



REVERSE PHASE – HPLC METHOD FOR THE ANALYSIS OF TINIDAZOLE IN PHARMACEUTICAL DOSAGE FORM & BULK DRUG

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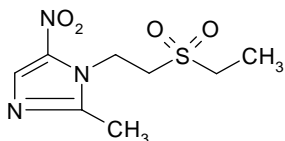
ABSTRACT

A simple rapid and reproducible high performance reverse phase liquid chromatographic method has been developed for the estimation of Tinidazole in bulk drug sample and pharmaceutical dosage form was developed using ss wakoil II. C18, 250 x 4.6mm, 5µm column with mobile phase composition of acetonitrile and phosphate buffer 3:1 (PH 5), flow rate of 1.0 ml/min and UV detection at 295nm linearity was observed over concentration range of 10-80mcg/ml. The accuracy of the proposed method was determined by recovery studies and found to be 101-103% the proposed method was validated and results conformed with ICH parameters.

Keywords: Tinidazole, RP - HPLC.

INTRODUCTION

Tinidazole is chemically 1-(2- Ethyl Sulfonyl Ethyl) -2 - methyl - 5- nitroimidazole



Which is used for the treatment of abdominal abscess & brain abscess. A tablet formulation contain 500mg of Tinidazole is available (Fasigyn, Pfizer Pharmaceutical India Pvt. Ltd)

A literature revealed that few methods are available like LC method for the estimation of Tinidazole in human plasma and its pharmacokinetic 2-4 spectrophotometry^{11,12}. The aim of this study is to develop a simple, rapid, precise and accurate reverse phase HPLC method for the determination of Tinidazole in bulk drug samples or in pharmaceutical dosage form.

EXPERIMENTAL

Instrumentation:

Quantitative HPLC was performed on a gradient high pressure liquid chromatograph (Shimadzu HPLC class-VP Series) with two LC-10AT VP Pumps, variable wavelength programmable UV/Vis detector SPD-10A VP system controller (Shimadzu), a disposable guard column LC-18 (Pelliguard)™, LC-18, 2cm, SS wakoil II RPC-18 column (250x4.6mm, ID particle size 5µm) was used. The HPLC system was equipped with the software class-VP series version 6.01 (Shimadzu). The flow rate of mobile phase was maintained at 1ml/min and detection was carried at 295nm at room temperature.

Chemicals and Reagents:

Water of HPLC grade was collected from a milli-Q system potassium dihydrogen phosphate AR (Ranbaxy) and ortho phosphoric acid AR (Ranbaxy) mobile phase were purchased from the market.

Preparation of Mobile Phase:

A mixture of acetonitrile and 0.02 M potassium dihydrogen phosphate buffer (adjusted to pH 5.0 using orthophosphoric acid) in the ratio of 75 : 25 v/v was filtered through 0.45 µ membrane filter and then used as mobile phase and sonicated for 10 min.

Preparation of Standard Solution:

Standard stock solution of Tinidazole was prepared in mobile phase of concentration 500 µg/ mL. The stock solutions were diluted to obtain working standard solution of concentration of 10µg/ML to 50

µg/ mL. The resulting solutions were sonicated for 10 min was 100 µL was injected. The retention time for Tinidazole was found to be 3.05 min. The Linearity range for Tinidazole was found to be 10-80 µg/mL.

Preparation of Sample Solution:

Fasigyn, tablets five in number were weighted. An amount equivalent of 5mg of Tinidazole was transferred into 10mL volumetric flask. The powder was first dissolved with a few drops of mobile phase and the volume then made upto 10 mL with mobile phase. The solution was filtered through membrane filter with pore size of 0.45 micron. The sample stock solution was adequately dilute to obtain Tinidazole concentration of 10 µg/mL. The resulting solution was sonicated for 10 min and 100 µL of the sample was injected. The peak area from the chromatogram was tabulated and the amount of Tinidazole present in the tablet formulation was determined from the linearity curve.

