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Research Article

DEVELOPMENT AND VALIDATION OF ANALYTICAL TECHNIQUE FOR NON STEROIDAL ANTI INFLAMMATORY DRUGS (NSAIDs) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

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ABSTRACT

Drug analysis plays an important role in the development of drugs, their manufacture and therapeutic use. Pharmaceutical industries rely upon quantitative chemical analysis to ensure that the raw material used and final products obtained meet the required specifications. The number of drugs and drug formulations introduced into the market has been increasing at an alarming rate. These drugs or formulations may be either new entities or partial structural modifications of the existing ones or novel dosage forms (controlled/ sustained release formulations) or multi component dosage forms. Very often, there is a time lag from the date of introduction of a drug into the market to the date of its inclusion in pharmacopoeias. This happens because of the possible uncertainties in the continuous and wider usage of these drugs, reports of new toxicities (resulting in their withdrawal from the market), development of patient resistance and introduction of better drugs by competitors. Under these conditions, standards and analytical procedures for these drugs may not available in pharmacopoeias. It becomes necessary, therefore, to develop newer analytical methods for such drugs. Considering all these views some drug formulations from Non Steroidal Anti Inflammatory Drugs (NSAIDs) were selected for the present study.

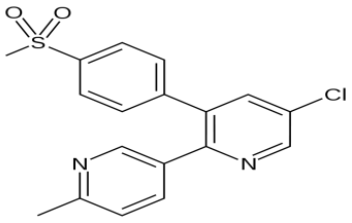
KEYWORDS: validation of Analytical Technique, NSAIDs

INTRODUCTION

ETORICOXIB

AIMS & OBJECTIVES

To Develop Analytical methods and Validation of Etoricoxib by High Performance Liquid Chromatography (HPLC).



It is 5-chloro-2-(6-methylpyridin-3-yl)-3-(4-methylsulfonylphenyl) pyridine

Molecular Formula: $C_{18}H_{15}ClN_2O_2S$ Molecular Weight: 358.84

DRUG PROFILE

ETORICOXIB

Etoricoxib is a nonsteroidal anti-inflammatory drug (NSAID) that exhibits anti-inflammatory, analgesic and antipyretic activities [1-4]. Etoricoxib is a potent, orally active, highly selective cyclooxygenase-2 (COX-2) inhibitor. This drug is used for treatment in rheumatoid arthritis, osteoarthritis and pain [5-8].

Different Company's manufactures Etoricoxib in India and their trade names:

Sl.No.	Brand name	Company name	Type of dosage form	Quantity available
1	Eldoflam	Elder	Tablet	60, 90 & 120mg
2	Etocox	Aristo	Tablet.	60, 90 & 120mg
3	Etoshine	Sun Pharma	Tablet.	60, 90 & 120mg
4	Etrobox	Ranbaxy	Tablet.	60, 90 & 120mg
5	Nucoxia	Zydus cadila	Tablet.	60 & 90 mg

EXPERIMENTAL

ESTIMATION OF ETORICOXIB BY REVERSE PHASE HPLC METHOD

MATERIALS AND METHOD

Instrument: HPLC (Waters)

Chemicals & Reagents:

Etoricoxib working standard was a gift sample from M/S Sun Pharmaceutical Industries Ltd., Vapi, India and formulations were obtained from drug stores in market.

Acetonitrile HPLC grade from Merck. Potassium dihydrogen phosphate (AR grade), HPLC grade water from Rankem.

Preparation of mobile phase

700.0 ml of Acetonitrile of HPLC grade and 300.0ml of 50mM potassium dihydrogen phosphate (6.804 grams dissolved in 1 liter water) were taken and both were mixed and sonicated for 15 minutes and they are filtered through 4.5micron filter paper and further sonicated for 5 minutes.

Standard preparation

10mg of standard drug was taken in 10ml standard volumetric flask and dissolved in the mobile phase using sonicator. And the stock solution was further diluted to micro gram level concentration with the mobile phase.

Linearity of detector response

Linearity study was carried out at five different concentrations and it was found to be linear in range of 4 to 20 micro gram concentrations (4mcg/ml, 8mcg/ml, 12mcg/ml, 16mcg/ml, 20mcg/ml) were prepared for Etoricoxib and the **peak areas** Vs **concentrations** are plotted in the graph as shown below.

Chromatography

The flow rate was maintained at 1ml/min. Temperature of the column (Thermo hypurity C18, 100*4.6mm,5µ) was ambient, the average pressure was 1585 psi and the effluents were monitored at 235nm. The mobile phase used was Acetonitrile and 50mM potassium dihydrogen phosphate (70:30).

