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Development of Simple Spectrophotometric Method for the Determination of Propranolol using p-Chloranilic acid in Bulk and Dosage forms

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ABSTRACT

A new spectrophotometric method for the determination of propranolol in bulk and in tablet forms using p-Chloranilic acid (PCA) has been developed which is based on reaction between the drug and PCA to form charge transfer complex in acetonitrile. The formed reddish pink complex was measured at 526 nm. The development of colored complex is due to the formation PCA anions which arised due to the complete transfer of n- electrons from donor (D) to Acceptor (A) moieties in a medium of acetonitrile. Beer's law is obeyed in the concentration range of 8-14 µg/ml. The proposed method was found to be simple, accurate, sensitive and selective compared to the existing methods for the determination of propranolol.

Keywords: Propranolol, p-Chloranilic acid (PCA), charge transfer complex, Acetonitrile, Beer's law.

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INTRODUCTION

Propranolol (1-isopropylamino-3-(1-naphthoxy)-2-propranolol), is a beta adrenergic blocking drug which is extensively used in the treatment of cardio arrhythmia, sinus tachycardia, angina pectoris, hypertension and several other cardio vascular disorders. It is used to control numerous conditions like dysfunction and anxiety [1]. In low activity sports, propranolol is used to reduce the cardiac frequency, force of contraction and coronary flow. Hence, it has been added in the forbidden list of substances by the international Olympic Committee. The amine functional group is responsible for the complex formation with p-Chloranilic acid (PCA) in the present method. Propranolol was determined by various analytical methods such as spectrophotometric [2, 3], Fluorimetric [4], HPLC [5], atomic absorption [6, 7], thin layer chromatography [8] and gas chromatography [9]. In the present study, a simple, sensitive, low-cost, accurate spectrophotometric method for the determination of propranolol in bulk and dosage forms using PCA has been developed.

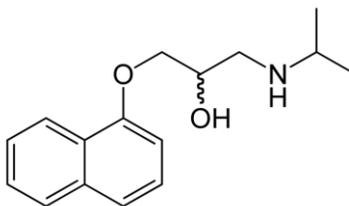


Fig. 1 Structure of propranolol

EXPERIMENTAL

Instrumentation

Shimadzu UV-Visible double beam spectrophotometer (model 2450) with 1cm matched quartz cells was used for the spectral measurements.

Chemicals and Reagents

Chloroform, Acetonitrile, methanol 1, 4-dioxane and PCA were procured from Merck. All the chemicals are of analytical grade.

Preparation of standard solutions

10 mg of propranolol was dissolved in 10ml of acetonitrile and a stock solution of 1000 µg/ml was obtained and from this solution 0.5 ml was pipetted out to a 10 ml volumetric flask and made upto the mark with distilled water to get the concentration of 50 µg/ml. To this solution further dilutions are made to get the concentrations of 6 – 24 µg/ml.

Procedure

A series of clean and dry 10 ml volumetric flasks are taken and to each flask, propranolol solution which ranges from 0.8 – 1.4 ml (8 – 14 µg/ml) is added and followed by the addition of 1 ml of 4.2×10^{-3} mol/dm⁻³ PCA solution using a micro burette. Immediately after the addition of reagent a reddish pink colour was obtained and the absorbance was recorded at 526 nm against the blank. A calibration curve was plotted from the amount of drug present in the given drug solution.

RESULTS AND DISCUSSION

The absorption spectrum shows λ_{\max} at 526 nm for the drug propranolol with PCA. The calibration curve was obtained from the series of concentrations ranging from 6.5-24.5 µg/ml. The optical characteristics such as Beer's law limit, molar absorptivity, Sandell's sensitivity and the regression analysis is made for slope, intercept and correlation coefficient and the results are presented in table 1.

