ISSN: 0974-6943

Research Article

Development and validation of spectrophotometric method for estimation of Etoricoxib in tablet dosage forms

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Received on:24-08-2008; Accepted on :09-12-2008

ABSTRACT

A simple, specific, precise and accurate Spectrophotometric method was developed for the determination of Etoricoxib by Spectrophotometric method in tablet dosage forms. Etoricoxib shows absorption maximum at 284 nm. The calibration curve was developed at wavelength 284 nm and validated according to ICH Guidelines. Spectrophotometric method linear response obtained was in the concentration range of 2-24 µg/ml with correlation coefficient 0.9995, recovery of the drug was found to be 99.40% and relative standard deviation was found to be less than 2 % for precision studies. The newly developed methods can be used for routine analysis of Etoricoxib in tablet dosage forms.

Key words: Etoricoxib; Spectrophotometric; Validation

INTRODUCTION

Etoricoxib is a COX-2 selective inhibitor. It is (5-chloro-2-(6-methyl pyridin-3-yl)-3-(4-methylsulfonylphenyl) pyridine). Structure of Etoricoxib is shown in Figure 1. Etoricoxib is used in the treatment of rheumatoid arthritis, osteoarthritis, chronic low back pain, gout, and ankylosing spondylitis, acute pain. Etoricoxib selectively inhibits isoform 2 of cyclo-oxigenase enzyme (COX-2).COX-2 selective inhibitor shows less marked activity on type 1 cycloxigenase compared to traditional non-steroidal anti-inflammatory drugs[1]. Review of literature[2-8] revels that no method is described for estimation of Etoricoxib by Spectrophotometric method. The present paper describes Spectrophotometric method for the determination of Etoricoxib.

EXPERIMENTAL

Preparation of stock solution of Etoricoxib:

Accurately weighed Etoricoxib (10 mg) was transferred to a 100 ml volumetric flask, dissolved in 10 ml with methanol and made-up the volume up to mark with distilled water. The final solution contained 100 µg/ml of Etoricoxib.

Determination of wavelength of maximum absorbance of Etoricoxib:

5 ml stock solution of Etoricoxib was transferred to a 50 ml volumetric flask. It was diluted up to the mark with water. The absorbance of the final solution was scanned in the range 230 to 400 nm against distilled water as blank. The spectrum is shown in figure 2.

Preparation of calibration curve for Etoricoxib:

Stock solutions of Etoricoxib (2 to 12 ml) were pipetted out in to a series of eleven volumetric flask of 50 ml. The volume in each volumetric flask was made up to the mark with distilled water and the mixture was shacked. That produced the concentration range of 2-24 µg/ml of Etoricoxib. The absorbances of the solutions were measured at 284 nm against water as blank. The linearity, slope, intercept, correlation coefficient and optical characteristics are summarized in Table1.

Recovery Studies and Validation of the Method according to ICH Guidelines [9-11]

To study the accuracy of the above proposed method, recovery studies were carried out by the addition of the standard drug solution to the placebo and recovery of drug was calculated. Result of recovery studies are summarized in Table 2. Precision of the method was studied by carrying out interday and intraday analysis and is expressed as relative standard deviation. Specificity was checked by spiking reference standard by placebo. The results were found to be satisfactory and are reported in Table 2.

Estimation of Etoricoxib in tablet dosage forms:

The twenty tablets (of same respective batch numbers) were accurately weighed and crushed to fine powder. The powder equivalent to 10 mg of Etoricoxib was transferred into 100 ml volumetric flask. 10 ml methanol was added and content in flask was sonicated to dissolve and than the volume was made

