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DEVELOPMENT AND VALIDATION OF NEW UV-SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF CIPROFLOXACIN HYDROCHLORIDE IN BULK AND PHARMACEUTICAL DOSAGE FORM

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ABSTACT

A Novel, specific, accurate, and precise Zero order derivative spectroscopy method was developed and validated for the estimation of Ciprofloxacin HCl in Bulk and pharmaceutical dosage forms. The stock solution was prepared by weighing 100 mg of standard Ciprofloxacin HCl in 100 ml volumetric flask with distilled water. The stock solution was made to produce 1000 µg/ml with distilled water. Further dilutions were prepared as per procedure. The drug solution showed the maximum absorbance at 272 nm. The linearity was found in the concentration range of 2-12 µg/ml. The correlation coefficient was found to be 0.999. The regression equation was found to be Y=0.083x+0.002. The method was validated for linearity, accuracy, precision, LOD, LOQ and ruggedness. The LOD and LOQ for estimation of Ciprofloxacin HCl was found to be 0.049µg/ml and 0.498µg/ml respectively. Recovery of Ciprofloxacin HCl was found to be in the range of 99.22-99.83 %. Proposed method was successfully applied for the quantitative determination of Ciprofloxacin Hydrochloride in Bulk and pharmaceutical dosage forms.

KEYWORDS: Ciprofloxacin HCl, Zero order UV- Spectroscopy, Distilled water, Accuracy.

INTODUCTION

Ciprofloxacin is an antibiotic used to treat a number of bacterial infections.[1] It is a second-generation fluoroquinolone with a broad spectrum of activity.[2-3] Chemically it is the monohydrochloride monohydrate salt of 1-cyclopropyl-6-fluoro-1, 4-dihydro-4-oxo-7-(1-piperazinyl)- 3-quinolinecarboxylic acid. It is a faintly yellowish to light yellow crystalline substance.[4] It functions by inhibiting DNA gyrase, a type II topoisomerase, and topoisomerase IV[5-6], enzymes necessary to separate bacterial DNA, thereby inhibiting cell division. Although human cells do not contain DNA gyrase, they do contain a topoisomerase enzyme that functions in the same manner.[7]

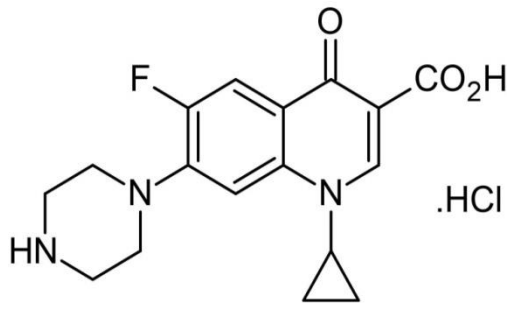

Figure 1: Chemical structure of Ciprofloxacin Hydrochloride.

It has a molecular formula of $C_{17}H_{19}ClFN_3O_3$ and molecular weight of 367.8 g/mol. It has the structural formula (Fig.1).[8]

The literature survey reveals that various analytical methods have been developed such as Stability indicating HPLC[13], UV spectroscopy methods[9-11] for the estimation of ciprofloxacin Hydrochloride present in combinational pharmaceutical formulations and Comparative purity study of UV& FTIR Techniques[12] for the determination of Ciprofloxacin Hydrochloride tablets. Most of the reported methods are using several solvents, expensive reagents and often time-consuming. Because of simplicity of UV spectrophotometry and also precise, reliable, minimum solvent usage and requires less analysis time, it is widely used for the estimation of drug content in bulk and pharmaceutical products.

A detailed review of the literature regarding the existing methods revealed that there is a need for the development of the spectrophotometric method, which is simple for the estimation of ciprofloxacin HCl present in Bulk and Pharmaceutical dosage forms. An effort was made in the present method to develop a novel, simple, sensitive, accurate, reliable and reproducible with minimum Relative Standard Deviation (RSD) values for

the estimation of ciprofloxacin Hydrochloride in Bulk and Pharmaceutical dosage forms.

