-[2-(ethylsulphonyl)ethyl]-2-methyl 5-nitroimidazole. The structural formula is C8H13N3O 4S. It is slightly soluble in acetone and dichloromethane. Sparingly soluble in methyl alcohol and practically insoluble in water. Tinidazole is a prodrug and antiprotozoal agent. The nitro group of Tinidazole is reduced in Trichomonas by a ferredoxin-mediated electron transport system. The free nitro radical generated as a result of this reaction is believed to be responsible for the antiprotozoal action. Several analytical methods that have been reported for the estimation of Tinidazole in pharmaceutical formulations which include HPLC, Spectrophotometric and derivative spectrophotometry methods. The objective of the work was to develop simple, accurate, precise and economic RP-HPLC method with lesser run time to estimate the Tinidazole in pharmaceutical dosage forms.

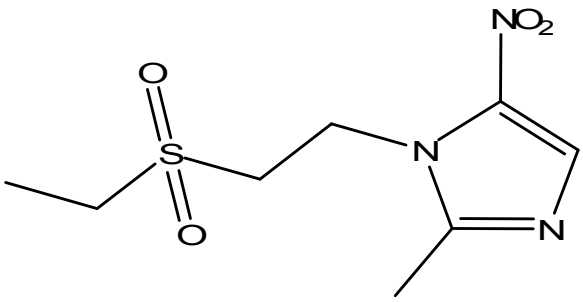

Fig.1: Chemical Structure of Tinidazole

MATERIALS AND METHODS:

The liquid chromatographic system consisted of following components: A Shimadzu HPLC model containing LC - 20AT

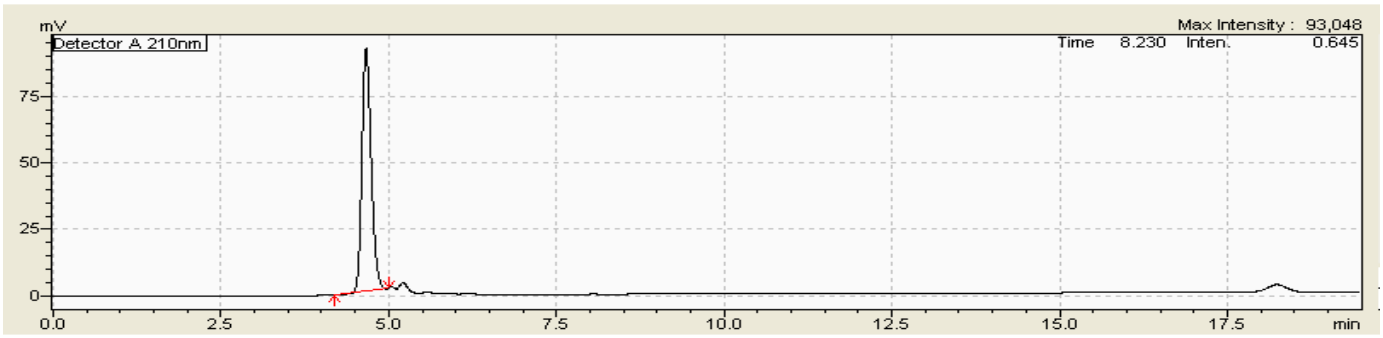

Fig.2: Typical RP-HPLC Chromatogram of Tinidazole by the proposed method.

Table 1: Optimized Chromatographic conditions for the proposed method

Parameters	**Optimized condition**
Column	ODS2 reverse phase column [Use Symmetry C18, 250 X 4.6mm, 5µ]
Mobile phase	Methanol
Flow rate	0.5 ml / min
Injection volume	10 µl
Detection	310 nm
Temperature	25^{0}C
Retention time	4.610 min

(VP Series) Pump, variable wavelength PDA detector and Hamilton syringe (20 µL).

Chromatographic analysis was performed using empower Column used in HPLC is ODS 2 C18, 250 X 4.6mm, 5µ ((isocratic). The mobile phase consisting of Methanol. The optimized chromatographic conditions are summarized in Table.1. The standard solution of Tinidazole was prepared by dissolving100mg of Tinidazole in 100ml of diluent and subsequent dilutions were made with methanol to obtain working standard of 100µg/ml. The mobile phase and the drug solution were soinicated for 10min and filtered using micropore filter paper of 0.45µ size. The various dilutions of Tinidazole in the concentration of 5-25µg/ml were prepared. The solutions were injected using a 10 µl fixed loop in to the chromatographic system at the flow rate of 0.5ml/min and the effluents were monitored at 310 nm, chromatograms were recorded. The Tinidazole was eluted at 4.610min as shown in Fig: 2 the method was extended for determination of Tinidazole in pharmaceutical dosage form. The pharmaceutical dosage form containing 500mg strength was taken.

