Using the Second Derivative of the Zero-Order Spectrum for the Quantitative Determination of Ciprofloxacin in its Pure Form and Pharmaceutical Preparations

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Abstract

A new, cost-effective, rapid, and precise spectrophotometric method was developed for the quantitative determination of ciprofloxacin in its pure form and some pharmaceutical formulations. This method utilizes the second derivative of the zero-order spectrum. The study demonstrated that this technique enables the quantitative determination of ciprofloxacin within a concentration range of 5–100 µg/mL. The peak height above the baseline at the wavelength of 316 nm was utilized for measurements. The results indicated that the method is accurate and exhibits good reproducibility, with a recovery percentage ranging from 99.5% to 102.5% and a relative standard deviation (RSD%) of 2.5–2.807 for the respective wavelengths. The method was successfully applied to several pharmaceutical formulations.

Introduction

Ciprofloxacin is an antimicrobial agent and a member of the quinolone class, exhibiting activity against both Gram-positive and Gram-negative bacteria. It has broad clinical applicability [(1).] Ciprofloxacin inhibits cell division by targeting bacterial DNA replication through binding to an enzyme known as DNA gyrase, thereby preventing DNA replication, which is essential for bacterial DNA differentiation. Ciprofloxacin can be administered orally or intravenously [(2)].

Following its discovery in 1962, nalidixic acid became a prominent treatment for urinary tract infections caused by Gram-negative bacteria. However, its indication for systemic infections was later limited due to the development of antibiotic resistance in many microorganisms [(3)]. The scientific name of the drug is:

1-cyclopropyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperazinyl)-3-quinolinecarboxylic acid [(4)].

Figure 1 illustrates the chemical structure of the drug.

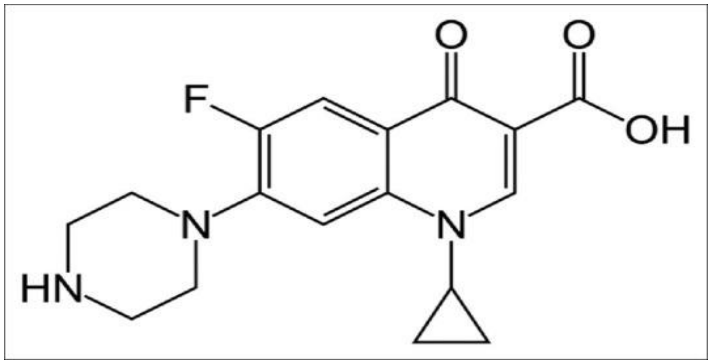

Figure 1: The Chemical Structure of Ciprofloxacin [(5)]

A review of the scientific literature focusing on drug quantification highlights the significant interest of many researchers in quantitatively determining ciprofloxacin in its pure form and pharmaceutical formulations due to its medical importance. Ciprofloxacin has been quantified using various techniques, including capillary electrophoresis [6][7], titration [8], and high-performance liquid chromatography (HPLC), which is commonly employed for the quantification of ciprofloxacin in drugs [9][10], urine, and plasma [11][12], as well as in animal tissues [13]. Other methods include ultraviolet spectrophotometry [14] and several additional techniques.

In this study, the derivative spectrum method was employed to quantitatively determine ciprofloxacin in selected pharmaceutical formulations. The method proved to be cost-effective, accurate, sensitive, and did not require prior separation procedures or extensive use of chemicals.

Scientific Section

Instruments

For the measurements, the following instruments were used: UV-Vis spectrophotometer (Shimadzu-1800, double beam), Precision balance (Germany – Sartorius), Canon laser printer (Model 2900) for recording spectra.

Absorption spectra of the drug were recorded at a medium scan speed using a 1 cm quartz cell within the wavelength range of 190–350 nm.

Solutions

To prepare a stock solution of 1000 µg/mL, 0.1 g of ciprofloxacin (Indian origin, obtained from the local market) was dissolved in a sufficient amount of distilled water, and the volume was adjusted to the mark using the same solvent in a 100 mL volumetric flask.

Similarly, a solution of 1000 µg/mL was prepared from the pharmaceutical formulation CIPROSAM 500 (tablets containing 500 mg ciprofloxacin, manufactured by SDI). This was achieved by dissolving the average weight of one tablet in an appropriate amount of water, then adjusting the volume to the mark in a 10 mL volumetric flask.

Methodology

Various aliquots of the ciprofloxacin standard solution, containing 50–1000 µg, were transferred into a series of 10 mL volumetric flasks, and the volumes were adjusted to the mark with distilled water. The absorption spectra were recorded against the blank (distilled water). Subsequently, the required derivation process was applied to the zero-order spectra to obtain the second derivative spectra for concentrations ranging from 5–100 µg/mL.

Results and Discussion

Absorption Spectra

The absorption spectra of ciprofloxacin (5–100 µg/mL) were recorded against the blank solution over a wavelength range of 190–380 nm, with a medium scan speed, a rate of change of 0.1 nm, and a bandwidth of 2 nm. Figure 2 illustrates the absorption spectra of the drug, which exhibit a peak at the wavelength of 316 nm.

