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ORIGINAL RESEARCH ARTICLE

A Validated UV-Spectrophotometric Method for the Estimation of Tinidazole in Bulk and Pharmaceutical Dosage Form

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ABSTRACT:

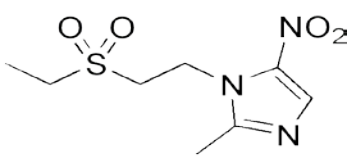
A new, simple, precise and accurate method for the estimation of Tinidazole in bulk and pharmaceutical dosage forms has been developed. 0.1N HCl was chosen as the solvent system. The λmax was found to be 278nm. The responses were linear in the range of 10-80µg/ml. The regression equation of the calibration graph and correlation coefficient were found to be y = 0.026x - 0.042 and 0.999 respectively. Validation of the method was done in order to demonstrate accuracy, precision, interday and intraday assay, robustness and ruggedness of the proposed method. The %RSD values for both intraday and interday precision were less than 1%. The recovery of the drug from the sample was ranged between 99.12% and 100.96%. Commercial tablets containing 500mg and 300mg of Tinidazole (COZIT and TINA respectively) were analyzed by the proposed method and the results were well within the claimed limits.

Keywords: Tinidazole, Intraday Assay, Robustness, Validation

INTRODUCTION:

Tinidazole (**Fig.1**) is an antiparasitic drug belonging to the family of nitroimidazoles with potent activity against protozoans. Chemically, it is 1-[2-(ethyl sulphonyl) ethyl] – 2- methyl – 5-nitro – 1H- imidazole. Literature surveys reveal different spectrophotometric methods[1-3] and HPLC[4-8] for its determination. There was no simple method for estimation of Tinidazole, so a new, simple, accurate and validated method for determination of Tinidazole was developed by UV spectrophotometric method.

Fig 1: Chemical structure of Tinidazole



MATERIALS AND METHODS

Chemicals & Reagents: Analytically pure Tinidazole was obtained as a gift sample from Emcure Pharmaceuticals Ltd., Pune (India). Commercial tablet formulations were purchased from the local market. All chemicals and reagents used were of Analytical Grade, obtained from Merck.

Instrument: A SHIMADZU double beam UV/Visible recording spectrophotometer (Model: 1700) with 2 nm spectral bandwidth was employed for all spectrophotometric measurements using 10mm matched quartz cells and Borosil glass wares were used for the study. Calibrated electronic single pan balances Sartorius CP 225 D, pH Meter (LABINDIA), Enertech Fast Clean Ultrasonicator were also used during the analysis.

Standard Stock Solution and Working Standard Solutions: The standard stock solution of Tinidazole was prepared by transferring accurately weighed 10 mg of drug to 10 ml volumetric flask and dissolving it with 0.1N HCl to get a concentration of 1000 µg/ml. The solution was diluted accordingly to get a concentration of 100µg/ml and was kept as the stock solution. The prepared stock solution was diluted with 0.1N HCl solution to get working standard solutions of concentrations 10-80 µg/ml.

Determination of λmax: The standard solution of Tinidazole (10 µg/ml) was scanned in the wavelength region of 200-400 nm and the λmax was found to be 278 nm. (**Fig.2**)

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Preparation of calibration curve: The working standard solutions of Tinidazole were scanned in the UV region and the absorbances were observed against 0.1N HCl solution as blank at 278nm. Finally the calibration curve was plotted between concentration (x-axis) and absorbance (y-axis).

Assay of tablet dosage form: 10 tablets of brand TINA (manufactured by Bombay Tablet Mfg. Co, Mumbai) containing 300mg of Tinidazole were weighed, average weight determined and finely crushed to powder. An accurate weight of powder equivalent to 10mg of the drug was transferred to 100ml volumetric flask. The drug was extracted 4 times by adding solvent in potions, 20 ml each time and the volume was made upto the mark by using solvent. It was then diluted (within the linearity range), absorbances of the sample solution were recorded at determined λmax and the concentration of the drug in sample was found out. Similarly, the assay of COZIT (manufactured by Emcure Pharmaceuticals Ltd, Pune) containing 500mg of Tinidazole was carried out.

VALIDATION

Accuracy: The accuracy of the proposed method was tested by recovery studies at 80%, 100%, and 120% by adding a known amount of pure drug to the pre-analyzed formulation of concentration 10µg/ml.

Precision: The precision of the proposed method was ascertained by actual determination of 6 replicates of a fixed concentration of the drug (40µg/ml) within the Beer's range and finding out the absorbance by the proposed method.

Intraday Assay: The intraday assay of the proposed method was ascertained by actual determination of 6 replicates of a fixed concentration of the drug (40µg/ml) within the Beer's range and finding out the absorbance by the proposed method at 3 different time period of the same day.

Interday Assay: The interday assay of the proposed method was ascertained by actual determination of 6 replicates of a fixed concentration of the drug (40µg/ml) within the Beer's range and finding out the absorbance by the proposed method on 3 different days.

