SPECTROPHOTOMETRIC METHODS FOR ESTIMATION OF PRULIFLOXACIN IN BULK DRUG AND ITS DOSAGE FORM

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Abstract: Simple, accurate, precise, sensitive and highly selective spectrophotometric methods were developed for the estimation of Prulifloxacin. The estimation of Prulifloxacin was carried out by various agents like UV (method I) FC Reagent (method II) and MBTH (method III). And these methods were found to be linear in the range of 1-6µg/ml, for method I and II and 10-60µg/ml for method III. And **B**eers law range were found to be 1-15µg/ml, 1-15µg/ml, and10-250µg/ml, and with mean recovery of 97.5 %, 97.5 % and 103.5 % of r Prulifloxacin for methods I, II and III respectively. The developed method was validated according to ICH guidelines and it found to be accurate and precise Thus the proposed method can be successfully applied for simultaneous determination of Prulifloxacin and in routine analysis work.

Keywords: Prulifloxacin, Spectrophotometre. Validation, Beer's Law.

Introduction: It is *RS*)-6-Fluoro-1-methyl-7-[4-(5-methyl-2-oxo-1,3-dioxolen-4-yl)methyl-1-piperazinyl]-4-oxo-4*H*-[1,3]thiazeto[3,2-*a*]quinoline-3-carboxylic acid. Prulifloxacin prevents bacterial DNA replication, transcription, repair and recombination through inhibition of bacterial DNA gyrase or the topoisomerase IV enzyme, thereby inhibiting DNA replication and transcription. Drug for the treatment of lung and urinary infections. Prulifloxacin may be determined by several methods including gas chromatography–mass spectrometry (GC–MS)[5], liquid chromatography with UV detection (LC–UV)[6-8]. Literature survey revealed that there is few UV-visible methods have been reported.

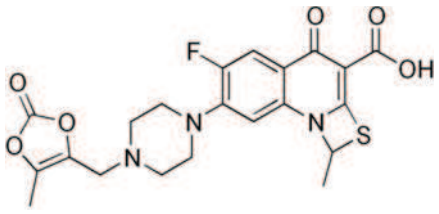

Fig. 1: Chemical Structure

Experimental Work: UV/Vis double beam spectrophotometer models Systronics double beam spectrophotometer with 1 cm UV matched quartz cells were used. Prulifloxacin having π electrons in their structure and hence absorb electromagnetic radiation between 200-400 nm. This character of these drugs is used for their estimation.

1. UV Method :

a) Selection of Solvent: Different solvents such as water, dimethyl formamide, 0.1 N NaOH have been tried and spectrum have been obtained. From the spectrum obtained 0.1 N NaOH showed good absorbance intensities compared to other solvents. Hence 0.1 N NaOH is selected as solvent for present study whereas further dilutions are made with distilled water.

b) Fixation of Scanning Speed: The scanning speed affects the absorbance of compounds and decreases the resolution between the adjacent bands. An optimum scan speed should be fixed so that it maintains pen fidelity. For the proposed study medium scanning speed was used.

c) Fixation of Wavelength for the Estimation: The overlaid spectra of prulifloxacin was obtained. From the overlaid spectra the absorbance maxima for prulifloxacin was found 273nm.The absorbance intensities were also good and well resolved peaks were found. Hence for present study 273nm were selected.

Estimation of Prulifloxacin: Estimation of prulifloxacin in dosage forms by calibration curve method was carried out

For brand(Rulizo) the amount of prulifloxacin was found to be 576mg(96%) with standard deviation 0.0017.

i. Preparation of Standard Solution: 1.100 mg of prulifloxacin was taken in 100 ml standard flask. To this solvent is added for dissolving the drug. It was shaken for one min. To get clear solution and the volume was made up to 100 ml with solvent.

