values than others, both formulated and commercial. Hence the corresponding binder - disintegrant combinations were considered suitable for nimesulide tablets. The above tablets also fulfilled all the other official requirements.

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Visible Spectrophotometric and HPLC Methods for Estimation of Suprofen from Bulk Drug Samples

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One visible spectrophotometric and one HPLC method have been developed for estimation of suprofen from bulk drug sample. Developed visible spectrophotometric method is based on formation of chloroform extractable coloured complex of drug with copper (II) acetate in presence of potassium chloride and acetate buffer pH 5.8. The coloured complex shows absorbance maxima at 682.0 nm. Beer's law was obeyed in the concentration range of 0-10 mg/ml of suprofen. Developed HPLC method was a reverse phase chromatographic method using Inertsil C_{18} column and acetonitrile:water::35:65 pH 2.7 as mobile phase with detection at 254 nm. Caffeine was used as internal standard for HPLC method. Linearity was observed in concentration range of 20-250 µg/ml of suprofen. Results of analysis for both the methods were validated statistically.

Suprofen, chemically α-methyl-4-(2-thienylcarbonyl) benzene acetic acid is an anti-inflammatory agent[1]. Few analytical methods for estimation of suprofen from biological fluids, including one GC[2], one HPTLC[3] and three HPLC[4-6] are reported. One HPLC[7] method is reported for determination of suprofen in drug substance and capsules. However no spectrophotometric method is reported for the estimation of the drug from pharmaceuticals or bulk drug sample. An attempt has been made in the present study to develop a simple visible spectrophotometric and an HPLC method for analysis of suprofen from bulk drug sample.

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A Jasco UV/visible recording spectrophotometer with 1 cm matched quartz cells and Shimadzu delivery module LC-10AD with UV SPD-10A detector and Chromatopac C-R7A integrator were used for present study.

For colorimetric method standard drug solution in chloroform (10 mg/ml) was diluted with the same so as to give several dilutions in the concentration range of 0-7 mg/ml of suprofen. To 5 ml of each dilution taken in

TABLE 1 : ANALYSIS OF SUPROFEN SAMPLE

Method	Amount Present	% Recovery*	S.D
	(mg/ml)		
Spectrophotometric Using copper(II) acetate	2.50	98.72	0.572
	5.00	99.32	0.436
	(µg/ml)		
HPLC	80	99.70	0.051
	120	99.60	0.055

*Average of three determinations

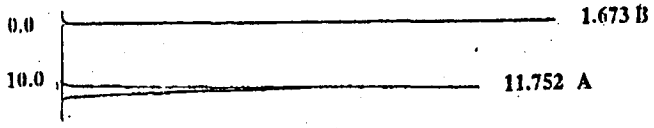

Fig. 1 : High pressure liquid chromatographic pattern of suprofen and internal standard

HPLC retention times of suprofen was found to be 11.752 min. (A) and that for internal standard caffeine was 1.673 min. (B)

a separating funnel, 3 ml copper (II) acetate solution (0.25 M in distilled water), 1 ml potassium chloride solution (2.0 M in distilled water) was 4 ml acetate buffer solution pH 5.8 were added. Reaction mixture was shaken gently for 5 min and allowed to stand so as to separate aqueous and chloroform layer. Coloured chloroform layer was separated out and absorbance was measured at 682.0 nm against reagent blank. A calibration curve was prepared.

Sample solution of bulk drug containing 2.5 mg/ml and 5 mg/ml of suprofen were prepared in chloroform and treated as per the procedure of calibration curve. Absorbance was measured at 682.0 nm and the concentration of drug in sample solution was determined from calibration curve. Results of analysis are reported in Table-1.

For HPLC method Inertsil C_{18} ODS 3V(5µ) 250 x 4.6 mm column and acetonitrile:water::35:65 pH 2.7 with ortho phosphoric acid as mobile phase were used. Instrumental conditions were: Detection at 254 nm, Flow rate 2.0 ml/min, AUFS 0.032, attenuation 8 and Chart speed 1.0 cm/min. Caffeine was used as an internal standard.

Column was saturated with mobile phase for about an hour at above specified conditions. After the chromatographic conditions were set and the instrument was stabilised to obtain a steady baseline a mixed standard dilution of pure drugs containing 50 µg/ml each of suprofen and caffeine were prepared in mobile phase, filtered through 0.2 µ membrane filter and loaded in injector of instrument fitted with 20 µl fixed volume loop. The solution was injected three times and chromatogram recorded. The mean retention times for suprofen and caffeine were found to be 11.75 and 1.672 min respectively. The representative chromatogram of suprofen and internal standard caffeine is reported in Fig. 1.

Standard stock drug solution of suprofen and caffeine with concentration of 500 µg/ml each separately were prepared in mobile phase. For preparation of drug solutions for calibration curve 0.5, 1.0, 1.5, 2.0 and 2.5 ml stock solution of standard suprofen was transferred to series of 10 ml volumetric flasks. In each flask 1.0 ml of caffeine standard stock solution was added and volume made up to the mark with mobile phase. Each solution was injected after filtration through 0.2 µ membrane filter and chromatogram recorded. The calibration curve was plotted between concentration of drug and ratio of peak area of suprofen and caffeine (internal standard). Linearity was found to be in concentration range of 20-250 µg/ml of suprofen.

Bulk drug samples of suprofen was prepared in mobile phase containing 200 µg/ml of drug. To two separate 10 ml volumetric flasks 4 ml and 6 ml of bulk drug samples was transferred, to each was added 1 ml of standard caffeine solution and made up the volume to the mark with mobile phase. The solution was then filtered through 0.2 µ membrane filter. The final dilution of bulk drug

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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