MATERIALS AND METHOD

Instrument

A Shimadzu-1800 UV-Vis double beam spectrophotometer connected to a computer loaded with Shimadzu UV Probe software with 1 cm matched quartz cells was used for spectrophotometric measurements in above proposed spectrophotometric methods.

Chemicals

Ciprofloxacin Hydrochloride was given as a gift sample by yarrow Chem products Bangalore. Tablets of Ciprofloxacin HCl were procured from local market.

Solvent

Distilled water

Selection of analytical wavelength

Appropriate dilutions were prepared for drug from the standard stock solution and the solution was scanned in the wavelength range of 200-400 nm. The absorption spectra thus obtained were derivatized from Zero order method. It shows maximum absorbance at 272 nm were shown in Fig.2

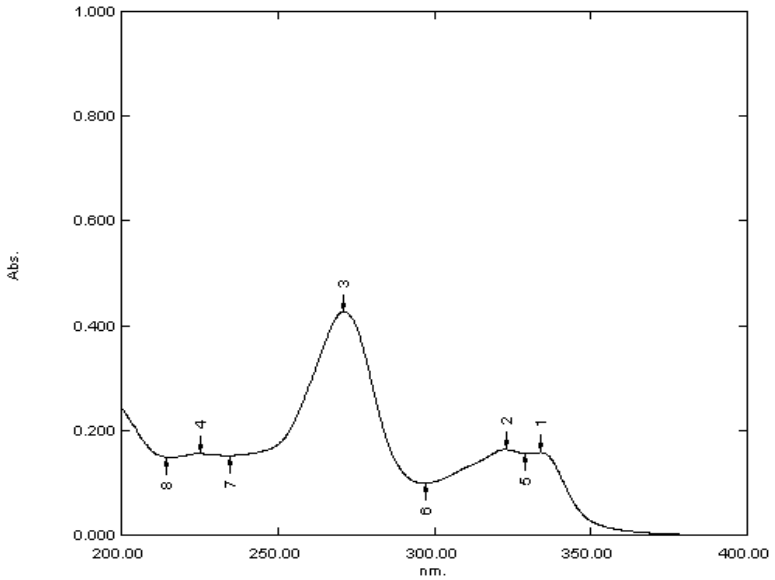

Fig. 2: Zero order spectra of Ciprofloxacin Hydrochloride showing absorbance at 272 nm.

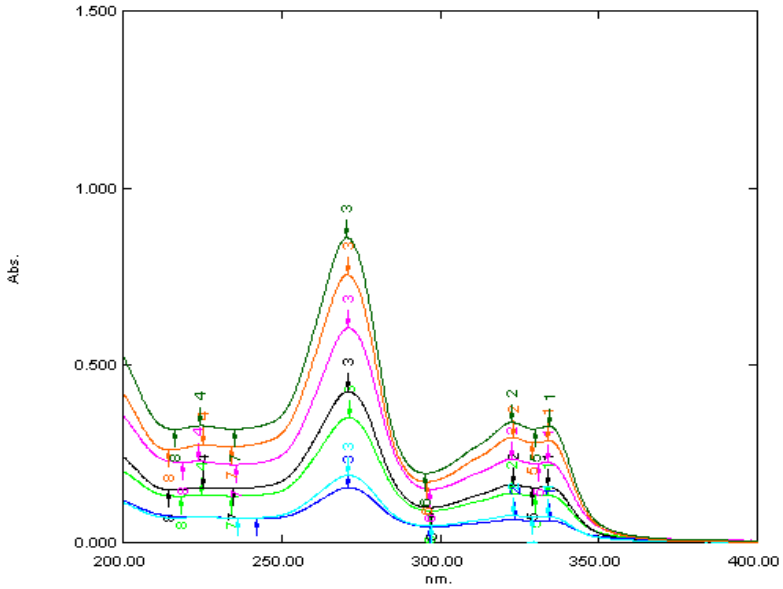

Fig. 3: Zero order overlain spectra of Ciprofloxacin Hydrochloride at 272 nm.