Robustness: The robustness of the method was carried out by changing the solvent system to Glacial Acetic acid.

Ruggedness: In order to determine the ruggedness of the proposed method, the method was carried out simultaneously by two analysts.

RESULTS AND DISCUSSION

The regression equation of the calibration curve was found to be y=0.026x-0.0042. The calibration curve is shown in (**Fig 3**) and represented in (**Table 1**)**.** The assay results of the commercial formulations are shown in (**Table 2**).

The method was found to be accurate and precise which was evident from its low %RSD values.(**Table 3 & 4**). Similarly the %RSD for Intraday and Interday Assay were found to be 0.00745 and 0.0116 respectively.(**Table 5 & 6**). The %RSD for Robustness was found to be 0.0644 and 0.0312 for the proposed method by taking 0.1N HCl solution and glacial acetic acid respectively (**Table 7**) while the %RSD for Ruggedness was found to be 0.0261 and 0.0425 respectively when performed by two analysts separately. (**Table 8**). The limit of detection and limit of quantification were found to be 0.51mg and 1.553mg respectively

Fig 2: Overlay Spectra of Tinidazole showing λmax at 278nm

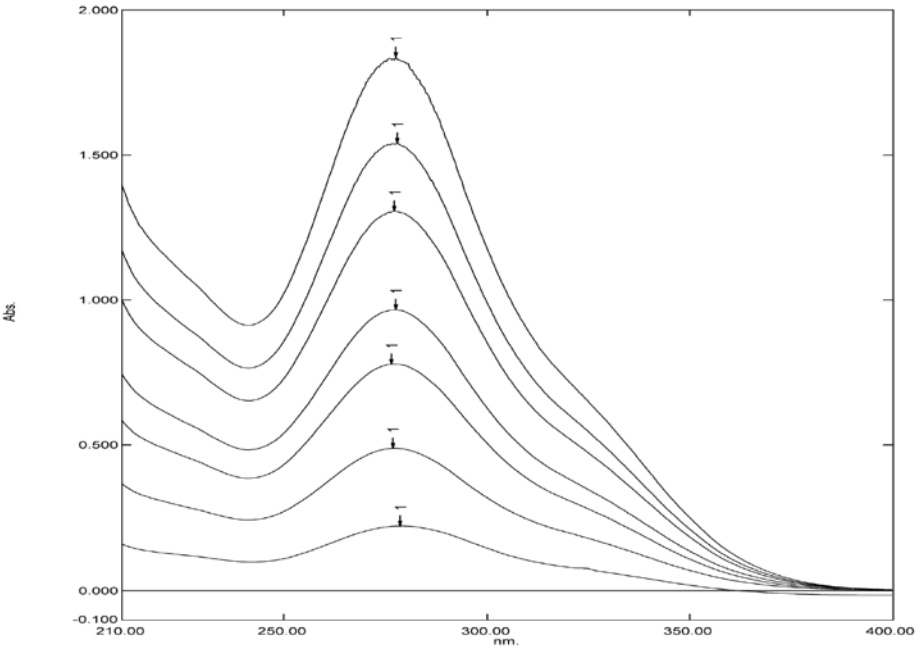

Fig 3: Calibration curve of Tinidazole at 278nm

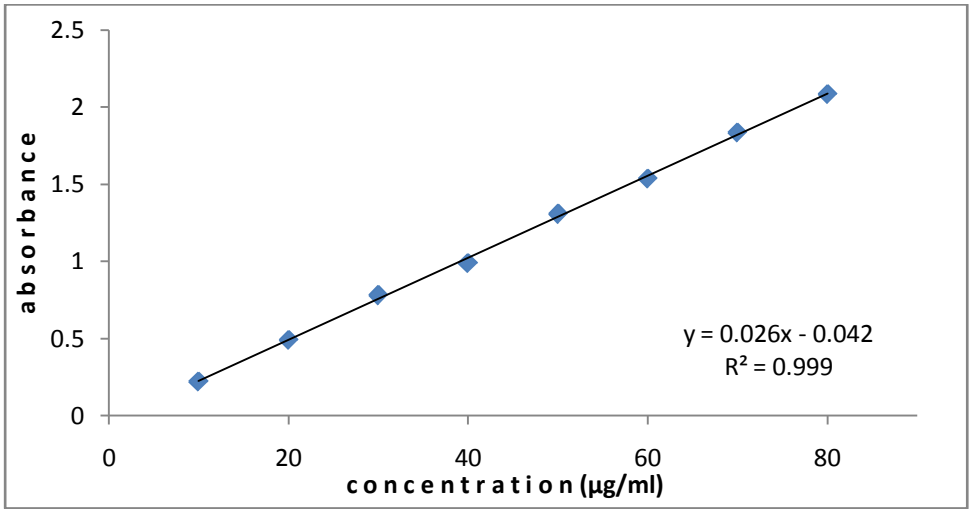

Table 1: Linearity table of Tinidazole in 0.1N HCl solution

Concentration (µg/ml)	Absorbance
10	0.222
20	0.490
30	0.780
40	0.988
50	1.307
60	1.539
70	1.836
80	2.084

