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International Journal of ChemTech Research
CODEN(USA): IJCRGG ISSN : 0974-4290
Vol.2, No.4, pp 2052-2055, Oct-Dec 2010

Spectrophotometric Method for the Determination of Lincomycin Hydrochloride in Pure Form and Pharmaceutical Formulations

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Abstract: A simple specific, precise and accurate spectrophotometric method has been developed for the estimation of lincomycin hydrochloride in bulk and tablet dosage form. In the developed method water was used as the solvent. The absorption maximum of the drug was found to be 196nm.The method was statistically validated according to international conference on harmonization (ICH) guidelines. Percent mean recovery was obtained to be 99.3%, whereas the coefficient of variance was found to be less than 2%.The drug follows a linear Lambert-Beer law relationship with respect to the drug concentration in the range of 5-30µg/mL, with linearity coefficient of 0.9999.
Key Words: Spectrophotometric, Lincomycin hydrochloride, water.

INTRODUCTION

Lincomycin hydrochloride is systemic antibiotic, belongs to the group of lincosamide, which is active against most common gram positive bacteria. Lincomycin hydrochloride inhibits cell growth and microbial protein synthesis, by interacting strongly and specifically with the 50 S ribosomal subunit, at mutually related sites[1]. Lincomycin hydrochloride consists mainly of methyl 6-amino-6,8-dideoxy-N-[(2S,4R)-1-methyl-4-propylpropyl]-1-thio-D-erythro-α-galacto-octopyranoside hydrochloride monohydrate, an antimicrobial substance produced by streptomyces lincolnensis var.lincolnensis or by any other means[2]. It has approved to be excellent for infectious disease like acne, anthrax, pneumonia and also for the treatment of furunculosis, carbuncles, impetigo, burns and wounds[3]. It is official in Indian and British pharmacopoeia. A few analytical methods have been reported for its quantitative estimation in pharmaceutical formulations which includes biological fluids using HPLC methods[4-6]. In view of the above fact, some simple analytical methods are in need for its quantitative estimation. So in the present study, a simple, specific, precise, economical, accurate and validated spectrophotometeric method has been developed for the estimation of lincomycin hydrochloride in bulk and tablet dosage form, using water as the solvent system.

EXPERIMENTAL

Instrumentation

All spectral and absorbance measurements were made on a Shimadzu UV/visible double beam spectrophotometer (model 1700) with 1cm matched quartz cells were used for all the spectral measurements. Shimadzu-AX-200 electronic balance was used for weighing the samples.

Materials and Reagents

Class 'A' volumetric glassware were used. Ultrasonicator was used in the initial steps of extraction. Whatmann filter paper No.41 was used to filter the solution. All the chemicals used were of analytical grade procured from Qualigens Fine chemicals, Mumbai.

Double distilled water was used. The pharmaceutical grade of lincomycin hydrochloride was supplied as a gift sample by Wallace Pharmaceutical Pvt. Ltd, Goa. As this drug has no marketed tablet formulations yet, we have prepared tablets (Tablet1 and Tablet 2) by varying the ratio of using most commonly used excipients like lactose, HPMC, PVP and magnesium stearate by keeping the strength as constant (1000 mg of lincomycin hydrochloride) and analyzed the drug.

Preparation of standard solution of lincomycin hydrochloride

Accurately weighed 50mg of lincomycin hydrochloride was transferred to 50mL volumetric flask. Drug was then dissolved and made up to the volume to 50mL with water. It was further diluted to a concentration of 100µg/mL with distilled water, which was then used as the stock solution for the further dilutions.

Determination of wavelength of maximum absorbance of lincomycin hydrochloride

From the above prepared standard moprolol solution 2 mL was transferred to 10mL volumetric flask and diluted to 10mL with distilled water. The absorbance of the final solution was scanned in the range 190-400nm against distilled water as blank. The absorbance maximum of drug was found to be 196 nm.

Preparation of calibration curve for lincomycin hydrochloride

Dilutions of the standard moprolol solution were prepared (0.5,1.0,1.5,2.0,2.5 and 3.0mL) diluted to 10mL using distilled water in the range of 5 to 30µg/mL in a series of six dilutions in volumetric flasks of capacity 10mL.The absorptions of the solutions were measured at 196nm using distilled water as blank. The absorbance values are shown in Table-1.

Estimation of lincomycin hydrochloride from tablets

Twenty tablets of lincomycin hydrochloride (of same batch) were taken and the average weights of these tablets were determined. Then these tablets were finely powdered and triturated well. A quantity of powder equivalent to 250mg of lincomycin hydrochloride was transferred to 100mL volumetric flask and mixed with distilled water and there after the volume was made up to 100mL with the same. The solution was filtered through whatmann filter paper No.41. From the filtrate, further dilutions were made with distilled water to get a final solution of 15 µg/mL concentration. The absorbance of this solution was measured at 196 nm using distilled water as blank. The amount of drug present in the tablet was calculated using the standard calibration curve of the drug.

