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SPECTROPHOTOMETRIC DETERMINATION OF NORFLOXACIN USING CHROME AZUROL S

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ABSTRACT

An accurate, simple, fast and cheap spectrophotometric method has been developed for the determination of norfloxacin in pharmaceutical pure and dosage forms. The method is based on the reaction of the drug with chrome azurol S in citrate buffer at pH=4. This reaction produces a complex purple colored product which absorbs maximally at 518 nm. Beer's law was obeyed in the range of 6.38-63.86µg/mL with molar absorptivity of 0.354×10^4 L $mole^{-1}cm^{-1}$and the correlation coefficient R^2 =1. The effects of analytical parameters on the reported system were investigated. The results were validated statistically. The proposed method was applied to commercially available tablets. Interferences of the other ingredients and excipients were not observed.

KEYWORDS: Norfloxacin, Chrome azoul S, Complex formation, Spectrophotometry.

INTRODUCTION

Norfloxacin, [1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-(piperazinyl)quinolone-3-carboxylic acid](1) is a fluoroquinolone carboxylic acid derivative used as a broad spectrum antibacterial used for urinary infections including cystitis and prostatitis.[1] It is a synthetic antibiotic and used in the treatment of gastrointestinal or genitourinary infections. Its mechanism of action is through the inhibition of gyrase of the bacterial DNA thereby interfering with bacterial cell growth.[2,3,4] Norfloxacin is active against both gram-negative and gram-positive micro-organisms.[5] Many methods have been applied for the determination of norfloxacin such as HPLC[6,7,8], LC[9], electro analytical methods[10,11], chemiluminescence[12,13], Voltammetry[14], TLC[15], capillary electrophoresis[16], spectrofluorimetry[17], Spectro-photometric methods have been widely applied to the analysis of norfloxacin. Simple, rapid and accurate method has been described.[18] The method is based on the reaction of the drug as a p electron donor

with 2,3-dichloro-5,6-dicyanobenzoquinone,The absorbance was measured at 460 nm and the method was applied to pharmaceutical analysis. Aspectro- photometric method has been described for the determination of norfloxacin in pure and dosage forms by complexation with iron(III) and copper(II) ions.[19] UV spectrophotometry has been applied for the simultaneous estimation of norfloxacin and tinidazole.[20] Spectrophotometric has been applied for determination of norfloxacin by ion-pair complex formation with cobalt (II) tetrathiocianate.[21] 2,3,5,6- Tetrachloro-1,4- benzoquinone[22], Nd^{+3}[23], Chloranilic acid[24] have been applied to the determination of the drug in various samples. Norfloxacin was determined using bromophenol blue, The absorbance was measured at 416 nm.[25]

MATERIALS AND METHODS

All the reagents and chemicals used were of Analytical Reagent Grade. Norfloxacin was kindly supplied by GSK Pvt. Ltd. Mumbai, India. Spectral and absorbance measurements were made with UV-Vis Spectrophotometer (OpTMA SP3000 from Korea) double beam spectrophotometer with 1 cm matched quartz cell.

Preparation a standard solution of norfloxacin

A standard solution of norfloxacin (1×10^{-3} M) was prepared by dissolving 31.93 mg of norfloxacin in 10 mL of HCl (0.1N)with shaking and then completing the volume to 100 ml with deionized water, and working solutions were prepared as needed.

Preparation a solution of Chrome azuorle S

A solution of reagent Chrome azourle S (CAS) was prepared with a concentration of (1×10^{-3} M) by dissolving suitable weight of the reagent in HCl (0.1N) and completing the volume to 100 ml with deionized water.

Preparation of Buffer solutions

Different buffer Solution used 0.1M Citrate buffer, 0.1M borate buffer and 0.1M (pH=2.0-12.0) universal Britton buffer solution.

Proposed Procedure

To different aliquots of Norfloxacin (6.38–79.83µg/mL) in flasks, add 121.05µg/ml of Chrome azourle S and added 0.5ml from citrate buffer at pH=4. Transfer to 10 mL volumetric flask. Make the volume up to the mark with deionized water. Measure the absorbance of the solution at 518 nm against reagent blank.

