IJPPR
INTERNATIONAL JOURNAL OF PHARMACY & PHARMACEUTICAL RESEARCH
An official Publication of Human Journals
ISSN 2349-7203
HUMAN

Human Journals
Research Article
April 2023 Vol.:27, Issue:1
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Development of Novel Colorimetric and FTIR Spectroscopic Methods for the Quantification of Barnidipine Hydrochloride in Active Pharmaceutical Ingredient

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Submitted: 21 March 2023
Accepted: 27 March 2023
Published: 30 April 2023

HUMAN JOURNALS
www.ijppr.humanjournals.com

Keywords: Barnidipine hydrochloride, Colorimetry, FTIR, Bulk, Validation.

ABSTRACT

Two simple sensitive and precise colorimetric methods (method A & method B) and FTIR spectroscopic method were developed for the estimation of barnidipine hydrochloride in bulk drug. Method A is based on the formation of an orange colored complex of barnidipine hydrochloride with 1, 10- phenanthroline in the presence of ferric chloride, which has absorption maximum at 510nm. Method B is based on the formation of a yellow colored complex of barnidipine hydrochloride with isoniazid, which has absorption maximum at 456nm. The FTIR spectroscopy involving the measurement of the area of the infrared band corresponding to the N-H stretching centered at 3325cm^{-1}. The proposed methods are statistically validated and found to be useful for the routine determination of barnidipine hydrochloride in bulk.

INTRODUCTION

Barnidipine hydrochloride (BRN) is used to control blood pressure in patients affected by essential hypertension, hypertension arterial and renovascular hypertension[1,2]. Barnidipine hydrochloride works by blocking the L- type 'voltage- dependent' calcium channels, barnidipine selectively blocks the calcium ion influx in the smooth muscle cells and inhibits the barnidipine displays a high affinity to the inactivated state of the channel. Chemically it is 5-*O*-[(3*S*)-1-benzylpyrrolidin-3-yl] 3-*O*-methyl (4*S*)-2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate;hydrochloride[4,5]. The available methods for analysis of the drug in biological fluids and pharmaceuticals products are RP-HPLC method[5-7], LC-MS[8] method and HPTLC method[9].

The present work deals with the estimation of BRN in active pharmaceutical ingredient by colorimetry and FTIR spectroscopic methods. In the colorimetric method (Method A), BRN is first condensed with 1,10- phenanthroline and ferric chloride to form an orange chromogen, which absorbs intensively at 510nm. In the colorimetric method (Method B), BRN is condensed with isoniazid to form a yellow chromogen, which absorbs intensively at 456nm. FTIR Spectroscopic method is based on the measurement of the area of the infrared band corresponding to the N-H stretching centered at 3325cm^{-1}. The methods are alternative and comparable in specificity and accuracy to chromatography methods, which although highly specific and accurate, are more time consuming, performed in several steps and are rather expensive.

MATERIALS AND METHODS

Instrumentation: All spectral and absorbance measurements were made on UV-Vis Spectrophotometer- 1900s and FTIR Model ABB 3000 was used for FTIR spectroscopic method.

Reagents

1. 1, 10- phenanthroline (1% w/v)
2. Ethanol (95% w/v)
3. Ferric chloride (1% w/v)
4. Isoniazid (1% w/v)

All the reagents used were of analytical grade.

Preparation of standard solution

A 1 mg/ml stock solution of BRN was prepared by dissolving 100mg of drug in 100ml of ethanol.

Method A

Appropriate aliquots of BRN were pipetted out into a series of 10ml volumetric flask. To each flask 2ml of 1,10 phenanthroline and 2ml of ferric chloride reagent were added, heated on boiling water bath for 15 minutes, cooled and made up to the volume with ethanol. The λ_{max} of the orange chromogen was found to be 510nm (Figure-1). The absorbance of orange coloured chromogen was measured at 510nm against the reagent blank. The orange chromogen was suitable for more than 3 hours.

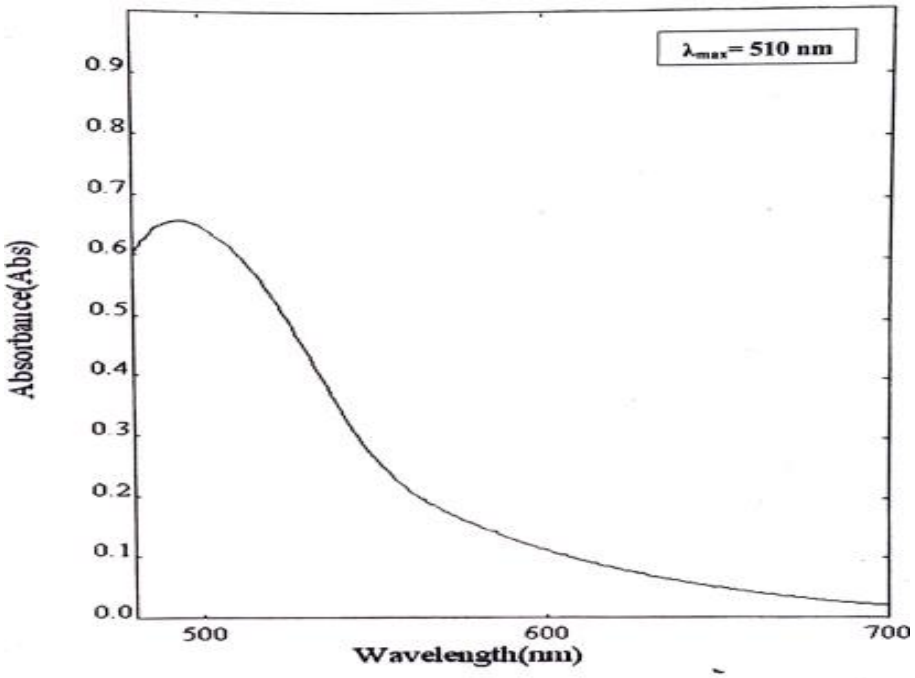

Figure No. 1: λ_{max} of the orange chromogen by Method A

Method B

Appropriate aliquots of BRN were pipette out into a series of 10ml volumetric flasks. To each flask 2ml of Isoniazid and 1ml of stock solution were added, mixed thoroughly and made up to volume with ethanol. The λ_{max} of the yellow colored chromogen was found to be 456nm (Figure- 2). The absorbance of the yellow colored chromogen was measured at

