SHORT COMMUNICATION

# A simple Ultraviolet spectrophotometric method for the determination of etoricoxib in dosage formulations

Shipra Singh, Amrita Mishra, Anurag Verma, Ashoke K. Ghosh, Arun K. Mishra

Department of Pharmaceutical Chemistry, Central Facility of Instrumentation, College of Pharmacy, IFTM Lodipur-Rajput, Moradabad, India

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## ABSTRACT

The present study was undertaken to develop a validated, rapid, simple, and low-cost ultraviolet (UV) spectrophotometric method for estimating Etoricoxib (ETX) in pharmaceutical formulations. The analysis was performed on λ max 233 nm using 0.1 M HCl as blank/diluent. The proposed method was validated on International Conference on Harmonization (ICH) guidelines including parameters as linearity, accuracy, precision, reproducibility, and specificity. The proposed method was also used to access the content of the ETX in two commercial brands of Indian market. Beer's law was obeyed in concentration range of 0.1–0.5 μg/ml, and the regression equation was Y = 0.418x + 0.018. The mean accuracy values for 0.1 μg/ml and 0.2 μg/ml concentration of ETX were found to be 99.76 ± 0.52% and 99.12 ± 0.84, respectively, and relative standard deviation (RSD) of interday and intraday was less than 2%. The developed method was suitable and specific to the analysis of ETX even in the presence of common excipients. The method was applied on two different marketed brands and ETX contents were 98.5 ± 0.56 and 99.33 ± 0.44, respectively, of labeled claim. The proposed method was validated as per ICH guidelines and statistically good results were obtained. This method can be employed for routine analysis of ETX in bulk and commercial formulations.

**Key words:** Etoricoxib, quality control, UV spectrometry, validation

## INTRODUCTION

Chemically Etoricoxib (ETX) is 5-chloro-3-(4-methanesulfonylphenyl)-2-(6-methylpyridin-3-yl) pyridine [Figure 1].[1] It is a selective COX-2 inhibitor, which belongs to a family of pain killers called non-steroidal anti-inflammatory drugs (NSAIDs). It is mainly used to treat patients suffering from joint pain and swelling caused by osteoarthritis, rheumatoid arthritis, ankylosing spondylitis, and gout. It is also used to reduce swelling and joint stiffness.[2]

**Address for correspondence:**
Dr. Arun K. Mishra,
Central Facility of Instrumentation, College of Pharmacy,
IFTM, Lodipur-Rajpur, Moradabad, India.
E-mail: arun_azam@rediffmail.com

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Analysis is an essential step of formulation development, and it must include a simple, reliable, and cost-effective method. Keeping in view this objective, the present work was undertaken to develop and validate simple UV spectrophotometric method for estimation of ETX in dosage formulations.

Earlier methods including high performance liquid

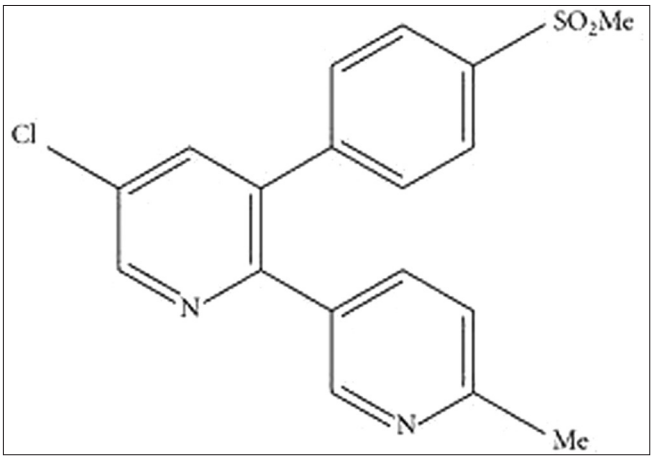


**Figure 1:** Chemical structure of Etoricoxib

chromatography (HPLC),[3,4] high performance thin layer chromatography (HPTLC),[5,6] liquid chromatography–mass spectrometry (LC–MS),[7] capillary zone electrophoresis,[8] and ultra performance liquid chromatography (UPLC)[9] for quantification of ETX in pharmaceutical dosage forms are reported. These reported methods involve a tedious sample preparation process, costly equipments, as well as time-consuming steps. The long run time of these processes limits their applicability for large number of samples.