ISSN: 0974-6943

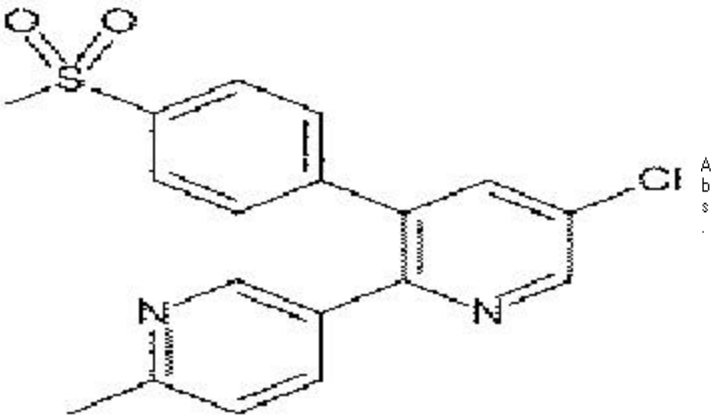

Figure1: Chemical structure of Etoricoxib

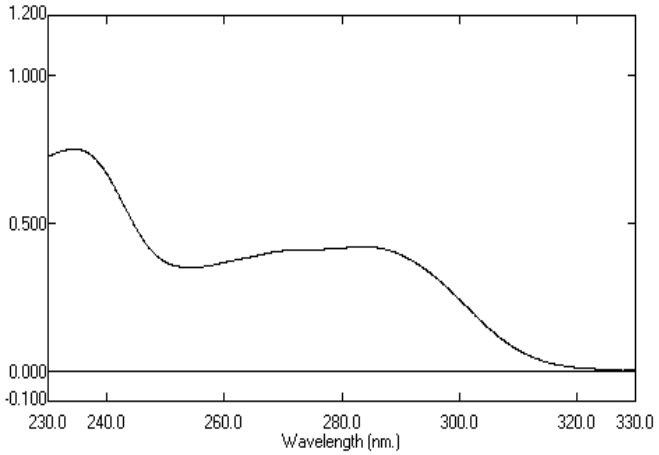

Figure2:Spectrum of Etoricoxib at wavelength 230nm to 330nm

Table 1: Optical parameters, regression and validation

Parameters	Observations
Linearity range (µg/ml)	2-24
Molar absorptivity (lit mole^{-1}cm^{-1})	1.42×10^4
Sandell's sensitivity (mg/cm^2/0.001absorbance unit)	2.5×10^{-2}
Regression equation (y= a + bc)	
slope (b)	0.0401
intercept (a)	-4.2×10^{-3}
Correlation coefficient (r)	0.9995
Specificity	No interference w.r.t. placebo
Precision :	
-Repeatability (n = 6)	0.689
-Intraday (n=3)	1.173
-Interday (3 days)	1.062
Accuracy Percent recovery	99.40±0.807

Table 2: Recovery of method from placebo solution

Method	Percent of solution in placebo	Amount* Recovered (µg/ml)	Actual amount added (µg/ml)	Percent recovery*	Mean recovery ±Standard Deviation
A	80	7.978	7.968	100.12	99.40±0.659
	100	9.844	9.960	98.84	
	120	11.860	11.952	99.23	

Table 3: results of estimation of etoricoxib (etropain tablet)

Tablet	Labeled Amount (mg/ml)		Amount found	Percent amount ± SD RSD
ETROPAIN	90	91.78	101.97 ± 0.115	0.113

up to mark with distilled water. The solution was filtered through Whatman filter paper no. 40. 10 ml of this solution was diluted to 100 ml with distilled water. The absorbance of these solutions was measured at 284 nm using water as a blank. The concentrations of Etoricoxib present in tablet dosage forms were determined and are tabulated in Table 3.

Supplementry information:

Instruments:

1) UV-Visible spectrophotometer, UV-1601 (Shimadzu)
2) Weighing balance, HR 200 (Afcoset)
3) Ultra sonic bath, SW 45 (Toshcon/ Tosniwal)

Reagents:

Methanol AR (Merck Limited),Water; distilled (In house produced)

RESULTS & DISCUSSION

The method for the estimation of Etoricoxib in tablet dosage form was developed. Drug shows absorption maximum at 284 nm. Spectrophotometric method linear response obtained was in the concentration range of 2-24 µg/ml with correlation coefficient 0.9995, recovery of the drug was found to be 99.40% and relative standard deviation was found to be less than 2 % for precision studies. The method was statistically validated according to ICH guidelines. The developed validated methods are simple, rapid, precise and accurate. The newly developed methods can be used for routine analysis of Etoricoxib in tablet dosage forms.

ACKNOWLEDGEMENT:

Authors are sincerely thankful to Department of Pharmaceutical Science S.G.R.R.I.T.S Dehradun for providing necessary facilities and Zydus Cadila, Changodar, Ahmedabad Plant, for providing pure etoricoxib drug sample.

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Source of support: Nil, Conflict of interest: None Declared