Preparation of Standard stock solution

Accurately weigh100mg of Ciprofloxacin Hydrochloride was transferred into 100ml volumetric flask and diluted with up to the mark. From this pipette out 10ml into 100ml volumetric flask and diluted with Distilled water up to the mark, from this solution pipette out 0.2, 0.4, 0.6, 0.8, 1.0 and 1.2ml in 10ml individual volumetric flask and add Distilled water up to the mark , this gives 2, 4, 6, 8, 10 and 12µg/ml concentrations.

Preparation of Sample solution

The commercially available Ciplox contains 300 mg of Ciprofloxacin Hydrochloride. From this twenty Tablets were weighed and powdered. The Tablet powder equivalent to 100 mg of Ciprofloxacin Hydrochloride was transferred into 100 ml volumetric flask then it was diluted with the distilled water and made up to the mark and the solution was filtered through whatman filter paper NO. 41. From the above solution 10 ml was pipetted out into 100 ml volumetric flask and the volume was made up to the mark with distilled water. The final concentration of Ciprofloxacin Hydrochloride was brought to 4 µg/ml.

Method validation

The method is validated according to the ICH guidelines.[14,15,16]

RESULTS AND DISCUSSION

Method: Zero order derivative spectroscopy

Linearity

The working standard solution were diluted serially with distilled water to obtain the range of 2-12µg/ml. a calibration curve for Ciprofloxacin Hydrochloride was obtained by measuring the absorbance at the λmax of 272nm and absorbance values are shown in Table.1 and Calibration graph were presented in Fig.3. Statistical parameters like slope, intercept, coefficient of correlation, and Sandel's sensitivity were determined and presented in Table.2.

Table 1: Results of calibration curve for Ciprofloxacin Hydrochloride at 272 nm by zero order Spectroscopy.

SL. NO	Concentration in µg/ml.	Mean Absorbance±Standard deviation
1.	2	0.167±0.0025
2.	4	0.339±0.0015
3.	6	0.511±0.0015
4	8	0.680±0.0020
5.	10	0.850±0.0020
6.	12	0.997±0.001

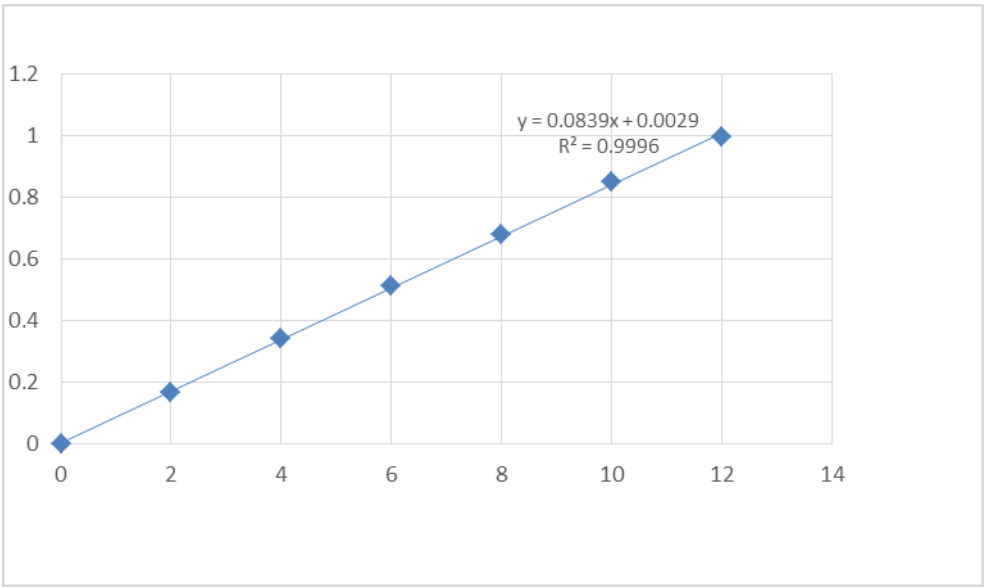

Fig.3: Calibration curve for Ciprofloxacin Hydrochloride at 272 nm by Zero order Spectroscopy.

