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DEVELOPMENT AND VALIDATION OF SPECTROPHOTOMETRIC METHOD FOR ESTIMATION OF PRULIFLOXACIN IN TABLET DOSAGE FORM

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ABSTRACT: This paper represents method development and validation of prulifloxacin by UV spectrophotometer in Acetonitrile:Water (5:5) medium and intensity of UV absorption and stability. The maximum absorption peak at 279nm. The linear regression analysis data for the calibration plots showed good linear relationship with r^2=0.9995 in the concentration range 1 to 14 µg/ml with respect to peak area. According to the International Conference on Harmonization (ICH) guidelines the method is simple, high sensitivity, reliability, good for prulifloxacin qualitatively as well as quantitavily.

Key words: UV spectrophotometer, Prulifloxacin, Method development and Validation, Calibration curve.

INTRODUCTION

Prulifloxacin is the lipophilic prodrug of ulifloxacin, a is new thiazeto-quinolone antibacterial agent with broad-spectrum vitro activity against various Gram-negative and Gram-positive bacteria and acts directly on bacterial DNA gyrase inhibiting cell reproduction that leads to cell death. Prulifloxacin has a chemical structure that allows its absorption from the gastrointestinal tract and can therefore administered orally. Its half-life is quite long and the molecule remains in the bloodstream for about 11 hours. This characteristic allows a 600-mg tablet to be administered only once a day, for a very convenient dosing. The active metabolite of prulifloxacin (ulifloxacin) is mostly cleared, in an unchanged form, through the urinary tract; this allows the drug to be consistently active until its clearance. [1,2,3]

EXPERIMENTAL

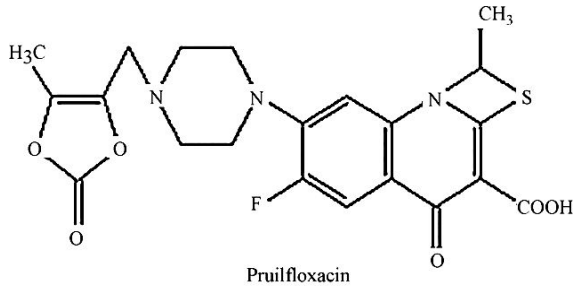
MATERIALS AND METHODS

Instrument

UV spectroscopic method was performed on a Jasco V 630 UV-Visible Spectrophotometer at 279 nm using 1.0 cm quartz cells. [4]

Selection of solvent

The ideal property of a solvent should be that the drug should be completely soluble in the solvent used. The drug should be stable in the solvent used and should be economical and volatile. After suitable literature survey, practical experience and taking above factors into consideration the suitable solvents selected were Acetonitrile**:**Water.(5:5)



Pruilfloxacin

Figure1: Chemical structure of prulifloxacin

Wavelength selection

Stock solution of drug was prepared in Acetonitrile: Water and UVspectrum of drug was taken and it was

found that prulifloxacin showed maximum absorbance at 279 nm as shown in Fig 2. [5, 6]

Standard solution and calibration curve

Stock solution of standard drug was prepared by dissolving 10 mg of Prulifloxacin in 100ml of Acetonitrile:Water. For calibration curve the standard solutions were prepared by dilution of the stock solution with mobile phase to reach a concentration range 1-14 µg/ml for Prulifloxacin.The absorbance were plotted against the corresponding concentrations to obtain the calibration graph.

Sample preparation for tablet analysis

To determine the content of prulifloxacin in conventional tablets (label claim: 600 mg prulifloxacin per tablet), Twenty tablets were weighed, their mean weight was determined and they were finely powdered and powder equivalent to 1 tablet of prulifloxacin was weighed. Then equivalent weight of the drug was transferred into a 10 ml volumetric flask containing 10 ml Acetonitrile: water, sonicated for 20 min. Solution was taken and after suitable dilution the sample solution was then filtered using 0.45-micron filter (Millipore).

VALIDATION OF UV METHOD: [7, 8, 9]

1. Linearity and range

Linearity plot is showed in figure 2. The response for prulifloxacin was linear in the concentration range of 1µg/ml – 14µg/ml**.** The regression equation calculated by least square method was y = 0.1011x + 0.0275 with coefficient of correlation r2 = 0.9995.

2. Accuracy

To verify the capability of regression equations to predict the absorbance behavior of Prulifloxacin in dosage forms, the method was tested for precision and recovery. To study the recovery the pre-analyzed sample solutions a known amount of standard solutions of the pure drugs were added at different level As shown in Table 2 excellent recoveries were made at each added concentration.

3. Precision

Data obtain from precision experiments are given in Table 2. Precision was calculated for Intraday and for Interday. The data obtained shows that method is sufficiently precise. Precision is calculated as % Relative Standard Deviation.

4. Specifity

Specificity is a procedure to detect quantitatively the analyte in presence of component that may be expected to be present in the sample matrix. According to the results obtained by UV Spectrophotometric method is able to access the analyte in the presence of excipients and hence, it can be considered specific. It has been concluded that there was no spectral interaction in the analysis of pharmaceutical preparation of prulifloxacin. Therefore, calibration curve method was chosen for analysis of drug.

5. Analysis of marketed formulation[10]

Experimental results of the amount of prulifloxacin in tablets, expressed as percentage of label claim were in good agreement with the label claims thereby suggesting that here is no interference from any excipients, which are normally present in tablets. The drug content was found to be 98.83 % for prulifloxacin.

