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Spectrophotometric Method for the Estimation of Nicorandil in Tablet Dosage form

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A B S T R A C T

A simple and sensitive spectrophotometric method for the determination of Nicorandil in tablet dosage form was described. From the solubility data 0.1 N NaOH was used as solvent and shows absorption maximum at 260 nm. The Beer's Law range is 05–25 µg/ml. The linear regression for method found to be 0.99998. When tablet dosage forms where analyzed, the results obtained by the proposed methods are in good agreement with the labelled amount and the results were validated statistically.

Keywords: Nicorandil, UV spectroscopy, tablet dosage form, statistical validation

A R T I C L E I N F O

CONTENTS

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1. Introduction

Nicorandil is chemically known as 2-[(pyridin-3-yl carbonyl) amino]ethyl nitrate and shown in fig.1 It is used for the treatment of the signs and symptoms of anti-angina medication that has the dual properties of a nitrate and K+ATP channel agonist. In humans, the nitrate action of Nicorandil dilates the large coronary arteries at low plasma concentrations. At high plasma concentrations Nicorandil reduces coronary vascular resistance, which is associated with increased K+ATP channel opening. A literature survey revealed no spectrophotometric methods for the estimation

of Nicorandil in pure and tablet dosage form. HPLC and LC-MS methods were reported for the estimation of Nicorandil. In the present report, the paper describes a simple and sensitive spectroscopic method for the determination of Nicorandil in pure and tablet dosage form.

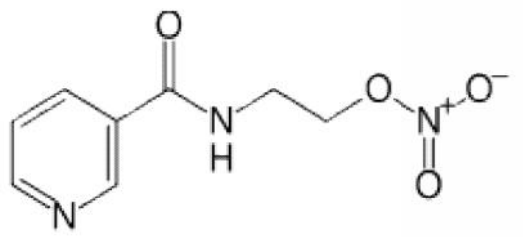

Figure 1: Structure of Nicorandil

2. Materials and Methods

Pharmaceutical grade of Nicorandil was kindly gifted from Lupin Pharmaceuticals, Pune. The brand of Nicorandil tablets used was Korandil and procured from a local Pharmacy. All the solvents and chemicals used were of analytical reagent grade and procured from Qualigens fine Chemicals (Mumbai).

Instruments:

Shimadzu AX - 220 digital balances, T 60- UV - Visible spectrophotometer with 1 cm matched quartz cells, Sonicator Sonica Ultrasonic cleaner model 2200 mH.

Method

Simple UV- Spectroscopy

The solubility of Nicorandil was determined in a variety of solvent ranging from non polar to polar using essentially a method of Schefter and Higuchi [6]. The drug was found to be very soluble in 0.1 N NaOH, chloroform, glacial acetic acid, ethanol, Distilled Water and freely soluble in 0.2 N methanolic hydrochloric acid. Considering the economic factor and the drug were stable in 0.1 N NaOH for 3 h, 0.1 N NaOH was selected as the solvent for method.

Preparation of standard stock solution:

10 mg Nicorandil was accurately weighed and transferred into a 50 ml standard flask and dissolved with minimum quantity of 0.1 N NaOH and made up to 50 ml with more 0.1 N NaOH (100 µg /ml).

Selection of λ_{max} and stability studies:

The standard stock solution was further diluted with 0.1 N NaOH to get 10 µg/ml concentration (1 ml to 100 ml). The solution was scanned between 200 and 400 nm using 0.1 N NaOH as blank. From the spectrum obtained, 260 nm was selected as λ_{max} for the analysis of Nicorandil. Stability studies were performed and Nicorandil was found to be stable for 3 h and shown in Fig: 2.

Calibration graph and linearity:

In this method, the aliquots (0.5–2.5 ml) of standard stock solution of Nicorandil were transferred into 100 ml standard flasks and made up to the mark with 0.1 N NaOH. The absorbance was measured at 260 nm against 0.1 N NaOH as blank. The sample solutions were found to be linear from 05-25 µg/ml. The calibration curve was plotted between concentration and absorbance and shown in Fig: 3.

