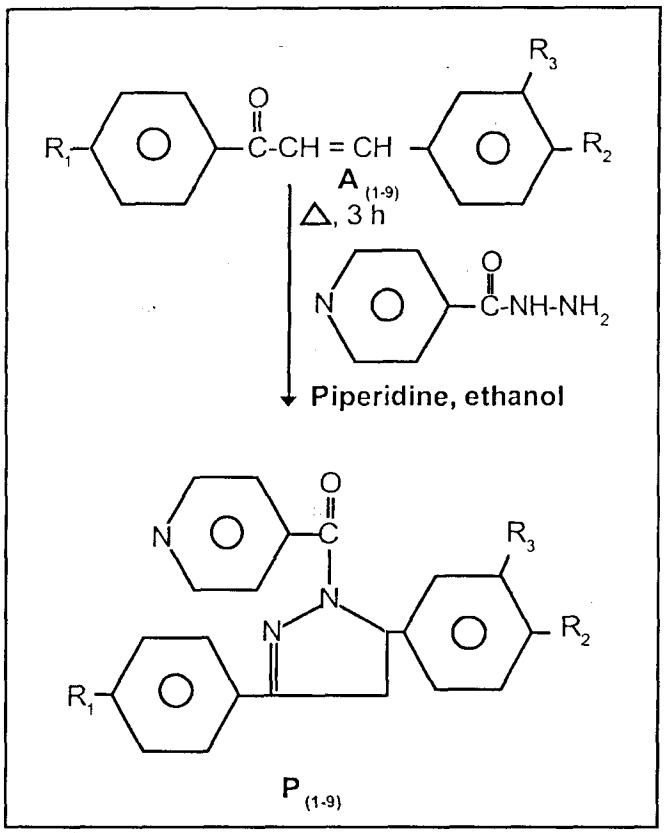

Fig. 1: Reaction scheme

pyrazoline) showed inhibitory activity only at the highest concentration of 2 µg/ml and compounds P_1 (Phenyl derivative of pyrazoline), P_4 (chlorophenyl derivative of pyrazoline), P_5 (methoxy phenyl derivative of pyrazoline), P_7 (dimethoxy phenyl derivative of pyrazoline) and P_9 (nitro phenyl derivative of pyrazoline) showed higher degree of antitubercular activity against *M. tuberculosis* at all the three concentrations used, and the above compounds showed activity comparable with that of the antitubercular activity of standard drug INH used at 0.4 µg/ml concentration and these compounds can serve as potent lead moieties in order to obtain ideal antitubercular agent.

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Estimation of Valdecoxib in Tablets by RP-HPLC Method

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A simple, efficient and reproducible reverse phase HPLC method has been developed for the determination of valdecoxib in tablets. The analyte was resolved by using a mobile phase

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(water:acetonitrile 50:50) at the flow rate of 1.2 ml/min on an isocratic HPLC system (Shimadzu) consisting of LC 10AT liquid pump, SPD 10 A UV/Vis detector, Shimadzu class LC 10 A software and Water's Spherisorb ODS column (0.4×150 mm , 0.5 μ particle size) at a wave length of 237 nm. An external standard calibration method was employed for quantitation. The linearity range was 0.2-5 μg/ml for valdecoxib and the elution time was 2.4 min. The percentage recovery obtained for two brands were 100.2±0.22 and 99.6±0.24, respectively.

Valdecoxib is a non-steroidal antiinflammatory drug that exhibits antiinflammatory, analgesic and antipyretic properties. Chemically it is 4-(5-methyl-3-phenyl-4-isoxazolyl) benzene sulphonamide[1]. It is a novel COX-2 inhibitor with a lower incidence of ulcer complication. It has been found to be an effective analgesic in post operative pain[2]. Most of the reviews on valdecoxib are associated with its clinical pharmacology, adverse effects, potential drug interaction and warnings[3,4]. The procedures[5,6] available for the quantification of valdecoxib in human plasma are very much limited and are based on sophisticated solid phase extraction-mass spectrometry (LC-MS-SPE) method and HPLC[7] method for bioequivalence of valdecoxib in plasma. However, there is no method reported for estimation of valdecoxib in formulation, the present paper aims to report an isocratic RP-HPLC method for estimation of valdecoxib in tablets.