20 tablets of Tinidazole (containing 500 mg) were weighed and powdered in glass mortar and the powder equivalent to 100mg of Tinidazole was transferred into 100ml volumetric flask and diluent was used to make up the volume to 100ml. Further dilutions were made with methanol to obtain working standard of 100µg/ml. Flask was soinicated for 10 min and the solution was filtered using micropore filter paper of 0.45µ size. From this solution various dilutions were made with the diluent, which were analysed. The concentration of the drug in tablet sample solution was calculated by comparing with peak area of standard. The proposed method was validated as per the ICH guidelines.

RESULTS AND DISCUSSION:

A suitability test was applied to representative chromatograms for various parameters. The results obtained were within acceptable limits (Table 2). Thus, the system meets suitable criteria. The calibration curve was obtained for a series of concentration in the range of 5-25 µg/ml and it was found to be linear. The precision was measured in terms of repeatability, which was determined by sufficient

Table 2: System Suitability Test Parameters for the proposed method

Parameters	Values	Required limits
Retention time	4.610	RSD $\leq$ 1%
Theoretical plates	3778	N > 2000
Tailing factor	1.16	T $\leq$ 2

Table 3: Summary of Validation Parameters for the proposed method

Parameters	Values
Calibration range (µg/ml)	5-25
Correlation Coefficient(r 2)	0.999
%Recovery	98.4-101.8
%RSD	$2.42x10^{-4}$

Table 4: Assay Results of Tinadazole tablets using proposed method

Brand used	Labeled amount (mg)	Amount found (mg)	% Recovery
Tablet(500)	500	498.9	99.78

number of aliquots of a homogenous sample. The % RSD was found and lying with in 2. This showed that the precision of the method was satisfactory. The accuracy of the method was inferred by establishing the precision and linearity studies of the standard. The % RSD was less than 2.0. This showed that the recoveries of Tinidazole by the proposed methods are satisfactory. The % RSD values were calculated from precision study was less than 2.0. The results of validation parameters are summarized in Table 3. The results of recovery studies obtained by the proposed method were validated by statistical evaluation and are given in Table 4.

CONCLUSION:

The validation study shows that the developed method is accurate, rapid, precise, reproducible and inexpensive with the acceptable correlation coefficient, RSD (%) and standard deviation which make it versatile and valuable. The advantages lie in the simplicity of sample preparation and the low costs of reagents used. The proposed method is simple and do not involve laborious time-consuming sample preparation the method was found to have a suitable application in routine laboratory analysis with a high degree of accuracy and precision.

REFERENCES:

1. Massart DL, Vandeginste BGM, Deming SN, Michotte Y, Kaufman L, Chemorenetrics Text Book, Amsterdam: Elsevier; 1998.
2. Willard HH, Merrit LL, Dean JA, Settle FA, High performance liquid chromatography; Methods and application. Instrumental methods of analysis.7th ed.New Delhi: CBS publishers and distributors: 2001.p.614-629.
3. Chatwal GR, Shama KA. High performance liquid chromatography; Instrumental methods of chemical analysis. 5th ed. Mumbai: Himalaya Publishing house; 2002, p.2.626-2.636
4. Weston A, Brown PR. High performance liquid chromatography; Instrumentation for HPLC and CE-Principles and practices. USA: Academic Press; 1997.p.1-71.
5. Munson JW. Pharmaceutical Analysis Modern Methods. Mumbai: International Medical Book Distributors; 2001.p-51-54.
6. Beckett A.H.,Stenlake J.B. Practical Pharmaceutical Chemistry. New Delhi: CBS Publishers and distributors; 2002.p.157.
7. Chatwal, G.S. Anand, instrumental of chemical Analysis. New Delhi: Goel Publishers; 2003 p.625.
8. Janson SO and Jhonson S, j.chromatography:1982p.241-42.

9. Helfmann E.ed. chromatography, 5th ed. Amsterdan:Elsevier;1992.
10. Poole CF and Poole SK., Chromatography. Today Amsterdan:Elsevier; 1991.