Upon literature survey and as per our present knowledge, there is no simple and reliable method for estimation of ETX from pharmaceutical dosage forms as well as in bulk formulation. In this study, a simple UV spectrophotometric method was developed and validated as per International Conference on Harmonization (ICH) guidelines.[10] This method involves simple instrument and cost-effective solvents, and consumes less time. The reproducibility of the proposed method was high, which accounts this method novel and reliable. The method was also used in the determination of the content of ETX in two marketed ETX products in India.

## MATERIALS AND METHODS

### Instruments and Materials

Schimazdu 1800 double beam UV/Vis spectrophotometer, digital balance (Citizen Co. Mumbai, India), and micropipette (The Modern scientific industries, Meerut, India) were used in this study. ETX was obtained as a gift sample from the Torrent Research Centre, Hyderabad, India. The other chemicals and reagents used were of analytical grade.

### Standard Stock Solution

Standard drug solution of ETX was prepared by dissolving 10 mg of ETX in 5 ml 0.1 N HCl in a 10-ml volumetric flask, shaken well, and finally the volume was adjusted to get a solution of concentration of 1 mg/ml. This 1 mg/ml solution was used as a stock solution.

### Calibration Curve

Five milliliters of 1 mg/ml aliquot solution was further diluted up to 50 ml by 0.1 N HCl in a 100-ml volumetric flask and the final volume was adjusted up to 100 ml. This was scanned spectrophotometrically in the wavelength region 190–800 nm to determine the wavelength of maximum ($\lambda$max) absorption. The $\lambda$max was found to be 233 nm against blank [Figure 2]. From 1 mg/ml stock solution, the serial dilution pattern was followed to obtain aliquots of 0.1–0.5 μg/ml concentration. The calibration curve was plotted between concentration and absorbance. The optical characteristics of different aliquots are presented in Table 1.

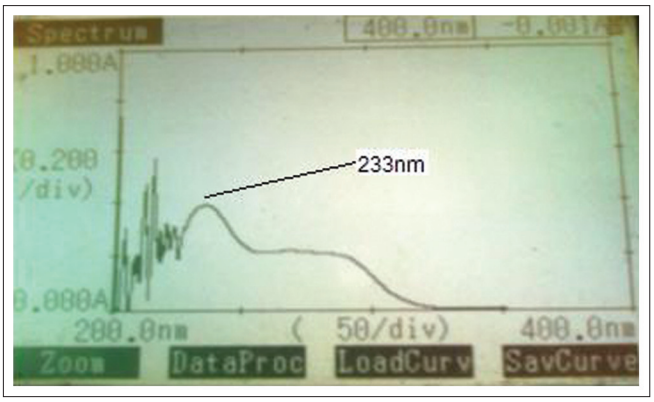


**Figure 2:** Scanned spectra of Etoricoxib

**Table 1: Calibration curve data of etoricoxib in 0.1 N HCl**

| Concentration (μg/ml) | Mean absorbance |
|---|---|
| 0.1 | 0.061 |
| 0.2 | 0.099 |
| 0.3 | 0.142 |
| 0.4 | 0.191 |
| 0.5 | 0.224 |

### Sample Solution Preparations

The proposed method was applied on two different commercial brands of ETX. Twenty tablets of each brand were weighed and powdered. Ten milligrams of ETX powder equivalents was weighed and transferred in a 10-ml volumetric flask by diluting it with 10 ml 0.1 N HCl. After a continuous shaking for 10 min, the solutions were filtered through Whatman filter paper separately. The filtrate was further suitably diluted in order to get 0.2 μg/ml concentration. Against a blank solution, the absorbance was measured at 233 nm wavelength. Finally, employing the calibration curve and linear equation, the concentration was calculated. Determination of accuracy was done by % addition method and is presented in Table 2. Amount of drug estimated through this method is presented in Table 2.

