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Spectrophotometric Method for Estimation of Atenolol in Tablet Dosage Form

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A simple and accurate spectrophotometric method has been developed for the quantitative estimation of atenolol in bulk and tablets. The method is based upon the reaction of atenolol with 1-chloro-2,4-dinitrobenzene, forming a complex, which is spectrophotometrically analyzed. The absorbance maxima (λ_{max}) was found to be 224.6 nm. Optimization of reaction was carried out with the factors: temperature, time, stability of complex, molar ratio of drug : reagent. The proposed method was validated as per ICH guidelines. The recovery studies confirmed the accuracy and precision of the method.

Key Words: Atenolol, Spectrophotometry, 1-Chloro-2,4dinitrobenzene, Reagent, Validation.

INTRODUCTION

Chemically atenolol is 4-[2-hydroxy-3-[(1-methylethyl) amino] propoxy] benzenacetamide. Atenolol is a cardioselective β-adrenergic blocker, mainly used in hypertension, angina pectoris and myocardial infarction. Atenolol is official in I.P.[1] (1996), U.S.P.[2] (2003) and B.P.[3] (1993). The U.S.P. (2003) recommends HPLC method for pure and dosage forms. The B.P. (1993) recommends potentiometric titration method for analysis of atenolol in bulk and spectrophotometric method for dosage forms. Literature survey reveals that numerous methods are available for the estimation of atenolol[4–10]. This paper presents a simple, accurate, sensitive, reproducible and economic method for the determination of atenolol in bulk and tablet form.

EXPERIMENTAL

The pure drug sample was obtained from ONS Pharmaceuticals, Jaipur. 1-Chloro-2,4-dinitrobenzene (AR grade) and methanol (spectroscopic grade) were obtained from Loba Chemie. The instruments include UV-Vis spectrophotometer (Elico SL 160) and FTIR-8300 (Shimadzu).

Procedure for bulk drug

Accurately weighed 100 mg of atenolol pure drug was dissolved in methanol to give a stock solution of 1000 g/mL concentration. From this stock solution, working standard solutions of drug (4–20 g/mL) were prepared by appropriate dilutions. Working standard solutions were scanned in the entire UV-Vis range

(200–800 nm). Standard solutions were prepared having concentrations 4, 6, 8, 10, 12, 14, 16, 18 and 20 μg/mL. In these solutions, 1 mL of 0.01% w/v 1-chloro-2,4-dinitrobenzene solution was added and kept for 70 min to complete the reaction. The absorbance maxima (λ_{max}) of atenolol : reagent complex was found to be 224.6 nm. The absorbances of these samples were measured at 224.6 nm and calibration curve was plotted (Fig. 1). The absorptivity coefficient was determined using calibration curve.

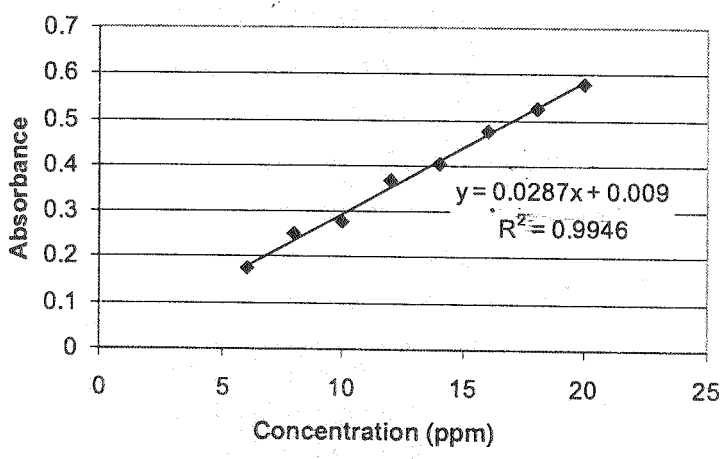


Fig. 1. Calibration curve of atenolol : reagent complex

Procedure for tablet formulations

Twenty tablets were weighed and ground to fine powder. An accurately weighed powder equivalent to 10 mg of atenolol was transferred into a 100 mL volumetric flask. The powder was dissolved in 60 mL methanol and the resulting solution was heated to 60°C and shaken for 15 min. After cooling, this was diluted to 100 mL with methanol and filtered through a sintered glass funnel (Borosil G3). From this stock solution, working sample solutions were prepared by appropriate dilutions and analyzed by developed method. The results of analysis of tablet formulations are recorded in Table-1. Recovery studies (Table-2) carried out gave satisfactory results. The optical characteristics and regression equation is given in Table-3.