RESULTS AND DISCUSSION

Preliminary investigations have shown that norfloxacin reacts with chrome azourle S in citrate buffer pH=4 to give the color complex, which is absorbed at a maximum of 518nm as shown in Figure (1).

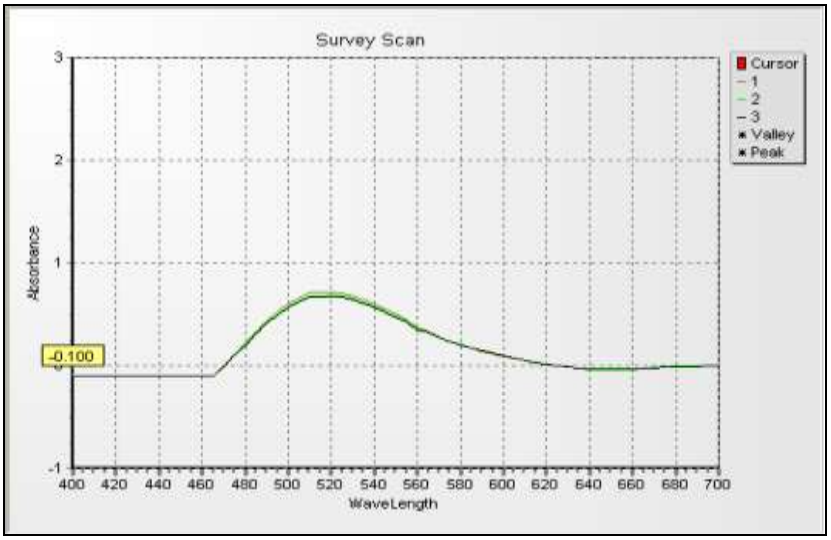

Figure (1): Absorption spectrum of NOR-CAS Formation.

To optimize the reaction conditions, different parameters have been investigated such as reagent concentration, color stability, pH buffer and amount of buffer (pH =4).

Effect of time on the stability of the color (NOR-CAS) complex

Developed color was stable up to 4 hours which was considered sufficient time for an analyst to carry out analysis (Figure 2).

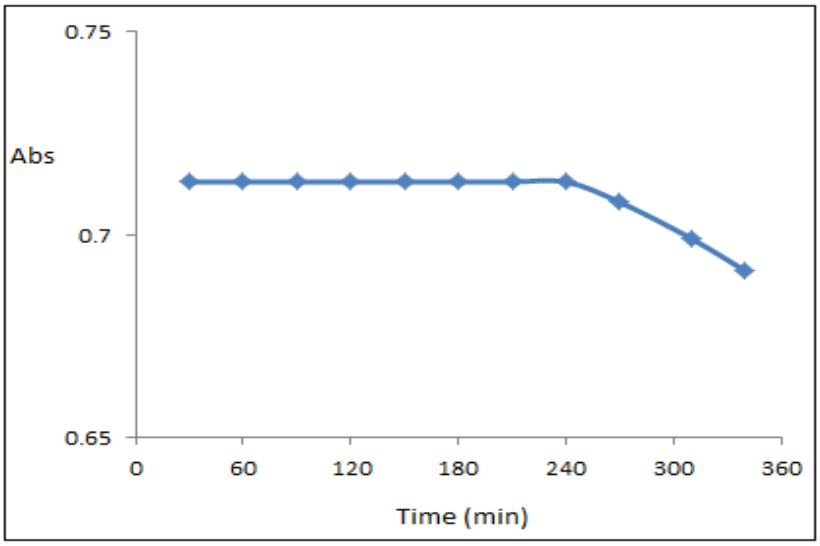

Figure (2): Effect of time on the color development.

Effect of pH buffer

The effect of pH was studied in the presence of various buffers such as Briton, Citrate and borate. It was observed that the maximum color intensity and constant absorbance were found in citrate buffer solution (0.1M) of pH= 4 for (NOR-CAS) system using 0.5 ml of citrate buffer solution (0.1M) as shown in (Figure 3).