Table 2: Assay results of the marketed formulations

Formulation	Label Claimed(mg)	Observed amount(mg)	Assay Result (%)
COZIT (Emcure Pharmaceuticals Ltd.)	500	491.68	98. 33
TINA (Bombay Tablet Mfg. Co.)	300	279.058	99.01

Table 3: Statistical analysis for ACCURACY of the proposed method

Samples	Concentration (µg/ml)		%Recovery	Statistical Analysis
	Pure	Formulation		
S1: 80%	8	40	99.12	Mean: 099.12 S.D: 0.1511 %RSD: 0.1224
S1: 80%	8	40	99.27	
S1: 80%	8	40	98.97	
S2: 100%	10	40	101.21	Mean: 100.88 S.D: 0.3156 %RSD: 0.2577
S2: 100%	10	40	100.58	
S2: 100%	10	40	100.86	
S3: 120%	12	40	100.12	Mean: 100.96 S.D: 0.8350 %RSD: 0.6818
S3: 120%	12	40	101.79	
S3: 120%	12	40	100.97	

Table 4: Statistical analysis for PRECISION of the proposed method

Concentration (µg/ml)	Absorbance	Amount Present	Statistical Analysis
40	1.086	40.153	Mean:40.119
40	1.086	40.153	

40	1.082	40.001	S.D:0.0659
40	1.087	40.192	%RSD:0.0616
40	1.086	40.153	
40	1.085	40.115	
40	1.086	40.153	
40	1.083	40.038	

Table 5: Statistical analysis for INTRADAY ASSAY of the proposed method

S. No	Concentration (µg/ml)	Absorbance			Statistical Analysis
		Sampling 1	Sampling 2	Sampling 3	
01	40	1.086	1.087	1.085	Mean:40.1538 S.D:0.0767 %RSD:0.0745
02	40	1.087	1.086	1.086	
03	40	1.086	1.086	1.085	
04	40	1.087	1.087	1.086	
05	40	1.087	1.085	1.086	
06	40	1.085	1.086	1.085	

Table 6: Statistical analysis for INTERDAY ASSAY of the proposed method

S. No	Concentration (µg/ml)	Absorbance			Statistical Analysis
		DAY 1	DAY 2	DAY 3	
01	40	1.082	1.085	1.083	Mean: 40.0384 S.D: 0.0188 %RSD: 0.0116
02	40	1.082	1.084	1.083	
03	40	1.085	1.085	1.085	
04	40	1.082	1.084	1.083	
05	40	1.083	1.082	1.083	
06	40	1.084	1.083	1.082	

Table 7: Statistical analysis for ROBUSTNESS of the proposed method

0.1N HCl				Glacial Acetic Acid			
Conc. (µg/ml)	Abs	Calculated amount (mg)	Statistical Analysis	Conc. (µg/ml)	Abs	Calculated amount (mg)	Statistical Analysis
40	1.085	40.1153	Mean:40.185 S.D: 0.0705 %RSD:0.0644	40	1.089	40.2692	Mean:40.3074 S.D: 0.0343 %RSD: 0.031
40	1.089	40.2692		40	1.091	40.346	
40	1.087	40.1923		40	1.089	40.2692	
40	1.086	40.1538		40	1.091	40.346	
40	1.089	40.2692		40	1.090	40.307	
40	1.085	40.1153		40	1.090	40.307	

Table 8: Statistical analysis for RUGGEDNESS of the proposed method

ANALYST-I				ANALYST-II			
Conc. (µg/ml)	Abs	Calculated amount (mg)	Statistical Analysis	Conc. (µg/ml)	Abs	Calculated amount (mg)	Statistical Analysis
40	1.086	40.1538	Mean:40.160 S.D: 0.0289 %RSD: 0.026	40	1.088	40.2307	Mean:40.1794 S.D: 0.0465 %RSD: 0.0425
40	1.086	40.1538		40	1.088	40.2307	
40	1.087	40.1923		40	1.086	40.1538	
40	1.087	40.1923		40	1.085	40.1153	
40	1.085	40.1153		40	1.087	40.1923	
40	1.086	40.1538		40	1.086	40.1538	

CONCLUSION

The proposed method was found to be simple, sensitive, precise and rapid for the determination of Tinidazole from pure and its dosage forms. The sample recoveries in all formulations were in good agreement with their respective label claims without interference of excipient and the other additives. Thus the proposed method can be used as an alternative method to the reported ones for the routine analysis of the drug in bulk and

pharmaceutical dosage forms and can also be used for dissolution or similar studies.

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