ISSN: 0975-8585

Research Journal of Pharmaceutical, Biological and Chemical Sciences

Development of Simple Spectrophotometric Method for the Determination of Propranolol in Bulk and Dosage Forms.

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ABSTRACT

A simple, sensitive and accurate spectrophotometric method has been developed for the determination of propranolol in bulk and in tablet forms. The proposed method is based on the charge transfer reaction between the drug and 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) at room temperature in acetonitrile. When a standard solution of Propranolol solution is mixed with DDQ, a reddish brown colour was observed with a maximum absorbance at 584 nm. This is due to the formation DDQ 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 6-12 µg/ml. The proposed method has been successfully applied for the determination of drug in bulk and dosage forms.

Keywords: UV-Visible double beam spectrophotometer, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ), Propranolol, Acetonitrile, 1,4 dioxane.

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ISSN: 0975-8585

INTRODUCTION

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[1,2]. Chemically it is (1-isopropylamino-3-(1-naphthyloxy)-2-propranolol)[3]. 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[4]. It is also used to control numerous conditions like dysfunction and anxiety[5]. The structure of Propranolol has an aromatic naphthyloxy group which is attached to a side alkyl chain with a secondary hydroxyl and amine functional group. This amine functional group is responsible for the complex formation with reagents used in the proposed method. Propranolol was determined by various analytical methods such as spectrophotometric[6-8], flourimetric[9], chemiluminescence[10], HPLC[11,12], capillary electrophoresis[13] methods in single dosage forms. In the present investigation, spectrophotometric method for the determination of propranolol in bulk and dosage forms using DDQ has been developed.

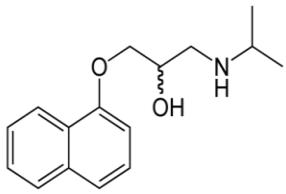

Fig 1: Structure of propranolol

MATERIALS AND METHODS

Instrumentation

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

Chemicals and Reagents

Chloroform, Benzene, 1,4-dioxane and DDQ. All the chemicals are of analytical grade.

Preparation of Sample Solutions

The contents of three tablets of propranolol (label claim 40mg) weighed and finely powdered , a portion of powder equivalent to 100mg of the drug was taken into 100ml 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

ISSN: 0975-8585

required aliquots were taken from the solution for the determination of the drug propranolol by the proposed method.

A series of 10ml volumetric flasks are taken and to them a standard solution propranolol is added which ranges in volume from 0.52 – 2.22ml (6 – 12 µg/ml). To these standard flasks DDQ reagent (SD fine Chem. India Ltd.,) dissolved in 1,4 dioxane was added by adjusting the concentration to $2.2x10^{-3}$ M. The DDQ solution was obtained by the recrystallisation process with chloroform and Benzene. After the addition of reagent immediately a reddish brown colour was observed, which showed a maximum absorbance of 584 nm against the blank. The amount of drug was computed from the calibration graph.

RESULTS AND DICUSSION

The absorption spectrum shows λ_{max} 584nm for the drug propranolol. The calibration curve was obtained from the series of concentrations ranging from 5.2-20.2 µg/ml.The optical characteristics such as Beer'slaw limit, molar absorbtivity , sandell's sensitivity and the regression analysis is made for slope ,intercept and correlation co efficient and the results are presented in table 1.

The selection of the method was verified by monitoring the drug solution in the presence of excipients at the same concentration level which was used in the tablet form.

CONCLUSION

The proposed method for the determination of propranolol is simple, accurate, linear and precise. Hence this method can be applied for the regular analysis of propranolol in bulk and in its pharmaceutical formulations.

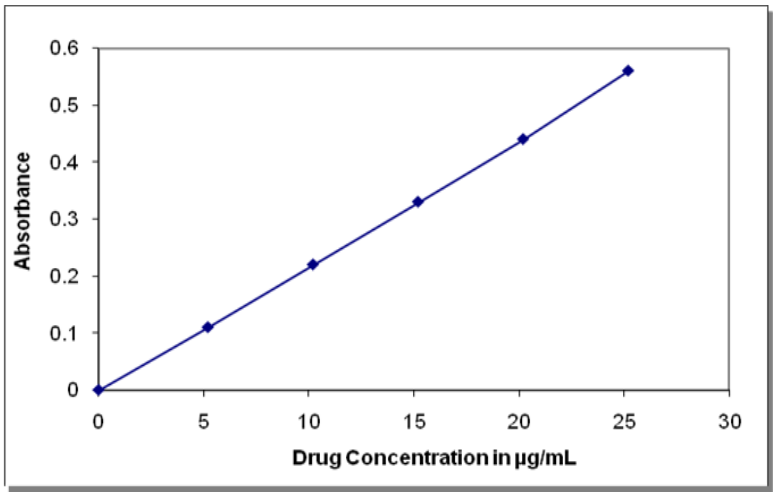

Fig 2: Calibration curve of Propranolol with DDQ

ISSN: 0975-8585

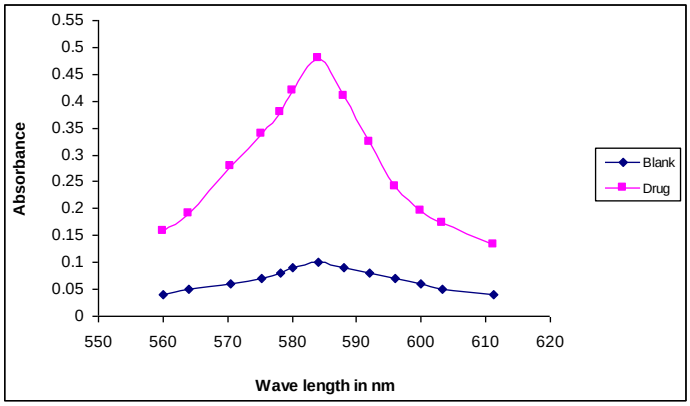

Fig 3: Absorbtion spectrum of Propranolol with DDQ

Table 1: Optical characteristics of proposed method

Parameter	Value
λ_{max} (nm)	584
Beer's law limit (µg/ml)	5.2-20.2
Molar absorbance ($L.mol^{-1}$ cm^{-1})	0.44
Sandells sensitivity ($\mu g.cm^{-2}$/0.001 A.U)	0.002273
Correlation coefficient (r^2)	0.99983
Slope (m)	0.22157
Intercept (c)	0.00399
%RSD	0.2273
Colour	Reddish-brown
LOD	0.135396
LOQ	0.450868

Table 2: Determination of Proponolol in dosage forms

Pharmaceutical formulation	Amount added (mg)	*Amount found (mg)	% recovery	±SD	RSD
Ciplar	10	9.96	99.66	0.015	0.15
Betacap	15	14.97	99.84	0.013	0.08

*Average of five determinations

Table 3: Method accuracy from Recovery assay

Pharmaceutical formulation	Labeled amount(mg)	*Amount found (mg)	% Recovery	± SD	RSD%
Ciplar	40	39.87	99.67	0.08	0.200
Betacap	10	9.94	99.43	0.03	0.003

*Average of five determinations

ISSN: 0975-8585

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