TABLE-1
RESULTS OF ASSAY

S.No.	Formulation	Label claim (mg/tab)	Estimated (mg/tab)*	Found (%)	RSD
1	X	50	49.80	99.6	
2	Y	50	49.90	99.8	0.6822
3	Z	50	49.85	99.7	

*Average of 3 measurements.

TABLE-2
RECOVERY DATA

S. No.	Drug added (mg)	Drug found (mg)	Recovery (%)	RSD
1.	10	9.8	98.00	
2.	15	14.8	98.67	0.7684
3.	20	19.69	8.00	
4.	25	24.99	9.60	

TABLE-3
OPTICAL CHARACTERISTICS AND PRECISION

S. No.	Parameters	Values
1.	Beer's law limit (μg/mL)	6–20
2.	Sandell's sensitivity (mg/cm/0.001 abs. unit)	0.0357
3.	Stability of coloured species (min.)	20
4.	Molar extinction coefficient (L mol^{-1} cm^{-1})	7.458×10^3
5.	% Relative standard deviation	0.6822
6.	Correlation coefficient	0.9946
7.	Regression equation (Y*)	
8.	Slope (a)	0.009
9.	Intercept (b)	2.87×10^{-2}

Y* = b + ac where C is concentration in g/mL and y is absorbance unit.

RESULTS AND DISCUSSION

The proposed method has been found to be accurate, simple and economical for routine analysis of atenolol.

Optimization of the method was carried out with the following parameters:

1. Temperature: The reaction occurred at room temperature.

2. Reaction time: Optimization of reaction time was done by measuring the absorbance at intervals of 5 min up to 120 min. The optimum reaction time was found to be 70 to 90 min. A minimum of 70 min was found to be sufficient to complete the reaction.

3. Identification of complex: Identification of complex was done by FTIR.

4. Stability of complex: Stability of complex was observed up to 120 min and it remained stable for 20 min.

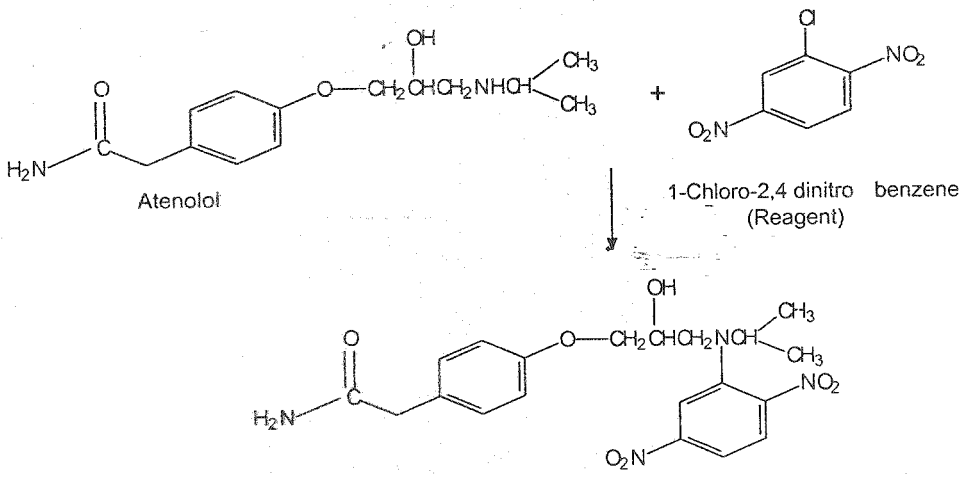
5. Molar ratio of drug : reagent: The molar ratio of drug : reagent was found to be 1 : 1.

All method validation parameters of ICH guidelines were applied. The molar absorptivity and Sandell's sensitivity values show the sensitivity of the method while precision is confirmed by % relative standard deviation. Results of assay and recovery studies are given in Tables 1 and 2.

The results are in good agreement with labelled value. The reproducibility, repeatability and accuracy of this method have been found to be good, which is confirmed by low relative standard deviation value.

The proposed method can be successfully applied for the estimation of atenolol in tablets. The mechanism of formation of complex is given as below.

Mechanism of reaction



Formation of atenolol : reagent complex

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