

was less than 1% in all the batches. The time required for complete wetting was found to be between 8 to 11 s. All the tablet formulations disintegrated rapidly *in vitro* within 10-16 s and *in vivo* within 12-16 s. The tablets containing SSG and CMC showed faster disintegration than containing CMC and treated agar. The release rate of sumatriptan succinate from the formulations F1, F3, F5 was found faster than F2, F4, F6 and conventional formulation (Suminat® 50mg, Natco Pharma Ltd., Hyderabad). The tablets, apart from fulfilling all official and other specification, exhibited faster release rates of sumatriptan succinate.

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A new Spectrophotometric Estimation of Chloroquine Phosphate from Tablets

N. RAMI REDDY¹, K. PRABHAVATHI AND I. E. CHAKRAVARTHY*

Department of Chemistry, S. K. U .P. G. Centre, Kurnool-518002.

¹Department of Chemistry, S. B. S. Y. M. Degree College, Kurnool-518004.

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A simple spectrophotometric method for determination of chloroquine was described. The method was based on bromination of the drug with excess brominating mixture in acidic medium. The yellow colour developed was measured at 350 nm against distilled water blank. Beer's law was obeyed between 40-200 µg/ml.

Chloroquine phosphate, chemically (RS)-4-(7-chloro-4-quinolyl amino)-pentyl diethylamine diphosphate, used as an antimalarial and antiamebic. IP¹ has reported non-aqueous method, while BP² has described titrimetry using 0.1 M sodium hydroxide as a titrant. Other methods of estimation include colorimetry^{3,4}, spectrophotometry⁵⁻⁷, spectrophotometric microdetermination⁸ and spectrophotometric charge transfer complexation^{9,10}.

In the present communication, a simple spectrophotometric method has been developed for the estimation of chloroquine phosphate from pharmaceutical preparations. The proposed method was based on the

bromination of the drug with excess brominating mixture in acidic medium. After bromination, the excess brominating mixture was treated with potassium iodide, gives yellow colour. The maximum absorbance was measured at 350 nm. The proposed method has not been studied earlier for estimation of chloroquine phosphate in tablets.

All measurements were done on a Milton Roy 1001 plus spectrophotometer using 10 mm matched quartz cuvettes. All analytical grade chemicals were used and all of the solutions were freshly prepared with double distilled water. 4 N hydrochloric acid was prepared and standardized with standard procedure. Potassium iodide (0.1 N) was prepared by dissolving 0.166 g in 100 ml distilled water. Brominating mixture solution (0.1 N) was prepared by

*For correspondence

dissolving 0.695 g of potassium bromate and 1.75 g of potassium bromide in distilled water and diluted to 100 ml with distilled water. Further dilution takes place to obtain the working concentration of 0.02 N brominating mixture solution.

One hundred milligrams of pure chloroquine phosphate was dissolved in methanol and diluted to 100 ml with methanol. This stock solution was further diluted to get desirable working concentration of 200 µg/ml.

The following procedure has been adopted for obtaining the standard curve. An aliquot of 0.2, 0.4, 0.6, 0.8 and 1.0 ml of the drug solution was transferred into a series of 25 ml standard flasks. To each flask, 1 ml of 4 N hydrochloric acid and 1 ml of 0.02 N brominating mixture were added. The flasks were shaken well and kept aside for 5 min for complete bromination. Then 0.1 N potassium iodide were added to each flask and diluted to 25 ml with distilled water. The yellow colour solution formed was measured at 350 nm against distilled water as blank. The calibration curve was obtained by plotting absorbance values against amount of standard drug in µg/ml. The amount of drug present in the sample was read from calibration curve. The calibration curve was found to be linear over a concentration range of 40-200 µg/ml.

Tablets were weighed, powdered and the powder equivalent to 50 mg of chloroquine phosphate was dissolved in methanol, filtered, residue was washed with distilled water and the volume of the filtrate was adjusted to 50 ml with methanol. This solution was further diluted as described under preparation of the standard solution. Further analysis was carried out as per the procedure described under calibration curve and the amount of chloroquine present in

each sample was estimated from calibration graphs. The results are tabulated in Table 1.