Table 1: Recovery studies

Drug	Amount Added	Amount Recovered	Average Recovery %
Tinidazole	30	15.24	101.6
	40	20.52	102.6
	50	25.93	103.7

Table 2: System suitability parameter

Parameter	Tinidazole
Theoretical Plates	10647
Tailing Factor	0.3
Resolution	0.872
Calibration Range	10-80µg/mL

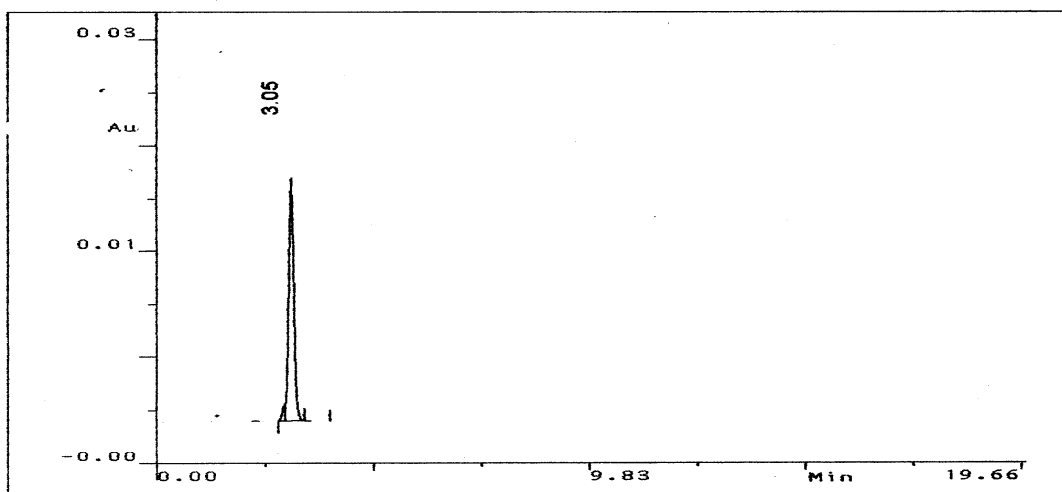
The proposed method was validated as per ICH parameters. Precision of the proposed HPLC method was carried out by injecting replicate of six of concentration 10 µg/mL and the precision of the proposed HPLC method was found to be 1.58% for Tinidazole. The low RSD values indicate that the proposed method had good precision. The precision of instrument was carried out by injecting replicate of six of concentration 10 µg/mL, which was found to be 0.3% RSD for Tinidazole. Accuracy of the method was also determined. The average recovery of Tinidazole were 101-103%, respectively. The sample recovery in the formulation was in good agreement with the label claim. High percentage recovery showed that the method was free from interferences of the excipients used in the formulations. Ruggedness of the method was determined by carrying out the assay by different analysts on different days. The test results were found to be satisfactory with

RSD for set of analysis on the same date being less than 0.8% and RSD between set of analysis on different days being less than 1.6% for Tinidazole. The percentage area on calculation was found to be 99-101% for Tinidazole. This shows that the results are reproducible. Robustness of the method was determined by carrying out the assay during which the mobile phase ratio and pH of mobile phase were

altered slightly. The percentage recovery found to be 72-102% for Tinidazole when mobile phase was altered slightly. System suitability parameters of Tinidazole are given in Table 2. Assay of the Tinidazole in tablet dosage form was found to be 105.7% of Tinidazole.

Sample Name: pipeMet100 Data File: ...OMNKS_06.DAT
 Method File: C:\WINDOWS\WINCHROM\PASHAN\NEW\MET
 Detector: UV-VIS System: HPLC
 Date: 09 Jan 2005 Time: 12:33:46
 Run: ch1: 7
 Type of Analysis : Percent On Area and Height

Pk.Width 4 Peak Thresh. 30 Area Rej. 5 H.Rej. 4



No.	Retn. Time	Height	Area	Height %	Area %	Peak type
1	2.89	267	33073	5.9987	3.6055	BV
2	3.05	4184	884232	94.0014	96.3945	VB
		4e+03	917305			

CHROMATOGRAM OF SAMPLE SOLUTION

CONCLUSIONS

The method was simple and had short runtime of 3.05 min, which makes the method rapid. The results of the study indicate that the proposed HPLC method was simple, precise, highly accurate, specific and less time consuming.

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