Table 1 Optical characteristics of proposed method

Parameter	Value
λ_{\max} (nm)	526
Beer's law limit (µg/ml)	6.5-24.5
Molar absorbance (L.mol ⁻¹ cm ⁻¹)	0.52
Sandells sensitivity (µg.cm ⁻² /0.001 A.U)	0.001923
Correlation coefficient (r ²)	0.999739
Slope (m)	0.021689
Intercept (c)	0.00604
%RSD	0.192308
Colour	pink
LOD	0.138317
LOQ	0.460594

Method validation

For the quantitative analysis of the drug propranolol, the method was validated according to ICH guidelines and the following characters of validation are addressed; Linearity, Accuracy, Precision, Specificity, LOD, LOQ and Robustness.

Standard calibration curve with PCA was constructed by plotting absorbance versus concentration. The statistical parameters were given in the regression equation calculated from calibration plots. The linearity of calibration graphs are proved by high values of correlation coefficient. The molar absorptivities of the colored complexes and relative standard deviation for the proposed spectrophotometric method were also calculated and shown in table 2.

Table 2 Determination of propranolol in dosage forms

Pharmaceutical formulation	Amount added (mg)	*Amount found (mg)	% recovery	±SD	RSD
Ciplar	5	4.93	99.13	0.025	0.507
Betacap	10	9.95	99.50	0.021	0.201

*Average of five determinations

Assay of pharmaceuticals

For the determination of the drug propranolol in tablet formulations, the contents of 3 tablets were weighed and finely powdered, a portion of powder equivalent to 100 mg of the drug was taken into 100 ml standard flask and dissolved with small portion of methanol and made up to the mark with the same solvent. The contents in the flask are filtered on whatmann No.41 filter paper and washed well with methanol for the complete recovery of the drug. The resulting concentration of the solution was found to be 1 mg/ml. This solution is considered as stock solution and the required aliquots were taken from the solution for the determination of the drug propranolol by the proposed method.

CONCLUSIONS

In the proposed analytical method, the drug propranolol was analysed in both bulk and in tablet form. The linearity of the calibration standards of the drug by proposed method was good from the result of correlation coefficient. The proposed analytical method is free from interferences due to the excipients and other impurities present in the tablet forms. This indicates that the proposed method is accurate than the existing methods for the estimation of the drug propranolol in tablets and biological fluids.

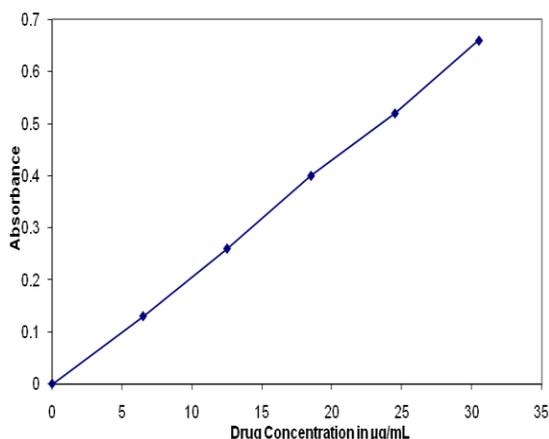


Fig. 2 Calibration curve of Propranolol with PCA

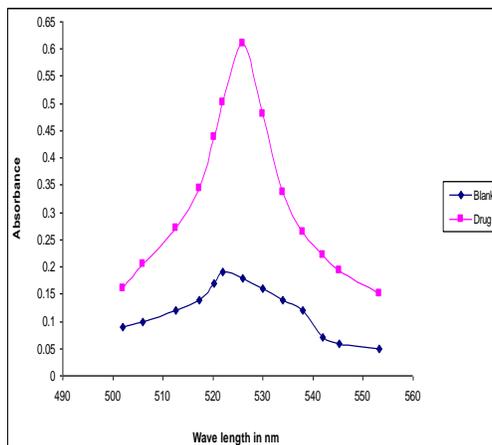


Fig. 3 Absorbance spectrum of Propranolol with PCA

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