Recovery studies and validation of the method according to ICH guidelines

Precision of the newly developed method was studied by carrying out intraday, interday analysis and expressed as percent coefficient of variance[9].Specificity of the method was checked by adding few excipients with in the range as specified in standard literature which are usually added in the preparation such as diluents, lubricant *etc*. to the preanalyzed samples. The absorbance of the solution so obtained after addition of excipients was then measured, compared with that of the absorbance of preanalyzed solution and the specificity was expressed in terms of percent interference, which was found to be less than 2% limit of detection (LOD) and limit of quantification (LOQ) were studied based on standard deviation of the response and slope curve. Recovery studies were carried out by addition of standard drug (spiking) to preanalyzed samples of the prepared formulation, taking into consideration the percentage purity of the added bulk drug.

TABLE-1: CALIBRATION CURVE FOR LINCOMYCIN HYDROCHLORIDE

Concentration (µg/mL)	Absorbance
5	0.149
10	0.287
15	0.426
20	0.568
25	0.705
30	0.834

TABLE -2: OPTICAL CHARACTERISTICS, PRECISION AND ACCURACY OF THE PROPOSED METHODS.

Parameters	Observations
λ_{max} (nm)	196
Beer's law limits (µg/ml)	5-30
Sandell's sensitivity (µg /ml/ cm^2/ 0.001 absorbance unit)	0.03574
Regression equation (Y*)	
Slope (b)	0.02797
Intercept (a)	0.00660
Correlation coefficient (r)	0.9999
Precision (%coefficient of variance)	
Repeatability	0.273
Intraday	1.07
Interday	1.17
% RSD	0.205
Range of errors**	
Confidence limits with 0.05 level	0.00489
Confidence limits with 0.01 level	0.00692
Limit of detection (µg/mL)	1.269
Limit of quantification (µg/mL)	0.354

- Y = bC + a where C is the concentration of lincomycin hydrochloride in µg/ml and Y* is the absorbance unit

TABLE-3: PERCENTAGE RECOVERY STUDY DATA OF TABLET FORMULATION

Drug	Level of % recovery	% recovery found*	SD
Lincomycin hydrochloride	80	99.26	0.568
	100	99.10	0.448
	120	99.47	0.229

* Mean of five replicates.

TABLE-4: ASSAY OF FORMULATIONS

Formulation	Label claim(mg)	Amount estimated(mg)	Mean (±SD) Mean(mg) found by	Mean (±SD) % labelled amount*
Tablet 1	1000	998.95	24.99±0.2017	99.89±0.806
Tablet 2	1000	999.79	24.81±0.772	99.97±0.044

*Mean of five replicates.

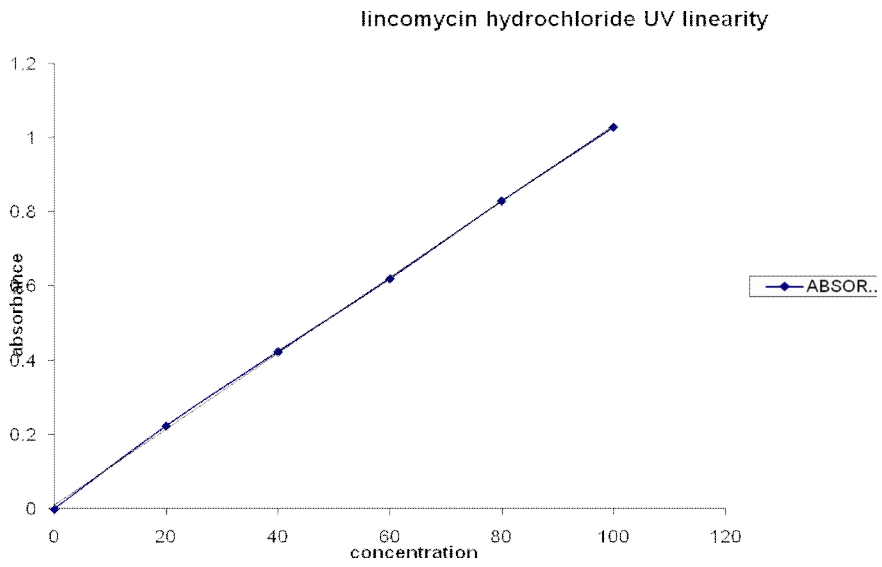

Fig.1.Calibration curve for Lincomycin hydrochloride

RESULTS AND DISCUSSION

The linear regression equation for lincomycin hydrochloride standard curve was calculated by y=0.02797x + 0.00660 (R^2 = 0.9999), where y =absorbance and x = value of various concentrations of standard solutions using UV spectrophotometeric method. The value of regression coefficient from the above straight-line equation depicts the linearity of the data range and for given data it shows that the Lambert-Beer law follows a linear relationship for lincomycin hydrochloride in the range of 5-30µg/mL.

For precision, repetability, intraday/interday, three replicate experiments were carried out and their %RSD readings were calculated at the selected λ_{max} .The low value of % RSD revealed good precision, as shown in Table-3.

The results of the estimation of lincomycin hydrochloride in the prepared formulation are summarized in Tables 3 and 4.

ACKNOWLEDGEMENT

The authors are grateful to Srinivas College of Pharmacy, Mangalore for providing research facilities. The authors wish to express their gratitude to Wallace Pharmaceutical Pvt. Ltd, Goa for proving the authentic sample (Lincomycin hydrochloride).

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