Table no.2: Regression parameters for Ciprofloxacin Hydrochloride by zero order spectroscopy.

Optimum condition	UV Method
λmax(nm)	272nm
Beer's law limits (µg/ml)	2-12
Molar extinction coefficient ($L.mol^{-1}$ cm^{-1})	0.08303
Sandell's sensitivity(mcg / cm^2-0.001 absorbance units)	0.0117
Regression equation (Y*)	Y=0.083x+0.002
Slope (b)	0.083
Intercept (a)	0.002
Correlation coefficient(r2)	0.999

Precision

Precision of the method was studied as intra-day and inter-day precision. Intra-day precision was determined by analysing the 2, 4, 6, 8, 10 and 12µg/ml concentration for three times in same day. Inter-day precision wasdetermined by analysing the same concentration of solution daily for three days. Precision results are shown in Table.3.

Table 3: Determination of precision results for Ciprofloxacin Hydrochloride at 272 nm by Zero order derivative spectroscopy.

Concentration (µg/ml)	Intra-day Absorbance ±SD**	%RSD	Inter-day Absorbance ±SD**	%RSD
2	0.16±0.0025	1.56	0.165±0.0026	1.57
4	0.339±0.0015	0.44	0.337±0.00252	0.75
6	0.511±0.0015	0.29	0.511±0.00252	0.49
8	0.680±0.0020	0.29	0.6810±0.002	0.29
10	0.850±0.0020	0.23	0.851±0.00252	0.24
12	0.997±0.001	0.10	0.995±0.00153	0.15

Accuracy

To assess the accuracy of the proposed method, recovery studies were carried out at three different levels i. e, 50%, 100% and 150%. In which the formulation concentration was kept constant and varied pure drug concentration. Accuracy results were shown in Table.4.

Table 4: Determination of accuracy results for Ciprofloxacin Hydrochloride by Zero order derivative spectroscopy.

Tablet	Spiked levels	Amount of sample (µg/ml)	Amount of standard (µg/ml)	Amount recovered	% Recovery ±SD**	%RSD
Ciplox 300 mg	50	4	2	5.98	99.64±0.34239	0.343
	100	4	4	7.98	99.83±0.38188	0.382
	150	4	6	11.78	98.22±0.20664	0.210

Ruggedness

Ruggedness was determined between different analysts. The value of %RSD was found to be less than 2 were shown in Table.5.

Table.5: Determination of Ruggedness results for Ciprofloxacin Hydrochloride at 272 nm by Zero order Spectroscopy.

Analysts	Analyst-1	Analyst-2
Mean absorbance	0.165	0.167
Standard deviation	0.0026	0.0025
%RSD	1.57	1.49

Limit of detection and Limit of Quantitation:

The LOD and LOQ of the present method were calculated based on standard deviation of the Response and slope of linearity curve. LOD and LOQ values of Ciprofloxacin Hydrochloride were found to be 0.0498µg/ml and 0.498µg/ml.

Table 5.5: Determination of LOD and LOQ results for Ciprofloxacin Hydrochloride at 272nm by Zero order spectroscopy.

SL.NO	Parameters	Values
1	SD of Intercepts*	0.001258
2	Average of Slopes*	0.08325
3	LOD(3.3×SD of intercepts/average of slopes)	0.0498
4	LOQ(10×SD of intercepts/average of slopes)	0.498

*Mean value obtained from 6 calibration curves.

CONCLUSION

In the present investigation, we have developed Novel, simple, accurate and Precise UV- spectrophotometric method like Zero order derivative spectroscopy for the routine estimation of Ciprofloxacin Hydrochloride in Bulk and pharmaceutical dosage form and the methods were validated in terms of linearity, accuracy, precision and ruggedness.

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