RESULTS & DISCUSSION

The method for the estimation of prulifloxacin in tablet dosage form was developed. Drug shows absorption maximum at 279 nm. Spectrophotometric method linear response obtained was in the concentration range of 1-14 µg/ml with correlation coefficient 0.9995, recovery of the drug was found to be 98.83 %. The method was statistically validated according to ICH guidelines. The developed validated methods are simple, rapid, precise and accurate. The newly developed methods can be used for routine analysis of prulifloxacin in tablet dosage forms.[11]

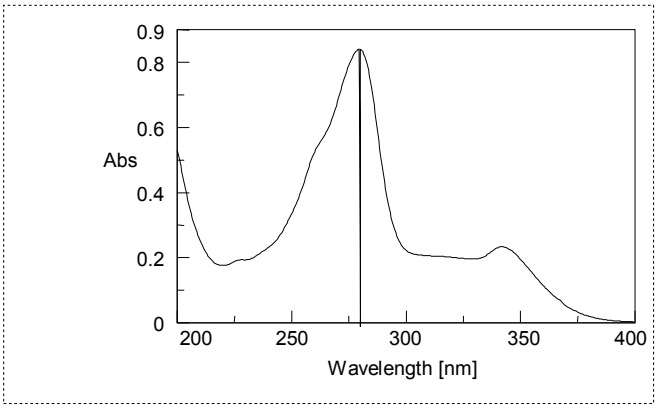


Figure 2: Spectrum of Prulifloxacin at wavelength 200nm to 400nm

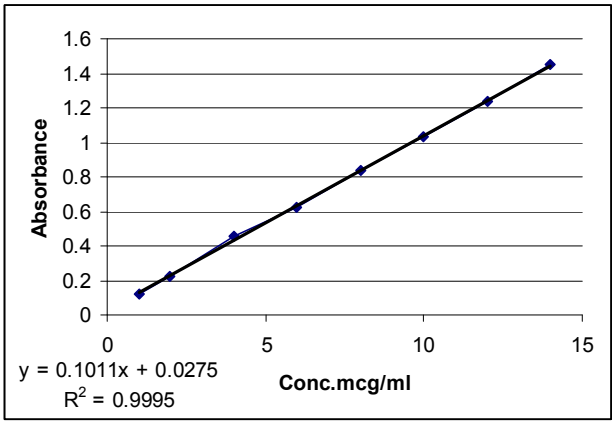


Figure: 3 Calibration Curve of Prulifloxacin

Sr.no	Conc(µg/ml)	Area
1	1	0.121
2	2	0.226
3	4	0.454
4	6	0.627
5	8	0.838
6	10	1.031
7	12	1.234
8	14	1.449

Table 1: Data of Calibration curve

Table 2: Accuracy and Precision data evaluated through Intra-day & Inter-day

Intraday					
	1µg/ml		8µg/ml		14µg/ml
	0.121		0.838		1.449
	0.122		0.838		1.448
	0.121		0.836		1.446
	0.122		0.838		1.449
	0.123		0.834		1.446
	0.121		0.836		1.449
Mean	0.121667		0.8367		1.4478
Std Dev	0.000816		0.001633		0.001472
%RSD	0.6706		0.1951		0.1017
%Accuracy	100.49		99.84		99.91

Interday					
	1µg/ml		8µg/ml		14µg/ml
	0.122		0.838		1.449
	0.124		0.835		1.448
	0.124		0.839		1.443
	0.123		0.841		1.445
	0.121		0.841		1.449
	0.122		0.834		1.446
Mean	0.1226		0.838		1.4466
Std Dev	0.001211		0.002966		0.002422
%RSD	0.9558		0.3539		0.1674
%Accuracy	101.32		100		99.83

REFERENCES

1. Robert A Nash, Alfred H Watcher, pharmaceutical process validation, 3rd ed, volume 129.
2. www.medicinescomplete.com/mc/merck/current/07908.htn
3. Hokanson, G.C. A life cycle approach to the validation of analytical methods during pharmaceutical product development, part I: the initial validation process Pharm tech.
4. Roger E. Schirmer; Modern Methods of Pharmaceutical Analysis, 2nd edition, Vol.1.
5. Remington: The Science and Practice of Pharmacy; Vol. II.
6. General Chapter 1225, Validation of compendial methods, United States Pharmacopeia 30, National Formulary 25,

Rockville, Md., USA, The United States Pharmacopeial Convention, Inc, (2007).
7. Joachim Ermer, John H. McB Miller; method Validation in Pharmaceutical analysis: A Guide to Best Practice.
8. Mehdi H., Maryam K., Mehdi B. and Hassan J., Derivative spectrophtometric method for determination of Losartan in pharmaceutical formulation, Iranian Journal of Pharmacology & Therapeutics, 3,2004, 21-25
9. Validation of analytical procedure and methodology adopted ICH guidelines, 1996.
10. David C. Lee, Michael Webb; Pharmaceutical Analysis
11. N. V. Nagaraja, J. K. Paliwal, R. C. Gupta, Choosing the calibration model in assay validation, Journal of Pharmaceutical and Biomedical Analysis, Volume 20, 1999; 433-438.

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