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Development of new and rapid method for UV spectrophotometric determination of Labetalol in marketed formulations

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ABSTRACT

A simple, rapid, and sensitive UV spectrophotometric method has been developed and validated for the determination of Labetalol in pharmaceutical preparations. The method was developed utilizing 0.5N Sodium Hydroxide. The standard and sample was scanned and the absorbance is scanned at 245.3 Linearity was observed in the concentration range from 20-45 µg/ml with a correlation coefficient (R^2) greater than 0.998. The method was validated by following the analytical performance parameters suggested by the International Conference on Harmonization (ICH). All validation parameters were within the acceptable range. Under experimental conditions described, calibration curve, assay of tablets and recovery studies were performed. Parameters of validation prove the precision of the method and its applicability for the determination of Labetalol in pharmaceutical tablet formulations. The method is fast and is suitable for high throughput analysis of the drug.

Key words: Labetolol, Spectrophotometry, Validation parameters, Correlation coefficient

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INTRODUCTION

Labetalol is designated chemically as 2-hydroxy-5-{1-hydroxy-2-[(4-phenylbutan-yl) amino] ethyl} benzamide. It is the first adrenergic antagonist capable of blocking both α and β receptors. It is a moderately potent hypotensive and is especially useful in pheochromocytoma. The drug is used to lower blood pressure in myocardial infarction and unstable angina. Beside these important pharmacological activities, Labetalol therapy exhibits hepatotoxicity and renal failure due to over dosage. Literature survey reveals that the drug can be estimated by various analytical methods which is listed in British Pharmacopoeia [1] and United States Pharmacopoeia [2], recommending non-aqueous titration and HPLC methods for the assay of its content in pharmaceutical formulations. Several analytical methods such as TLC [3, 4], HPLC [5-8], LC-MS [9-13], capillary electrophoresis [14-16], polarography [17], Voltammetry [18], NMR spectroscopy [19] and spectrofluorimetry [20-23] have been reported for the determination of the drug in its pure and commercial dosage forms. In addition, some spectrophotometric methods have also been developed for the quantitation of the labetalol. Labetalol has been determined in bulk and tablets spectrophotometrically [24] based on the reaction of the drug with folin-ciocalteau's reagent. Spectrophotometric methods [25] for the assay of labetalol have been described; two methods are based on the coupling reaction of labetalol with p-N, N-dimethylphenylenediamine and 3-methyl-2-benzothiazoline hydrazone hydrochloride in the presence of sodium hypo chloride and ceric ammonium sulphate as oxidant. BL. Belal and co-workers [27] have developed two spectrophotometric methods based on the phenolic group of the drug. In the first method, labetalol was coupled with diazotized benzocaine in the presence of tri methylamine whereas second method depends on the

coupling of the drug with diazotized *p*-nitro aniline in presence of sodium carbonate. However, some of these methods are tedious and/or time consuming. Therefore, In the present investigation a kinetically based spectrophotometric method is presented with the advantage of simplicity, reliability and less time of analysis 0.5N NaoH is used in spectrophotometric estimation of drug which is safe and inexpensive when compared with existing method with Ethanol.

MATERIALS AND METHODS

Materials and methods

Instruments used

1. Balance
2. Single pan electronic balance- sartorious GE412
3. UV visible spectrophotometer
4. UV visible double beam spectrophotometer
5. Systronics 2203(smart)
6. Matched quartz cells corresponding to 1 cm path length

Reagents

1. 0.5N NaoH
2. Reference standard Labetalol

Tablets brands used

Gravidol-100mg
Labebet-100mg

TRADE NAME	COMPANY NAME	DOSE	BATCH NUMBER	MANUFACTURE DATE	EXPIRY DATE
Gravidol	**Mercury laboratories**	**100mg**	**12104802**	**Apr-2012**	**Mar-2015**
Labebet	**Sun Pharma**	**100mg**	**11503207**	**Apr-2012**	**Mar-2015**

PROCEDURE:

Preparation of standard stock solutions:

The standard stock solution of drug was prepared by dissolving 100mg of the drug in 100 ml standard flask using 0.5N NaoH as a solvent to give a concentration of 1000 µg/ml. These stock solutions on further dilutions are used for establishing following parameters.

Concentration of solvent and Wavelength selection:

Solutions of concentration of 20 µg/ml, 30 µg/ml, 40 µg/ml was prepared.

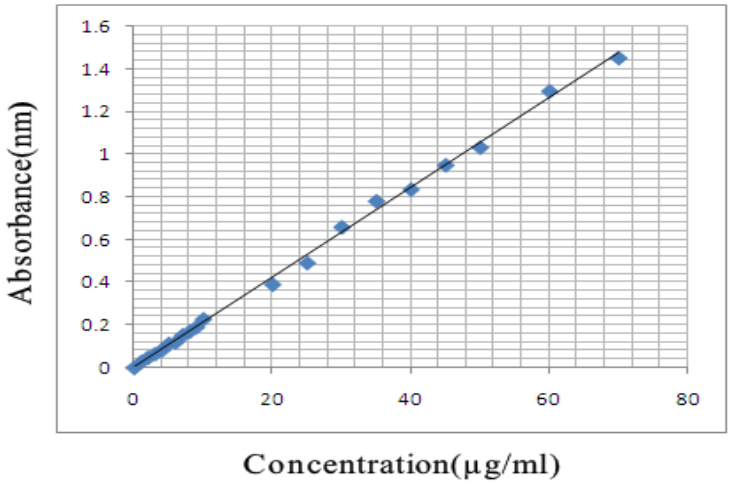
They were subjected to scanning from 200-400nm.

The dilutions were made using different normalities of Sodium Hydroxide namely 0.1N, 0.5N, and 1N.

From the different absorbance values obtained 0.5N NaoH and 245.3nm was selected for the present work.

Beer's law range:

The stock solutions were suitably diluted with water to get concentration range from 1 to 1000 µg/ml. The solutions are scanned in UV regions between 200 to 300nm the absorption were measured at λ_{max} found. Using absorbance values against concentrations plotted the calibration curve and the linearity range can be found.



Analysis of formulation
The proposed method is applied to the analysis of various marketed formulations

RESULTS AND DISCUSSION

1. The UV spectra of Labetalol were presented . the absorption maxima was observed at 245.3nm.Obeyance to beers law was confirmed by the linearity of the calibration curve of Labetalol. Labetalol showed linearity in the concentration range of 20-45µg/ml.
2. The quantitative estimation was carried out in tablet formulations by taking concentrations of 20.0-45.0 µg/ml. The brands of formulations shows the percentage purity values range from 100 to 102 the percentage deviation values were found to be between 0.09 to 0.1.
3. The quantitative results obtained were subjected to stasitical analysis to find out standard deviation and standard error values. The relative standard deviation values are below, indicating the precision of the methodology and low standard error values show the accuracy of the method.
4. The repeatability of the method was confirmed by the assay procedures with 3 different concentrations of 3 replicates each. The results obtained in repeatability test expresses the precision of the given method.
5. The validation of the proposed method was further confirmed by recovery studies. The recovery values vary from 99.54 to 101.27% w/w. This serves as a good index of accuracy and reproducibility of the study.

CONCLUSION

The proposed method of analysis is

- novel,
- simple,
- cost-effective,
- safe,
- accurate and
- Reproducible.

This method can be routinely employed in the analysis of Labetalol in tablet formulations precluding Using 0.5N NaoH as a solvent.

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