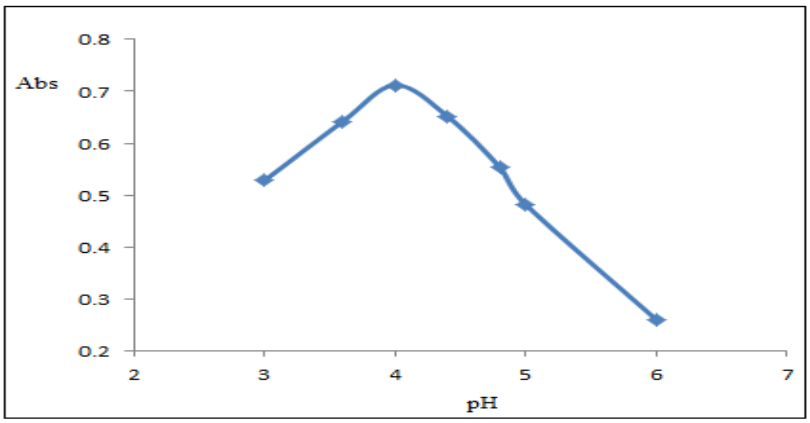

Figure (3): Effect of the pH value on absorption of NOR-CAS complex.

Effect of amount of citrate (0.1M) buffer (pH= 4)

The optimum of amount of citrate buffer solution (0.1M) for the assay of drugs was studied. 0.5 ml of citrate buffer solution (0.1M) pH=4 sufficient for complete color development for NOR-CAS complex as shown in (Figure 4).

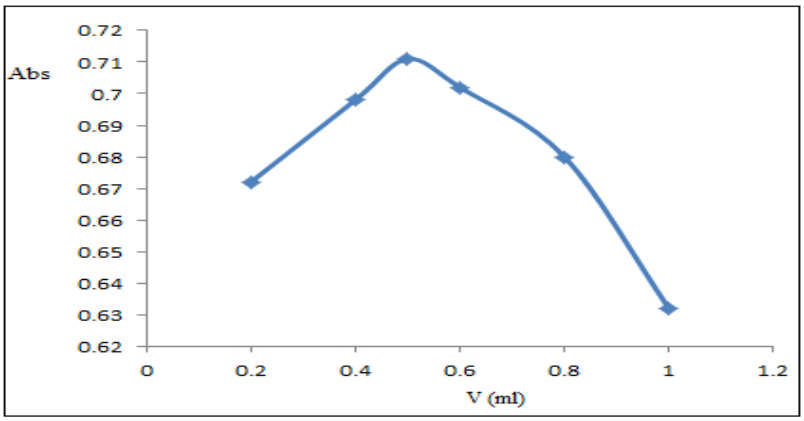

Figure (4): Effect of amount of citrate(0.1M) buffer (pH 4).

Effect of reagent concentration

The effect of Chrome azuorle S (CAS) concentration on the color development was investigated. 2.0 mL of Chrome azuorle S (10^{-3}M) reagent produced maximum color intensity (Figure 5).

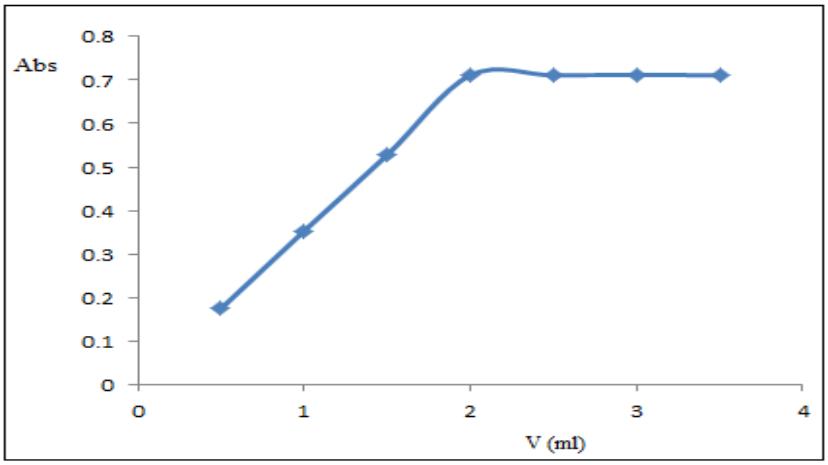

Figure (5): Effect of the amount of(CAS) on absorbance of NOR-CAS complex.