Estimation of valdecoxib in dosage form by RP-HPLC method was carried out using optimized chromatographic conditions. Valus and Valz, the products of strength 10 mg/tab, which are manufactured and marketed by Glenmark Pharmaceuticals Ltd and Torrent Pharmaceutical Ltd were estimated. High Performance Liquid Chromatography Shimadzu pump LC-10 AT equipped with universal injector 7725i Rheodyne with injection volume of 20 μl, fixed wavelength UV detector SPD 10 A and Shimadzu class 10 software was used. Valdecoxib was obtained as a gift sample from Glenmark Pharmaceuticals Ltd, Mumbai. Acetonitrile HPLC grade and water HPLC grade were used. Stationary phase of Spherisorb ODS column 0.4x150 mm, 0.5 μ particle size was used.

Ten milligrams of pure drug valdecoxib was taken in a 100 ml standard flask and dissolved in water:acetonitrile in the ratio of 50:50 (solution A, 100 μg/ml). From solution A, 2.5 ml was taken and made up to 25 ml (solution B, 10 μg/ml). The gradient dilutions were prepared by taking 0.2-5 ml of solution B in volumetric flasks and made up to 10 ml with mobile phase. These standard solutions were injected and peak areas were measured. The calibration curve was prepared by plotting concentration of valdecoxib versus peak area of the respective solution. The method was found to follow the regression equation y=26026x-4364.2 with a correlation coefficient of 0.9999.

Twenty tablets of two different formulations were weighed and average weight of one tablet was calculated. Weight of powder equivalent to 10 mg was taken and extracted with mobile phase and then made up to 100 ml separately (100 μg/ml- solution A). From this solution 1ml was taken and made up to 10 ml which gave a solution of 10 μg/ml (solution B). From the above solution B, 0.3 ml and 3 ml of solution were taken and made up to 10 ml with mobile phase. Chromatograms were obtained by injecting 20 μl of the above prepared solution into the chromatographic system. The amount of valdecoxib present per tablet and percentage labeled claim were calculated separately (Table 1). To determine the specificity, the condition of RP-HPLC method developed through percentage of organic solvent in mobile phase, ionic strength and flow rate were changed and the occurrence

TABLE 1: RESULT OF ANALYSIS OF VALDECOXIB IN FORMULATION

Drug	Label claim (mg/tab)	Amount present ± S.D* (mg/tab)	Relative standard deviation	% Relative error	% Recovery±S.D*
Valus	10	9.96±0.11	1.1	0.42	100.2 ± 0.22
Valz	10	10.12±0.13	1.3	1.2	99.6 ± 0.24

*Average of five determinations based on label claim. Valus marketed by Glenmark Pharmaceuticals, Mumbai, Valz marketed by Torrent Pharmaceutical Ltd, Ahmedabad; Recovery of 5 mg added to the pre-analysed pharmaceutical dosage form (average of 3 determinations).

of additional peak was observed. Repeatability and reproducibility studies demonstrated the precision of the method. Repeatability studies were done by consequently injecting the standard solution at five different concentrations. These solutions were prepared in duplicate and injected as per assay procedure. The % RSD was found to be 0.42 and 1.2, respectively for two brands.

The system suitability parameters like peak asymmetry factor, capacity factor, peak area or height of their repetitive injection were carried out as specified in USP. Although USP requires only two of these criteria for method validation, *parameters like column efficiency* (10,156), *capacity factor* (1.05) and peak asymmetry factor (1.25) were calculated in the present study. Tailing factor was found to be 1.225. The LOD and LOQ of the developed method were determined by injecting progressively low concentration of the standard solution into HPLC system under optimum condition. The LOD and LOQ of the drug valdecoxib were found to be 0.5 ng/ml and 200 ng/ml respectively. In the present study the mobile phase, standard solution and sample solution were subjected to 12 h at room temperature and under refrigeration. The stability of the solution were studied by performing the experiment and looking for change in Rt, Rs and tailing of the peak compared to the pattern of the chromatogram of freshly prepared solution. The solutions stored under room temperature as well as refrigeration were stable up to 1 h. The result obtained by the method was precise and reproducible for the drug valdecoxib. The high percentage recovery and low percentage deviation were satisfactory and it shows the accuracy, reliability and suitability of the method. Hence the method developed can be used for routine analysis of valdecoxib in tablet formulations.

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Analysis of Gatifloxacin in tablet dosage form

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Two simple methods, spectrofluorimetric method and spectrophotometric method for the determination of gatifloxacin in pharmaceutical formulation are described. For spectrofluorimetric method, the excitation and emission wavelengths were found to be 365 nm and 492 nm respectively. In the case of spectrophotometric method gatifloxacin in alkaline medium on treatment with ceric

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