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DEVELOPMENT AND VALIDATION OF SPECTROSCOPIC METHOD FOR THE ESTIMATION OF NAFTOPIDIL IN BULK AND DOSAGE FORM

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Abstract

A simple, rapid and sensitive method has been developed for the quantitative estimation of n in bulk and tablet. The wavelength 285 nm was selected for the estimation of drug using distilled water: acetonitrile (50:50) as a solvent. The drug obeyed Beer-lambert's law over the concentration range 5-25µg/ml. The accuracy of the method was assessed by recovery studies and was found between 99.73-100.17%. The method was statistically validated for the linearity, precision, accuracy, repeatability, LOD, LOQ and ruggedness. The method was successfully applied for routine analysis of this drug in bulk and formulations.

Key words: Naftopidil, UV-spectroscopy, Validation, Recovery

Introduction

Naftopidil,a phentermine derivative, a selective α1-adreno receptor antagonist, a calcium antagonist and a 5-HT1A agonist[1]. It is a renal urological drug that is utilized extensively for the treatment of arterial hypertension and benign prostatic hypertrophy (BPH) 2. Literature survey reveals that few chiral HPLC[3, 4,] RP-HPLC in bio samples [5-8] and phosphorimetric methods[9, 10] reported for the estimation of naftopidil. No simple UV-spectroscopic method has been reported for the estimation in dosage forms. In the present study to develop a simple, accurate and precise UV-spectroscopic method for estimation of naftopidil in bulk and dosage form. The validation has been carried out as per ICH guidelines.

Experimental

Apparatus

A shimazu-1700 double beam UV-Visible Spectrophotometer with 1 cm matched quartz cell was used for all spectral measurements.

Materials

All the chemicals used were of analytical grade. A gift sample of Naftopidil obtained from Hetero Laboratories, Hyderabad, India was used as working standard. The formulation (Naftomax-50mg) was purchased from local pharmacy.

Preparation of standard stock solution

Standard stock solution was prepared by dissolving 100 mg of Naftopidil in the distilled

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water:acetonitrile (50:50) and the volume was adjusted to 100ml with the same, to give a solution of concentration 1000µµg/ml. Different aliquots were transferred to series of 100ml volumetric flasks and volume was made up to the mark with distilled water: acetonitrile (50:50) to obtain series of concentrations. The absorbances of these solutions were measured at 285 nm and a calibration curve was constructed, by plotting the absorbance against the corresponding drug concentration.

Assay procedure

To determine the content of Naftopidil in solid dosage form, twenty tablets containing the drug were accurately weighed. Their average weight was determined and finely powdered. An amount of powder, equivalent to 25 mg of Naftopidil was weighed, dissolved in distilled water: acetonitrile (50:50) and shaken mechanically for 20min. The solution was filtered and diluted to 25ml with distilled water: acetonitrile (50:50). Aliquot of 1ml from this solution was further diluted ten times its volume with distilled water: acetonitrile (50:50) and scanned at 285 nm. The procedure was repeated six times.

Results and discussion

The drug Naftopidil was analyzed at 285nm in distilled water:acetonitrile (50:50) using UV-Visible spectrophotometer. Optical characteristics such as Beers's law limits, intercept and slope has been calculated using regression equation, which has been presented in Table 1.

Intra-day and inter-day precision: precision was determined by analyzing the drug at three different concentrations and each concentration for three times, on the same day. Inter-day precision was determined similarly, analyzing the samples daily, for three consecutive days. The results are summarised in Table 2.

To ensure the accuracy of method, recovery studies were performed by standard addition method at 50%, 75% and100% levels of drug concentration, to the pre-analyzed samples and percent recovery values were calculated. Recovery experiment indicated the absence of interferences from the commonly encountered pharmaceutical additives and excipients.

Sensitivity of the method was determined in terms of limit of detection (LOD) and limit of quantification (LOQ). The LOD and LOQ were calculated by using the formula, LOD=3.3×σσ/S and LOQ=10σ×/S, where σ is residual standard deviation of the regression line and S is the slope of the corresponding regression line. The LOD and LOQ were found to be 0.03387 µg/ml and 0 .10264 µg/ml. The results did not show any statistical difference between operators and an instrument suggesting that method developed was rugged.

The developed analytical method was validated[10] as per the guidelines laid by USP. The developed method was found to be simple, accurate, precise, sensitive and economical. The results of the validation tests were found to be satisfactory and therefore, this method can be applied to routine analyze of drug in formulations.

Table No. 01: Optical characteristics of Naftopidil

Parameters	Values
λ_{max} (nm)	285
Beers law limit (µg/ml)	5-25
Correlation coefficient (r)	0.999
Regression equation (y=mx+c)	Y=0.027X+0.023
Slope(m)	0.027
Intercept(c)	0.023
LOD (µg/ml)	0.03387
LOQ (µg/ml)	0.10264
Standard error	0.01402

Table No. 02: Results of assay, recovery, and precision and ruggedness data

Parameters	Results
Amount found (mg/ Tab)	50.185
% Recovery (n=3)	99.732
% RSD	0.03337
Precision [% RSD]	
Intra –day (n=3)	0.44889
Inter-day(n=3)	0.41281
Repeatability(n=6)	0.0501
Ruggedness [% RSD]	
Analyst I (n=3)	0.0511
Analyst II (n=3)	0.0505
Instrument I (n=3)	0.0495
Instrument II (n=3)	0.0470

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