Molar Ratios Determination of NOR-CAS complex

The molar ratio was determined using the molar ratio methods[26] and continuous variation[27] methods. the ratio were found to be 1:1 for NOR:CAS (Figures 6 and 7).

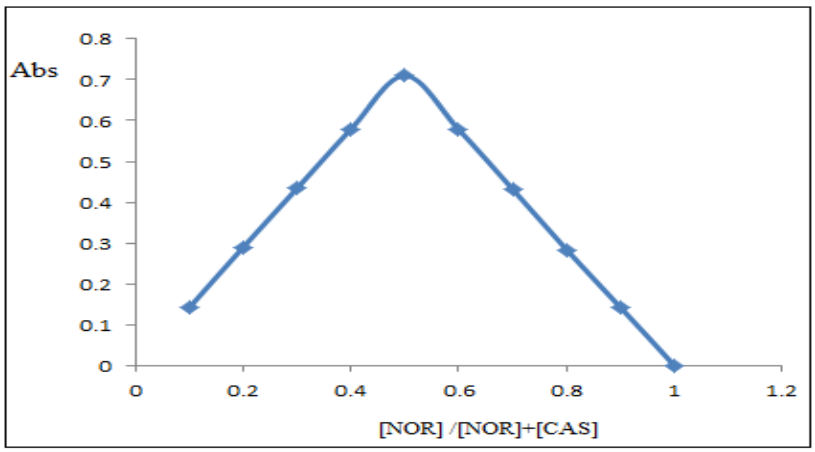

Figure (6): Continuous Variation plot for NOR-CAS complex.

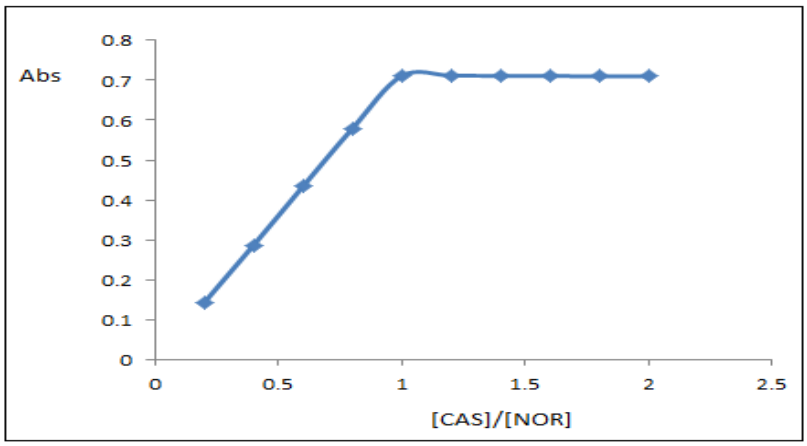

Figure (7): Molar ratio plot for NOR-CAS complex.

Linearity and Sensitivity

A linear relation was obtained between absorbance and concentration of NOR in the range of 6.38–63.86 µg/mL (Figure 8). The graphs show negligible intercept and they are described by the regression equation, A= mC + b (where A is the absorbance of 1 cm layer, m is the slope, b is the intercept and C is the concentration of the measured solution in µg.ml^{-1}) obtained by the least-squares method.[28] The high molar absorptivity of the resulting colored complex indicate the good sensitivity of the method. The Beer's law limits, Sandell sensitivity, molar absorptivity, linear regression equation, correlation coefficient and detection limit[29] determined for the method are given in Table 1.