Quantification of formulations:

Thirty tablets of formulation (Korandil) containing 05 mg of Nicorandil were accurately weighed to find out the average weight and powdered. Transferred the powdered tablets equivalent to 50 mg of Nicorandil into a 50 ml conical flask, extracted with 0.1 N NaOH for three times (3 x 10 ml), sonicated for 15 min and produced to 50 ml with 0.1 N NaOH using a standard flask. Half of the solution was filtered using Whatmann filter paper No. 41. From this clear solution, 5 ml was transferred to a 25 ml standard flask and produced to obtain 100 µg/ml solution with 0.1 N NaOH. The absorbance was measured at 260 nm using 0.1 N NaOH as blank. The amount of Nicorandil present in each formulation was calculated from the slope and intercept of respective calibration curve and shown in table: 2.

Recovery studies:

From each of the preanalyzed formulation, known quantities were taken (2.5 µg/ml) and the raw material solution was added in ascending amounts (2.5, 7.5, 12.5, 17.5 and 22.5 ml) to 100 ml standard flasks. The contents were mixed well, finally made up to the mark and filtered. The absorbance was measured at 260 nm using 0.1 N NaOH as blank and the amount of drug recovered from the each formulation was calculated by the mathematical relation followed by Sane *et al* and shown in Table: 3.

Statistical Validation:

The obtained results were treated for statistical validation parameters like Standard Deviation (SD) and Percentage Relative Standard Deviation (% RSD).

3. Results and Discussion

The solubility profile of Nicorandil was determined as per procedure followed by Schefter and Higuchi. Using various polar to non polar solvents and from the solubility studies the category of solvents for Rasagiline was hereby confirmed as freely soluble in 0.1 N NaOH, Dist. Water, very soluble in 0.1 M Hydrochloric acid, Acetonitrile, Acetic acid, Chloroform, Ethanol, and 0.1 M methanolic Hydrochloric acid.

Method

0.1 N NaOH was selected as solvent for simple UV-method because of its easy availability, cost factor and high stability. The proposed method for estimation of Nicorandil in pure and in tablet dosage form were found to be simple and sensitive. The drug in 0.1 N NaOH shows λ_{max} at 260 nm, with linearity range of 05 – 25 µg/ml. The optical parameters like Beer's law limits (05-25 µg/ml), Sandell's sensitivity (0.159776521), correlation coefficient (0.99998), slope (0.025664), intercept (0.00021), limit of detection (0.1278701), and limit of quantification (0.3468102) were calculated for Nicorandil in 0.1 N NaOH and produced in Table 1.

Quantification of Nicorandil from tablets dosage form was performed and the amount present was determined by average of six replicate analysis and the amount in percentage purity is found to be 100.94 and shown in table 1s. To evaluate the accuracy of the method and for knowing the interference from excipients recovery study was

performed. The Recovery of Nicorandil by UV-Spectroscopic method was found to be 98.69 and the results are shown in Table 3. The values of co-efficient of variance were satisfactorily low and recovery was close to 100 % indicating reproducibility of the methods. The excipients in the formulation did not interfere in the accurate estimation of Nicorandil in tablet dosage form. From the results, the UV-Spectroscopy method was found to be more precise. Since none of the spectroscopic method is reported for the estimation of Nicorandil in tablet dosage form, this developed method can be applied in industries for routine analysis of the Nicorandil in tablet dosage form.