In order to study the accuracy and suitability of the proposed method, known quantities of chloroquine phosphate were added to previously analyzed samples and the same mixtures were reanalyzed using the proposed method. Percent recovery was calculated using the equation, $\% \text{ recovery} = \frac{N(\sum XY) - (\sum X)(\sum Y)}{N(\sum X^2) - (\sum X)^2} \times 100$, where, X is the amount of standard drug added, Y is the amount of drug found (µg/ml) and N represented the total number of observations. The results are presented in Table 1.

The present study was carried out to develop a simple, rapid, precise and reproducible spectrophotometric method for the estimation of chloroquine phosphate in pharmaceutical formulations. Chloroquine phosphate in slightly acidic conditions undergoes bromination with brominating mixture. After bromination was completed, the excess brominating mixture was treated with potassium iodide to form a yellow coloured complex of potassium iodate having maximum colour sensitivity and stability. The colour obeyed Beer's law in the concentration range of 40-200 µg/ml. The experimental conditions were optimized by studying the effect of brominating mixture, hydrochloric acid, and potassium iodide by sequence of addition. The recovery studies conducted by addition of different amount of pure drugs to a reanalyzed sample solution and data are tabulated in Table 1. The recovery values were ranged from 99.6 to 100.2% indicates the accuracy of the method. Common excipients such as, lactose, starch, calcium lactate and gum acacia did not interfere with the assay. Statistical analysis of various parameters was performed and the results are summarized in Table 2. The values of standard deviation

TABLE 1: ESTIMATION OF CHLOROQUINE IN TABLETS

Sample	Labeled amount (mg)	% of amount found in mg		% Recovery*
		Proposed method*	Official method*	
Tablet 1 ^a	500	500.5	500.2	99.70
Tablet 2 ^b	250	249.6	249.8	99.60
Tablet 3 ^c	500	499.4	499.0	100.2
Tablet 4 ^d	250	249.8	248.9	99.90

*Average of five determinations based on label claim. ^aClo-kit marketed by Indoco, Mumbai, ^bLariago marketed by IPCA, Mumbai. ^cResochin marketed by Bayer India, Mumbai and ^dEmquin marketed by Merck India, Mumbai.

TABLE 2: STATISTICAL ANALYSIS OF ESTIMATION OF THE CHLOROQUINE

Sample	Labeled amount	Standard Deviation*	Coefficient of variation*	t_{cal}^a	t_{tab}^a
Tablet 1	500	0.4082	0.0816	1.2733	2.132
Tablet 2	250	0.4921	0.1972	1.5480	
Tablet 3	500	0.3399	0.0679	1.3250	
Tablet 4	250	1.080	0.4328	0.8013	

*Average of three determinations based on label claim, t_{tab}^a = Tabulated value or Theoretical value.

and coefficient of variation were satisfactorily low, indicates the reproducibility of the method. Results obtained with analysis of commercial formulations have also been subjected to statistical evaluation using student's 't' test to further evaluate the proposed method. The 't' values were found to be less than 't' theoretical with 4 degrees of freedom at 5% level of significance indicating that there is no significant difference between proposed method and the reference method. The proposed spectrophotometric method was found to be simple, precise, accurate and less time consuming. Therefore, it may be preferably used for routine analysis of chloroquine phosphate in pharmaceutical formulations.

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HPTLC Standardization of *Gymnema Sylvestre* R. Br. Using Gymnestrogenin as Reference

V. PURATCHIMANI AND S. JHA*

Department of Pharmaceutical Sciences, Birla Institute of Technology,
Mesra, Ranchi-835215.

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A simple and reproducible high performance thin layer chromatography method for the determination of gymnestrogenin in *Gymnema sylvestre* was developed and is described. This method involves separation of compounds by TLC on pre-coated silica gel 60F 254 plates with a solvent system of chloroform: methanol (9:1) and scanned using densitometric scanner in UV reflectance photomode at 293 nm. The linearity was observed in the range of 4 to 10 μ g. The gymnestrogenin content of 1.11 % w/w was observed in test sample. The average percentage

*For correspondence