sample solution was loaded in sample loop of the injection port of the instrument. The solution was injected and chromatogram recorded. The injection was repeated three times and peak area of suprofen and caffeine were recorded. The peak area ratio of drug to internal standard was calculated and amount of drug present in bulk drug sample determined using calibration curve. The results of analysis are reported in Table -1.

In present work two methods have been developed for estimation of suprofen from bulk drug sample. The first one is a colorimetric method, which is based on formation of chloroform extractable coloured complexes of the drug with copper (II) acetate. Conditions required for formation of coloured complex were optimised. The method was found to be simple, accurate and economical. Percentage recovery using this developed method was found to be in range of 98-100% and standard deviation below 0.60. The second method is a reverse phase HPLC method using C_{18} column. The method was developed using caffeine as internal standard. The total run time for the method was just 15 min and difference between retention time of drug and internal standard was more than 10 min. Percentage recovery of the method was close to 100% and standard deviation below 0.10. Since no formulation of suprofen was available in Indian market, analysis of suprofen from a formulation could not be carried out. However, developed methods could with minor modifications perhaps be used for estimation of suprofen from its formulation.

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Spectrophotometric Methods for the Determination of Sparfloxacin In Pharmaceutical Dosage Forms

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Two simple and sensitive spectrophotometric methods (method A and method B) have been developed for the determination of sparfloxacin in bulk and in pharmaceutical dosage forms. Method A is based on an observation that methanolic solution of sparfloxacin exhibits an absorbance maximum of 295.2 nm and method B is based on diazotisation of sparfloxacin with nitrous acid followed by its coupling with resorcinol in alkaline medium, to form a colored chromogen with an absorbance maximum of 450 nm. The methods are statistically validated and found to be precise and accurate.

Sparfloxacin is a recently developed fluoroquinolone drug which is extremely useful in treating many infections[1,2]. It has broad spectrum of activity against gram positive and gram negative organisms[3]. Chemically, sparfloxacin is 5-amino-1-cyclopropyl-7-(cis 3,5-dimethyl-1-piperazinyl)-6,8-difluoro-1,4-dihydro-4-oxo-3-quinoline carboxylic acid and not yet official in any pharmacopoeia.

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HPLC[4-7] and a few spectrophotometric[8-11] methods are reported earlier for the determination of sparfloxacin in biological fluids and in dosage forms. In the present communication, two spectrophotometric methods have been described.

An ELICO UV-Vis spectrophotometer model SL-150 with 1. cm matched quartz cells was used for spectral measurements. All the chemicals used were of AnalaR grade.

Aqueous solutions of hydrochloric acid (5 N), sodium nitrite (0.1% w/v), resorcinol (0.02% w/v) and sodium hydroxide (1N) were prepared in the usual way. Sparfloxacin was obtained as a gift sample from Cadila Healthcare Ltd., Ahmedabad.

A standard solution containing 1 mg/ml of sparfloxacin was prepared in methanol. From this, working standard solutions are prepared by dilution with methanol (10 µg/ml for method A and 400 µg/ml for method B).

Four brands of commercial tablets were analysed by the proposed methods. In each method, 5 tablets were accurately weighed and powdered. In each case tablet powder equivalent to 50 mg of sparfloxacin was treated with sufficient quantity of methanol and diluted to 50 ml with the same solvent and filtered. The filtrate was suitably diluted and analysed as given under the assay procedures for bulk sample.

Method A comprises of transferring into a series of 10 ml volumetric flasks, aliquots of sparfloxacin (0.2-1 ml, 10 µg/ml) followed by adjusting the volume to the mark with 0.1 N hydrochloric acid. The absorbance was measured at 295.2 nm against the blank. The concentration of sparfloxacin was deduced from the calibration graph.

In method B, aliquots of 0.1-1 ml of standard drug solution was transferred into a series of 10 ml volumetric flasks. Then 0.5 ml of hydrochloric acid, 1 ml of sodium nitrite, 1.5 ml of resorcinol and 0.5 ml of sodium hydroxide were successively added to each flask. The volume was made upto the mark with distilled water. The absorbance was measured at 450 nm after 5 min against the reagent blank prepared in a similar manner. The concentration of sparfloxacin was deduced from the calibration graph. Recovery experiments were performed by adding known amounts of the drug to previously analysed pharmaceutical preparations and also to various excipients used in formulations. The results are presented in Table -2.

Beer's law limits, molar absorptivity, sandell's sensitivity, slope and intercept of regression analysis using the least square method, precision and accuracy of the analysis of six replicate samples containing 3/4th of the amount of upper Beer's law limits in each method were

TABLE 1 : OPTICAL CHARACTERISTICS OF SPARFLOXACIN USING PROPOSED METHODS

Parameter	Method A	Method B
λ_{max} (nm)	295.2	450
Beer's law limit (µg/ml)	0.2-1.0	0.40
Molar absorptivity (lit.mole^{-1}.cm^{-1})	3.92×10^4	8.14×10^3
Sandell's sensitivity (µg/cm^2/0.001 abs.unit)	0.010	0.0480
Regression Equation*		
Slope (b)	1×10^{-2}	1.9×10^{-3}
Intercept (a)	4×10^{-3}	3.1×10^{-2}
Correlation Coefficient (R)	0.9987	0.9991
% RSD**	2.32	0.781
% Range of error (0.05 level)**	1.939	0.653

* Y = a+bX, where X is the concentration of sparfloxacin in µg/ml and Y is the absorbance at the corresponding λmax. ** Average of six determinations

TABLE 2 : ANALYSIS OF SPARFLOXACIN FORMULATIONS

Pharmaceutical Formulation	Label claim (mg/tablet)	Amount found		% Recovery*
		Method A (mg)	Method B (mg)	
Tablet-1	200	199.75	199.38	100.95
Tablet-2	200	199.23	198.59	99.93
Tablet-3	200	201.95	199.76	98.21
Tablet-4	100	99.87	101.50	101.25

* Recovery of 20 mg added to the pharmaceutical preparations.

summarised in Table-1. When pharmaceutical preparations (Tablets) containing sparfloxacin were analysed the results obtained by the proposed methods (Table - 2) are in good agreement with labelled amounts. The recovery in both the methods was found to be 98-101%. Diluents and excipients present in the dosage forms did not interfere in the proposed methods.

The basis of method A is that methanolic solution of sparfloxacin exhibits λ_{max} at 295.2 nm. In the case of method, B, diazotised sparfloxacin is coupled with resorcinol to produce an azodye[12-14] under alkaline conditions.

These results indicate that the proposed methods are sensitive, accurate, precise and reproducible and can be used for routine determination of sparfloxacin in bulk and in dosage forms.

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