456nm against the reagent blank. The yellow chromogen curve was stable for more than 3 hours.

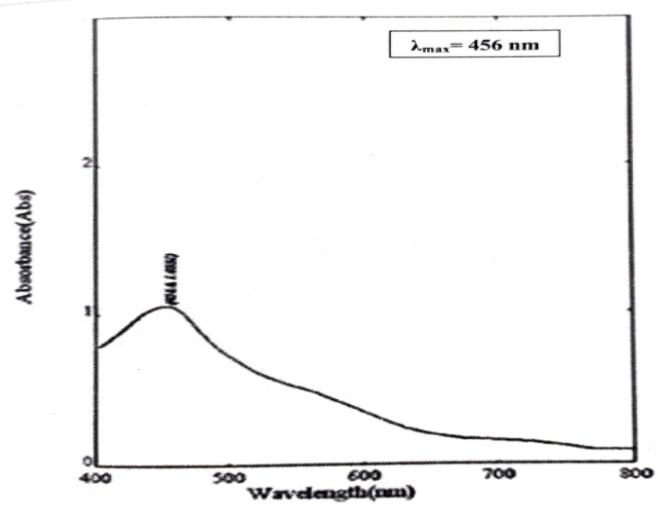

Figure No. 2: λ_{max} of the yellow chromogen by Method B.

FTIR Spectroscopic method

The stock solution was diluted suitable with ethanol to give a series of concentration ranging from 20-100µg/ml. The IR spectrum was recorded for the various concentrations. The absorbance of the band due to NH stretching at 3325 cm^{-1} was measured. IR spectrum of BRN is shown in Figure 3.

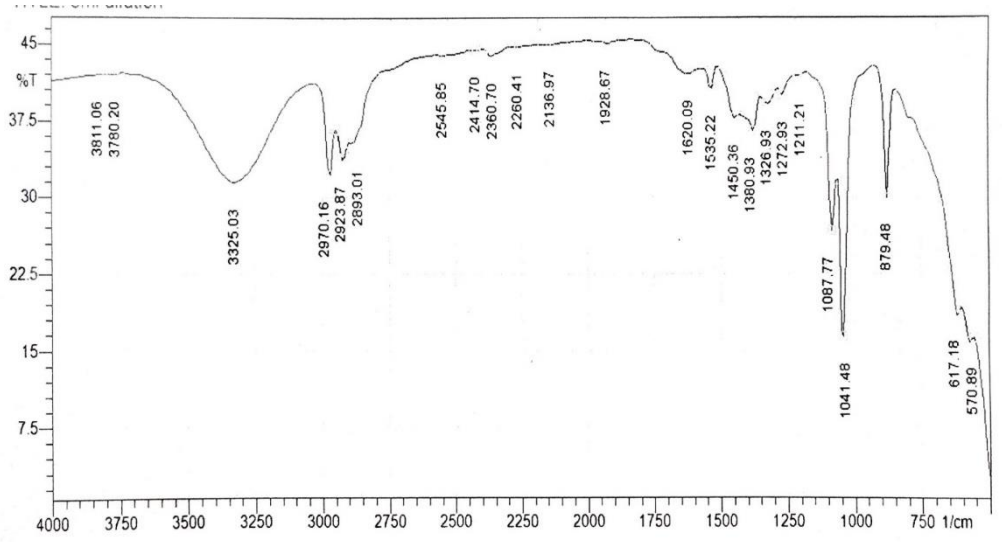

Figure No. 3: IR spectrum of BRN.

RESULTS AND DISCUSSION

The optical characteristics such as absorption maxima, Beer's law limits, Molar absorptivity and Sandell's sensitivity are furnished in Table- 1. The regression characteristics like slope (b), intercept (a), correlation coefficient (r) and percent relative standard deviation (% RSD) obtained from different concentrations were calculated and the results are summarized in table- 1.

Table No. 1: Optical and Regression Parameters

Parameter	**Method A**	**Method B**	**FTIR spectroscopic method**
λ_{max}/ Stretching	510nm	456nm	N-H stretching at 3325cm^{-1}
Linearity range (µg/ml)	10 - 60	50 -300	100 - 600
Molar absorptivity (Lmol-1cm^{-1})	43.01 x 10^3	52.02 x 10^3	----
Sandell's sensitivity (µg/cm^2/0.001 absorbance unit)	0.009083	0.008706	----
Regression equation (*y)			
Slope (b)	0.0127	0.0026	0.0122
Intercept (a)	0.0389	0.0022	0.0030
Correlation co-efficient (r)	0.9982	0.9968	0.9992
% RSD	0.832	0.729	0.767

*y = a+bc where c is the concentration of BRN in µg/ml.

ACKNOWLEDGEMENTS

We are thankful to the Department of Pharmaceutical Chemistry, Madras Medical College, Chennai- 03, for providing the instrumentation and laboratory facilities.

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