2. The final standard solutions were prepared in such a way that each standard flask contains 1, 1.5, 2, 2.5 and 3µg/ml of prulifloxacin .

ii. Preparation of Formulation Solutions: Twenty tablets each containing 600 mg of prulifloxacin were weighed. The average weight was calculated. The tablets were ground to fine powder and the quantity of powder equivalent to 100 mg of prulifloxacin was weighed and dissolved in 0.1N NaOH and sonicated and the volume was made up to 100ml with same solvent. The solution was further diluted to calibration range and scanned in UV region. The absorbances were noted at 273nm. The amount of prulifloxacin was calculated. The amount found and parameter label claim were shown in the Table 7

Quantitation by Visible Spectroscopy

2. Using FC Reagent:

a) Estimation of Prulifloxacin: Estimation of prulifloxacin in dosage forms by calibration curve method was carried out

For brand (Rulizo) the amount of prulifloxacin was found to be 585mg (97.5%) with standard deviation 0.00194.

i. Preparation of Standard Solution:

1. 100 mg of prulifloxacin was taken in 100 ml standard flask. To this dmf is added for dissolving the drug. It was shaken for one min. To get clear solution and the volume was made up to 100 ml with dmf.
2. To required amount of drug solution , add 1ml of FC reagent and 4.5ml of 10% sodium carbonate solution and shake it for 10 minutes and make up the volume with distilled water so that the final standard solutions in each standard flask contains 1, 1.5, 2, 2.5 and 3µg/ml of prulifloxacin .

ii. Preparation of Formulation Solutions: Twenty tablets each containing 600 mg of prulifloxacin were weighed. The average weight was calculated. The tablets were ground to fine powder and the quantity of powder equivalent to 100 mg of prulifloxacin was weighed and dissolved in dmf and sonicated and the volume was made up to 100ml with same solvent. The solution was prepared as discussed per the above procedure and scanned in Visible region. The absorbances were noted at 680nm. The amount of prulifloxacin was calculated. The amount found and parameter label claim were shown in the Table 13

3. Using MBTH

a) Estimation of Prulifloxacin: Estimation of prulifloxacin in dosage forms by calibration curve method was carried out

For brand (Rulizo) the amount of prulifloxacin was found to be 621mg (103.5%) with standard deviation 0.00356.

i. Preparation of Standard Solution:

1. 100 mg of prulifloxacin was taken in 100 ml standard flask. To this dmf is added for dissolving the drug. It was shaken for one min. To get clear solution and the volume was made up to 100 ml with dmf.
2. To required amount of drug solution , add 2ml of MBTH reagent and 2ml of CAS solution and shake it for 10 minutes and make up the volume with distilled water so that the final standard solutions in each standard flask contains 20, 25, 30, 35 and 40 µg/ml of prulifloxacin .

ii. Preparation of Formulation Solutions: Twenty tablets each containing 600 mg of prulifloxacin were weighed. The average weight was calculated. The tablets were ground to fine powder and the quantity of powder equivalent to 100 mg of prulifloxacin was weighed and dissolved in dmf and sonicated and the volume was made up to 100ml with same solvent. The solution was prepared as discussed per the above procedure and scanned in Visible region. The absorbances were noted at 627nm. The amount of prulifloxacin was calculated.

Results and Discussion: After the development of the method for the estimation of multi component dosage form, the validation of method was performed .The parameters covered were accuracy, precision, linearity range.

a) Accuracy: Accuracy of the method was determined by the recovery experiments. To the formulation, the reference standards of the respective drugs were added at the level of 50% and 100%.These were further diluted by the procedure as followed in the estimation of the formulation. The concentrations of the drugs present in the resulting sample solution were determined using assay method at 273 nm. The absorbance value for drug was noted at given wavelength and the concentration was determined and percentage RSD values were found within the limit.

b) Precision: Interday, intraday and repeatability studies were performed at given wavelength for prulifloxacin and the respective RSD values were calculated. All the RSD values were found to be within acceptable limit hence the proposed method is reliable.

c) Limit of detection(LOD) and Limit of Quantitation(LOQ): The LOD and LOQ were determined for the drug using the formula as per ICH guideline. The LOD for prulifloxacin was found to be 62ng/ml. The LOQ for prulifloxacin was found to be 18ng/ml. The values for LOD and LOQ indicate the method is sensitive and can be used for the estimation of the drugs in dosage form.

