SHORT COMMUNICATIONS

Fourth Derivative Spectrophotometric estimation of Ciprofloxacinamide and N,N'- bis Hydroxymethylciprofloxacinamide in presence of Ciprofloxacin

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A simple fourth derivative spectrophotometric method for determination of ciprofloxacinamide and N,N'-bishydroxymethyl ciprofloxacinamide in presence of ciprofloxacin is presented. The procedure consists of measuring the amplitudes of the fourth derivative spectra of ciprofloxacinamide and ciprofloxacin at 286 nm and 277.4 nm respectively and that of N,N'-bishydroxymethyl cirpofloxacinamide and ciprofloxacin at 286 nm and 276.9 nm, respectively. Zero crossing technique is used for these estimations.

THE simulatenous determination of ciprofloxacin(I) and ciprofloxacinamide(II) or (I) and N,N'- bishydroxymethylciprofloxacinamide(III) was not possible by direct UV-absorption measurements because of the spectral overlap of the main maximas of the compounds. The three compounds show UV-absorption maxima at 276.0 nm (I), 283.0 nm (II) and 282.4 nm (III). Derivative spectrophotometry[1,2] finds many applications in pharmaceutical analysis for the analysis of multicomponent formulations,[3-6] overcoming interference from excipients in the formulations and body fluids[7,8] and mixtures of similar compounds[9]. None of the spectrophotometric methods[10,11] reported for analysis of (I) are applicable to the simultaneous analysis of (I) and (II) or (I) and (III).

Shimadzu UV - 160A was the recording spectrophotometer employed for this work. Stock standard solutions of (I), (II) and (III) of strength 25 mcg/ml each were made in 0.01 N hydrochloric acid. For the simultaneous determination of (I) and (II) calibration curves were plotted in the concentration range of 0.0 to 12.5 mcg/ml by measuring the absorbance at 277.4 nm and 286 nm respectively from the fourth derivative spectra of each dilution. Similarly, for curves were plotted in the concentration range of 0.0 to 12.5 mcg/ml by measuring the absorbance at 276.9nm and 286 nm respectively from the fourth derivative spectra of each dilution. Five sample solutions of each of the three compounds were analysed and the results of analysis are stated in Table - 1. Similarly five mixed sample solutions of (I) and (II) and of (I) and (III) were analysed and the resulted are stated in Table - 1.

This proposed method was found to be accurate, rapid and simple for simultaneous estimation of (I) and (II) and of (I) and (III). The difference in the UV-absorption maxima of (I) and (II) was only 7.0 nm and that in the UV-absorption maxima of (I) and (III) was only 6.4 nm. Direct spectrophotometric estimation of any compound in presence of varying concentrations of the other was not at all possible as large errors were introduced due to interference. After observing the first, second, third and fourth order derivative spectras of the three compounds the fourth order derivative spectra was selected for this work keeping in view resolution and sensitivity.

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Table - 1 : Analysis of pure and mixed samples

Pure Samples						Mixed Samples					
Conc. Present		Conc. Estimated		% Estimated		Conc. Present		Conc. Estimated		% Estimated	
I	II	I	II	I	II	I	II	I	II	I	II
2	2	1.964	1.982	98.20	99.10	2	8	1.948	8.040	97.40	100.50
4	4	3.952	4.026	98.70	100.65	4	6	3.896	6.182	97.40	103.03
6	6	5.988	5.990	99.80	99.84	5	5	4.996	5.164	99.92	103.28
8	8	7.950	7.876	99.38	98.45	6	4	5.760	3.988	96.00	99.70
10	10	9.993	10.008	99.93	100.08	8	2	7.824	1.954	97.80	99.20
				Mean % 99.20	99.62					Mean % 97.70	101.14
				Std. deviation 0.737	0.860					Std. deviation 1.415	1.897
I	III	I	III	I	III	I	III	I	III	I	III
2	2	2.025	1.980	101.25	99.00	2	8	2.015	7.805	100.75	98.50
4	4	4.002	3.992	100.05	99.80	4	6	4.080	5.935	102.00	98.90
6	6	5.984	6.032	99.73	100.52	5	5	5.075	5.082	101.50	101.64
8	8	8.012	8.016	100.15	100.20	6	4	5.889	4.124	98.15	103.10
10	10	10.016	9.990	100.16	99.90	8	2	7.849	2.035	98.12	101.75
				Mean % 100.26	99.88					Mean % 100.10	100.77
				Std. deviation 0.576	0.568					Std. deviation 1.857	1.987

When the concentration related absorbance data from the fourth derivative spectra of the three compounds was transformed into standard curves, linear curves were obtained in the concentration range of 0 - 12.5 mcg/ml. Minimum analysable concentration limit was 1.0 mcg/ml for (I) and 1.5 mcg/ml for (II) and (III). Upto 10.0 mcg/ml of (II) or (III) did not interfere with the estimation of (I) and upto 10.0 mcg/ml of (I) did not interfere with the estimation of (II) or (III).

The reproducibility of the methods is also satisfactory as indicated by the low values of standard deviation. The standard deviation values were a little higher in analysis of the mixed samples as compared to those obtained from analysis of pure samples.

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Spectrophotometric determination of Cimetidine in pure form and in dosage forms using Cu^{2+}

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A simple and selective spectrophotometric method has been developed for the determination of cimetidine in pure form and in dosage forms using Cu^{2+} solution.

CIMETIDINE is a highly effective drug for the treatment of deuodenal ulcer and is a well established H_2 receptor. Nitrogen content determination by Kjeldahe method[1] is a widely used estimation method for cimetidine. In addition, spectrophotometric[2,4] chromatographic[5] and titrimetric methods[6-9] are also available for the purpose.

Cimetidine forms a bright green complex with Cu^{2+} at a pH of 2-7. This properly is exploited for developing an analytical method for the determination of pure cimetidine and five of its commercially available preparations. The method reported here is quite simple.

A Shimadzu UV-Visible Spectrophotometer with autocalculation provision is used for the analysis. Cimetidine sample was recrystallised from ethanol and its purity was confirmed by m.p. determination. The sample was made into a solution by dissolving a known mass of cimetidine (0.15 g) in water and the solution was diluted to a predetermined volume (250 ml). Five commercially available cimetidine tablets were taken for analysis. Ten tablets of each type were weighed accurately. They were finely powdered and known mass (0.15 g) of the powder was dissolved in

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