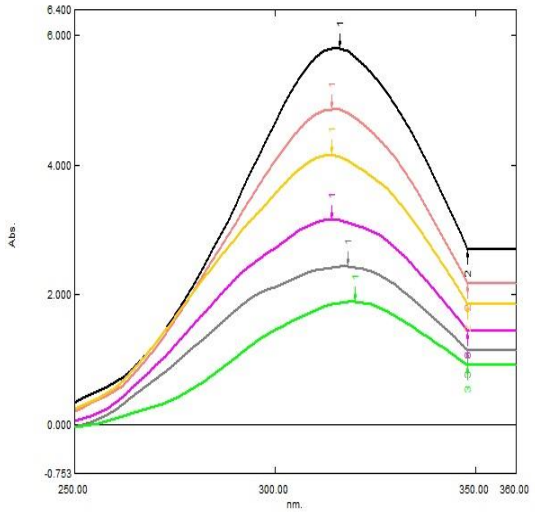

Figure 2: Absorption Spectra of Ciprofloxacin at Different Concentrations

Second Derivative Method

The derivative spectrophotometric technique is particularly useful for determining the concentrations of target substances in single-component estimations, even in the presence of pharmaceutical additives or complex mixtures, despite significant overlaps in absorption spectra [(15)].

In this study, the second derivative spectra were applied, and the results demonstrated that the method could be successfully implemented under optimal operational conditions such as bandwidth, scan speed, and rate of change. Multiple measurements were conducted on the second derivative spectra recorded under the optimal operating conditions of the spectrophotometer. These included peak height above the baseline and peak area, which were used for the quantitative analysis of ciprofloxacin in its pure form and pharmaceutical formulations. The second derivative spectra were derived from a series of solutions with varying concentrations (5–100 µg/mL) of ciprofloxacin. The results showed a direct proportionality between ciprofloxacin concentration and the measured peak height at 316 nm, as well as the peak area between 308–326

nm. The second derivative spectra of the drug were recorded, and Figure 3 illustrates these spectra for a range of different concentrations.

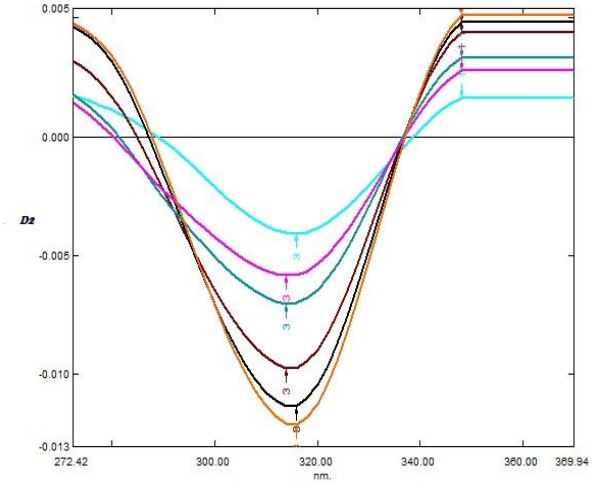

Figure 3: Second derivative spectra of ciprofloxacin at different concentrations.

Calculations and Calibration Curves

Given toFor the analytical properties and most of the statistical data of each of the proposed methods and under the conditions ofMy preferenceThe linearity of the graphs of the calibration curves was obtained.AndWhich ranged between (5-100(mcg/ml) while the estimation coefficient values ranged from0.9964 and 0.9988) and detection limit values (3.87- 4.11) for the indicated measurement areas. Peak height above the baseline (316 nm).The summit area is (308-326)Table 1 shows the equation of the straight line as well as the slope for the indicated areas.

Table 1: Drug analysis resultsCiprofloxacinBy derivativesecond

Compound	**Order of derivative**	**Mode of calculation**	**Concentration range**	**λ(nm)**	**Regression question**	**R2**	**LOD**
Ciprofloxacin	Second	Peak to baseline	5-50 µg/ml	316	y = -0.0001x - 0.001	0.9964	3.87
		Peak area		308-326	**y = -0.0002x - 0.0039**	0.9988	4.11

Accuracy and compatibility

Focus selected (30(mcg/ml) to findtheAccuracy andtheagreetoFor the methodSuggestedThe results showed that the method has good accuracy and consistency, as the value ranged fromRSD% (2.846-4.109) and valueRec% (100-101.67) for the drugCiprofloxacinAs in Table (2).

table2: Method accuracy and consistency

Compound	**Order of derivative**	**Mode of calculation**	**Drug Conc. µg/ml**		**Rec%**	**RSD%**
			Taken	**Found**		
Ciprofloxacin	Second	Peak to baseline of 316nm	30	30	100	**4.109**
		Peak area 308-326	30	30.5	101.67	**2.846**

Apply the method

The derivative pattern has been applied.secondFor the areas selected to work on (peak height relative to baseline)And the summit area) For direct estimation of the propertyCiprofloxacinIt was successful. Table 4 shows the results of the analysis of a number of different pharmaceutical preparations of the drug using the proposed method.

Table 3: Analysis resultsOneThe preparationatPharmacistAndContainer of the drugCiprofloxacin.

Pharmaceutical	**Order of derivative**	**Mode of calculation**	**λ**	**Drug Conc. µg/ml**		**Rec%**
				Taken	**Found**	
CIPROSAM 500mg SDI/IRAQ	Second	Peak height	316	20	20.5	102.5
		Peak area	308-326	20	19.9	99.5

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