Visible Spectrophotometric Determination of Valdecoxib in Tablet Dosage Forms

A. SUGANTHI*, H. B. SIVAKUMAR, S. C. VIJAYAKUMAR, P. RAVIMATHI AND T. K. RAVI
Department of Pharmaceutical Analysis, College of Pharmacy, Sri Ramakrishna Institute of Paramedical Sciences, Coimbatore-641 044, India.

A simple, accurate, rapid, and sensitive visible spectrophotometric method has been developed for the determination of valdecoxib in pure and pharmaceutical dosage forms. The method is based on the reaction of valdecoxib with potassium permanganate to form a bluish green coloured chromogen with an absorption maximum at 610 nm. Beer's law was obeyed in the range of 5-25 μg/ml. The proposed method has been successfully applied to the analysis of the bulk drug and its dosage forms

Valdecoxib is a nonsteroidal antiinflammatory drug that exhibits antiinflammatory, analgesic, and antipyretic properties. Chemically, it is 4–(5-methyl–3-phenyl-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 postoperative pain. Literature survey revealed that a HPLC method has been reported for the bioequivalence of valdecoxib in plasma[2-4]. However, there is no method reported for estimation of valdecoxib in formulation. The present paper aims to report a simple visible spectrophotometric method for estimation of valdecoxib in tablets.

Valus and Valz, containing 10 mg/tab, which are manufactured and marketed by Glenmark Pharmaceuticals Ltd. and Torrent Pharmaceuticals Ltd. were estimated. Valdecoxib was obtained as a gift sample from Glenmark Pharmaceuticals Ltd., Mumbai. A Jasco V-530 UV/Vis spectrometer with 1 cm matched quartz cells was used for all absorbance measurements. All chemicals used were of analytical grade and procured from SD Fine Chemicals, Mumbai.

***For correspondence**
E-mail: suganlemu@yahoo.co.in

TABLE 1: RESULT OF ANALYSIS OF VALDECOXIB IN FORMULATION

Drug	Label claim (mg/tab)	Amount found±S.D* (mg/tab)	% recovery±S.D*
Valus	10	10.1±0.12	99.9±0.11
Valz	10	9.95±0.14	99.4±0.12

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

TABLE 2: OPTICAL CHARACTERISTICS AND PRECISION

Parameters	Results
Absorption maxima	610 nm
Beer's law limit (μg/ml)	5-25
Correlation coefficient (r)	0.9999
Molar absorptivity (l/mol.cm)	7143.72
Sandell's sensitivity (μg/cm^2/0.001/AU)	0.0440
Regression equation (Y=mx+C)	
Slope (m)	0.0203
Intercept (C)	0.0312
% Relative standard deviation	0.11
Standard error of mean	0.21

A stock solution of valdecoxib (500 μg/ml) was prepared by taking 50 mg of drug in 100 ml of 1 M sodium hydroxide (solution A). The gradient dilutions were prepared by taking 0.25-1.25 ml of solution A in a series of volumetric flasks, and to each flasks, 0.5 ml of 0.5% w/v potassium permanganate was added, mixed, and warmed at 50° for one min, cooled to room temperature, and made up to 25 ml with double-distilled water. The absorbance of the bluish green colour that developed was measured at 610 nm against reagent blank. The calibration curve was prepared by plotting concentration of valdecoxib versus absorbance of respective solution. The method was found to follow the regression equation Y = 0.0203X+0.0312 with a correlation coefficient of 0.9999.

Twenty tablets of the two different brands chosen were accurately weighed and powdered. Tablet powder equivalent to 50 mg was taken in a 100 ml volumetric flask and dissolved in 1 M sodium hydroxide solution to get a concentration of 500 μg/ml (solution B). The solution B was treated as described above, and the absorbance of the chromogen was measured at 610 nm against the reagent blank. By single point standardization method, the amount of valdecoxib present per tablet and the percentage labelled claim were calculated separately (Table 1).

The optical characteristics such as Beer's law limits, molar absorptivity, Sandell's sensitivity, correlation coefficient, % relative standard deviation and standard error of mean for the proposed method is summarised in Table 2. The developed method was validated for accuracy and reproducibility by carrying out the recovery studies after confirming non-interference from excipients like talc, starch, lactose, dextrose, and magnesium stearate in tablet formulation. For this, known amount of pure drug was added to previously analyzed samples, and these samples were reanalysed by the proposed method; the recovery was close to 100%, indicating the reproducibility and accuracy of the method. The results are shown in Table 2.

This method is based on the reduction of potassium permanganate to potassium manganate by valdecoxib in presence of 1 M sodium hydroxide, thereby producing reduced species – the bluish green colour chromogen[5,6]. The colour intensity of the chromogen was intensified with 0.5 ml of 0.5% w/v potassium permanganate warmed at 50° for 1 min. Stability of colour complex was determined by measuring absorbance of the chromogen at specified time intervals and was found to be stable for 30 min. These results indicated that the proposed method is simple, accurate, sensitive, and reproducible.

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