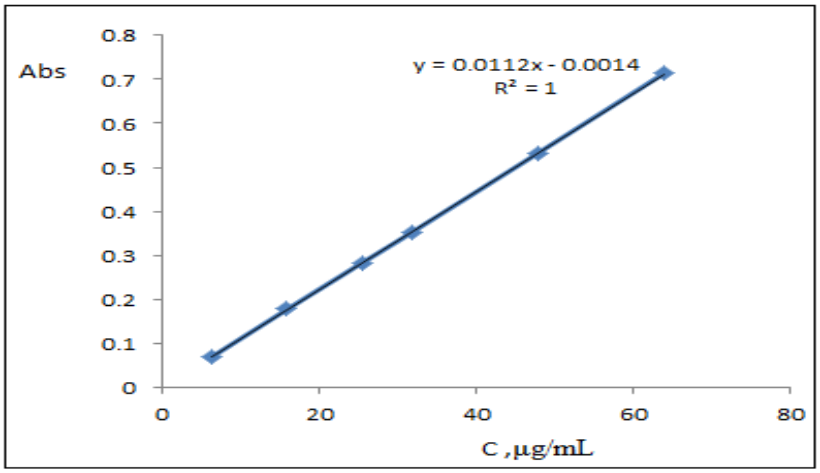

Figure (8): Calibration rang for Norfloxacin.

Table(1): Optical characteristics and statistical data for the regression equation of the proposed method.

Parameter	Value
λ_{max} (m)	518
Beer's law limit (µg/mL)	6.38–63.86
Molar absorptivity (L mole^{-1} cm^{-1})	0.354×10^{4}
Sandell's sensitivity (µg/mL per0.001 A)	0.09
Slope (m)	0.0112
Intercept (c)	0.0014
Correlation coefficient	1
Relative Standard Deviation*	1.73
Limit of Detection (µg/mL)	0.967
Limit of quantitation (µg/ml)	3.22

Y = mx + C

Where X is the concentration of analyte (µg/mL) and Y is absorbance unit.

* = Calculated from five determinations

Accuracy and Precision

The results obtained are summarized in Table 2. The low values of relative standard deviation (RSD) indicate good precision and reproducibility of the method. The average percent recoveries obtained were 95.55 – 100.83%, indicating good accuracy of the methods.[30-31]

Table (2): Study of the precision and of the accuracy of the method.

CRT Taken (µg/ml)	CRT Found* (µg/ml)	Standard Deviation SD	Relative Standard Deviation RSD %	analytical Error SD/(n)$^{1/2}$	Confidence limit (µg/ml)	Recovery% (%) R
6.38	6.35	0.116	1.73	0.049	6.35±0.136	99.52
15.96	15.90	0.144	0.90	0.064	15.90 ±0.177	99.62
25.54	25.72	0.340	1.32	0.152	25.72±0.421	100.70
31.93	31.65	0.421	1.33	0.188	31.65±0.521	99.12
47.89	47.25	0.390	0.82	0.174	47.25±0.484	98.66
63.86	63.65	0.218	0.34	0.097	63.65±0.26	99.67

* Average of five determinations.

Application to the pharmaceutical dosage forms

The proposed method has been successfully applied to the determination of NOR in pharmaceutical preparations Table 3. The ingredients in the tablets did not interfere in the experiments.

Table (3): Results of the estimation of NOR in tablets.

Formulation	Label claim (mg)	NOR Taken (µg/ml)	NOR Found (µg/ml)	Standard Deviation SD	Content determined* (mg)	Relative Standard Deviation RSD %	Recovery% (%) R
Uriflox	400	20	20.12	0.258	401	1.28	100.6
Noroxacine	400	20	20.15	0.193	402	0.96	100.7

*Five independent analyses

CONCLUSION

The reported method is rapid, simple, economical, and fairly sensitive. It can be used in routine analysis of pharmaceutical formulations of Norfloxacin in quality control laboratories. Moreover, the present method can be directly applied to the pharmaceutical sample without prior separation or treatment.

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