Validation of Visible Spectroscopy Method: After the development of the method for the estimation of multi component dosage form, the validation of method was performed .The parameters covered were accuracy, precision, linearity range.

a) Accuracy: Accuracy of the method was determined by the recovery experiments. To the formulation, the reference standards of the respective drugs were added at the level of 50% and 100%.These were further diluted by the procedure as followed in the estimation of the formulation. The concentrations of the drugs present in the resulting sample solution were determined using assay method at 627 nm and 680 nm. The absorbance value for drug was noted at given wavelengths and the concentration was determined and percentage RSD values were found within the limit.

b) Precision: Interday, intraday and repeatability studies were performed at given wavelength for prulifloxacin and the respective RSD values were calculated. All the RSD values were found to be within acceptable limit hence the proposed method is reliable.

c) Limit of detection(LOD) and Limit of Quantitation(LOQ): The LOD and LOQ were determined for the drug using the formula as per ICH guideline. The LOD for prulifloxacin with FC reagent and MBTH reagent was found to be 86 and 118ng/ml respectively. The LOQ for prulifloxacin with FC reagent and MBTH reagent was found to be 262.8 and 359.5ng/ml respectively. The values for LOD and LOQ indicate the method is sensitive and can be used for the estimation of the drugs in dosage form

Summary and Conclusion: For Visible spectroscopy calibration curve method is selected various solvents were tried among which DMF as solvent which gives good absorbance values with well resolved spectrum with good intensities. For the present work 627 nm and 680 nm was selected as λmax of prulifloxacin with MBTH and FC reagent respectively. The absorbance values were noted for each drug at both the absorbance maxima. The correlation coefficient for prulifloxacin at 627 nm and 680 nm was found to be 0.99 and 0.99 respectively. The linearity range of prulifloxacin with MBTH and FC reagent was found to be 5-25 µg/ml and 20-40µg/ml respectively. The developed method was validated for accuracy and precision. The percentage of recovery of prulifloxacin with MBTH and FC Reagent was found to be for 96.5% and 91.6% for 50% and 103.5% and 97.5% for 100% level.The low standard deviation values and good recoveries indicate the reproducibility accuracy of the developed method. As well as the %RSD the precision study also were with in the acceptable limit.The interday, intraday and repeatability studies were also performed. The %RSD values for precision studies were also within acceptable limits(<2%). Hence the proposed method is simple, rapid, sensitive an d accurate and can be used for the determination of the drugs in dosage form.

Table 1: Assay and Recovery of Prulifloxacin in Pharmaceutical Formulation

Pharmaceutical formulation	labeled amount mg/tablet	amount found			%recovery		
		method a	method b	method c	method a	method b	method c
tablet 1	600	621	5848	576	103.5	97.46	96
tablet 2	600	607	589	582	101.16	96.16	97

Table 2: Optical Characteristics

OPTICAL CHARACTERISTICS	**method a**	**method b**	**method c**
λmax(nm)	627	680	273
Beers Law Limit(µg/ml)	5-25	20-40	1-3
Molar Absorptivity(Litre.mole^{-1}.cm^{-1})	4.43 × 10^3	3.87 ×10^3	3.69 × 10^4
Sandell'sSensitivity(µg/cm^2/0.001abs.unit)	0.1041	0.1190	0.0125
Regression Equation(Y)*	0.0099	0.0073	0.0896
Slope(b)	0.005	0.0222	-0.0104
Intercept(a)	0.94	0.99	1
Correlation Coefficient(r)	0.038	0.023	0.015
%RSD**of Error	0.055	0.034	0.022

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