EXPERIMENT

Assay of twenty tablets of different manufacturer`s were procured from market, weighed and triturated finely, the powder equivalent to 10 mg of the pure drug was dissolved in mobile phase to get 20mcg/ml concentrations. Twenty microlitres of sample preparation was injected into injector of liquid chromatograph. From the peak response of Etoricoxib the amount of drug in sample was computed.

Formula:

$$\%\text{Of Sample} = \frac{\text{Mean Area of Sample}}{\text{Mean Area of Standard}} \times \frac{\text{Weight of Standard}}{\text{Dilution Factor}} \times \frac{\text{Dilution Factor of Sample}}{\text{Weight of Sample}} \times \text{Label Claimed}$$

Recovery experiment

To study the accuracy, reproducibility and the precision of the proposed method recovery experiments were carried out. A fixed amount of pre analyzed sample was taken and standard drug was added at three different concentrations and each level repeated for 4 times.

RESULTS AND DISCUSSION

Linearity Response of Detector (Figure 1)

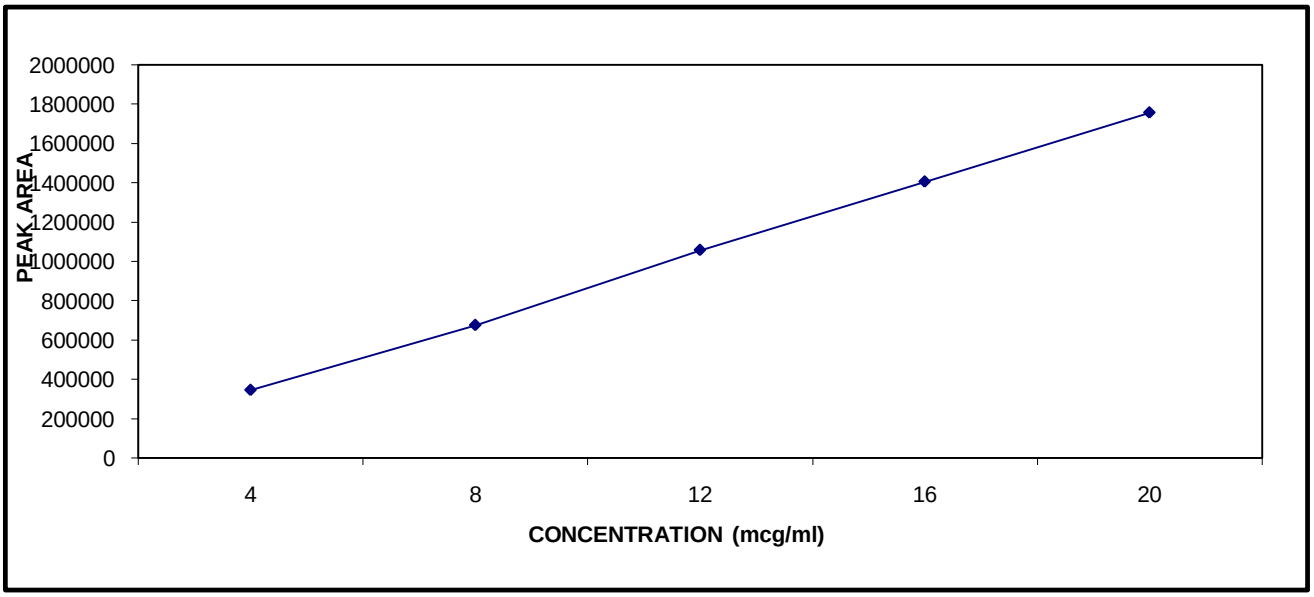


Table 1 Reproducibility experiment

Sl. No	Name of company	Amount found Mg/ tablet ± SD	%RSD	Percentage of Assay
1	ETO 1	90.012+0.358	0.35	100.15
2	ETO 2	90.285+0.235	0.46	100.35

Table 2 Recovery experiment ETO 1

Label claim amount of std added in mg	Amount of standard drug added in mg	Amount recovered in mg	% of recovery
90	0.0	89.55	99.50
90	5	95.54	100.56
90	10	101.47	101.47
90	15	104.55	99.57

Table: 3. Recovery experiment ETO 2

Label claim amount of std added in mg	Amount of standard drug added in mg	Amount recovered in mg	% of recovery
90	0.0	90.58	100.64
90	5	95.16	100.16
90	10	98.99	98.99
90	15	105.03	100.02

CONCLUSION

The accuracy of the method was noted and it was felt that method can be suitably adapted for other drugs and combination in further studies. HPLC method has been accurate and it gives details with regards best separation and calculation of concentration simultaneously.

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