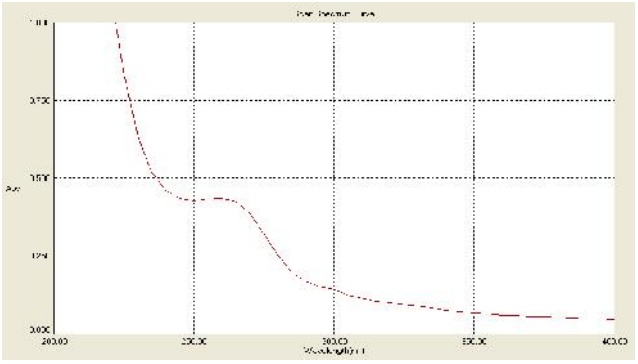

Figure 2: UV Spectrum of Nicorandil in 0.1 N NaOH (10µg/ml)

Table 1: Optical Characteristics of Nicorandil

Parameters	Method
λmax (nm)	260
Beers law limit (µg/ml)	5-25
Correlation coefficient (r)	0.999987
Régression équation (y=mx+c)	Y=0.025664X+0.00021
Slope(m)	0.025664
Intercept(c)	0.00021
LOD (µg/ml)	0.12787017
LOQ (µg/ml)	0.3468102
Standard error of mean of Regression line	0.1788658

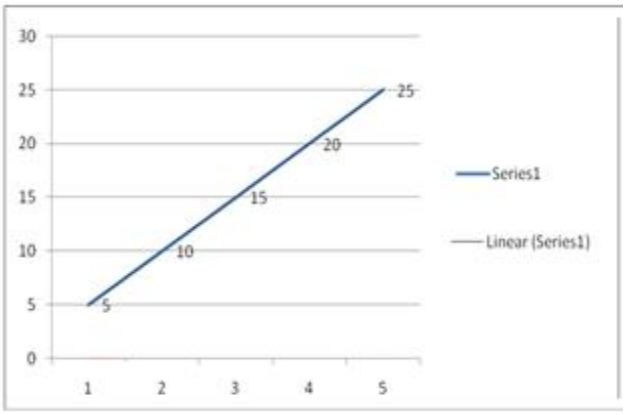

Figure 3: Calibration Curve of Nicorandil in 0. 1 N NaOH (10µg/ml)

Table 2: Results of Analysis of Commercial Formulations

S.No	Label claim (mg/tab)	Amount found(mg)*	Percentage purity*	Average	S.D	% R.S.D	S. E
1	05	4.9546	99.09				
2	05	5.2880	103.76				
3	05	4.2880	99.76	100.94	0.3999561	2.666374	0.17886580
4	05	5.4216	102.43				
5	05	4.9846	99.69				

SD is standard deviation, % RSD percentage relative standard deviation
*Average of six determinations

Table 3: Results of Recovery Studies

S.No	Amount Present (µg)	Amount Added * (µg)	Amount Found*(µg)	Amount Recovered*(µg)	Recovery%
1	2.5	2.5	4.9542	2.4542	98.16
2	2.5	7.5	9.7346	7.2346	96.46
3	2.5	12.5	14.8210	12.321	98.56
4	2.5	17.5	20.1021	17.6021	100.58
5	2.5	22.5	24.9342	22.4342	99.70

*Average of six determinations

4. Conclusion

Considering the solubility and stability, 0.1 N NaOH was selected as solvent and further dilutions were made with 0.1 N NaOH. Nicorandil 10 µg/ml solution was prepared and scanned in the UV region. From the spectra 260 nm was selected as an analysing wavelength calibration. Nicorandil obeys Beer's law in the range of 5-25 µg/ml, Correlation coefficient: 0.99998, Slope: 0.025664, Intercept: 0.00021, LOD: 0.12787017 µg/ml and LOQ: 0.3468102µg/ml was calculated. The amount of Nicorandil in formulation (Korandil) was found to be 100.94. The precision of the method was studied by making repeated analysis. The recovery studies were also carried out to ensure the accuracy of the method by adding known concentration of drug to a pre-analyzed formulation. The average percentage

recovery for formulation (Korandil) was found to be 98.96 %.Precision of the method was studied by intraday and interday Analysis on assay and recovery.

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