## RESULTS AND DISCUSSION

### Linearity

The linearity of the drug was obtained for 0.1–0.5 μg/ml concentration range of ETX. The calibration curve was obtained by plotting absorbance versus concentration and linear regression analysis was performed to get linear equation.[11] The linear equation found was $y = 0.418x + 0.018$ and $r^2$ was 0.997. The calibration curve was found to be linear in stated concentration.

### Accuracy

Accuracy of the method was estimated by standard addition recovery method. In this, known amount of standard ETX was added to pre-analyzed sample.[12] This was done for

**Table 2: Determination of accuracy (by percentage recovery method) and precision**

| Ingredient | Tablet amount (μg/ml) | % Addition | Amount added (μg/ml) | Amount recovered (μg/ml) | % Recovery | Precision %RSD* | |
|---|---|---|---|---|---|---|---|
| | | | | | | Interday | Intraday |
| Etoricoxib | 0.1 | 100 | 0.1 | 0.099 | 99.76 ± 0.52 | 1.01 | 0.98 |
| | 0.2 | 100 | 0.2 | 0.198 | 99.12 ± 0.84 | 0.91 | 1.02 |

*Average of three replicates

0.1 μg/ml, 0.2 μg/ml, and performed in triplicate. The accuracy values for 0.1 μg/ml and 0.2 μg/ml concentration of ETX were found to be 99.76 ± 0.52 and 99.12 ± 0.84%, respectively [Table 2].

### Precision

The precision of the assay was determined by repeatability (intraday) and intermediate precision (interday) and reported as % relative standard deviation (RSD).[13] For this, 0.1 μg/ml and 0.2 μg/ml concentration solution was measured three times in day and the same was measured in the next 3 days. The %RSD was calculated [Table 2].

### Robustness

Robustness of the method was determined by carrying out the analysis under different temperature conditions, i.e. at room temperature and at 18°C.[14] The respective absorbance of 0.3 μg/ml was noted and the result was indicated as %RSD [Table 3].

### Ruggedness

The ruggedness of the method was determined by carrying out the analysis by different analysts and the respective absorbance of 0.3 μg/ml was noted. The result was indicated as %RSD [Table 4].

### Limit of Detection and Limit of Quantification

The limit of detection (LOD) and limit of quantification (LOQ) for ETX were determined by using standard deviation of response and slope.[15] The LOD and LOQ values are presented in Table 5.

### Stability

The stability of ETX in 0.1 N HCl solution was studied by the developed method. Sample solutions (0.3 μg/ml) were prepared in triplicate and heated to maintain 50°C and 60°C for 60 min.[16] The absorbance data of these samples reveled information about the stability of ETX [Table 6].

### Determination of Active Ingredients in Different Brands of Tablets

The proposed and validated method was applied to estimate the amount of active ingredient, ETX, in two different brands of tablets using 20 tablets in each batch. Results of quantitative analysis are presented in Table 7. The findings of analysis suggest that both the marketed formulations fulfilled the % amount requirement (98–102%) with respect to labeled claim.

**Table 3: Robustness analysis**

| Concentration (μg/ml) | Absorbance at room temperature | Absorbance at 18°C |
|---|---|---|
| 0.3 | 0.142 | 0.142 |
| 0.3 | 0.141 | 0.143 |
| 0.3 | 0.141 | 0.141 |
| 0.3 | 0.143 | 0.141 |
| 0.3 | 0.141 | 0.143 |
| 0.3 | 0.141 | 0.143 |
| | %RSD = 0.58 | %RSD = 0.63 |

**Table 4: Ruggedness analysis**

| Concentration (μg/ml) | Absorbance by analyst 1 | Absorbance by analyst 2 |
|---|---|---|
| 0.3 | 0.143 | 0.141 |
| 0.3 | 0.142 | 0.142 |
| 0.3 | 0.142 | 0.142 |
| 0.3 | 0.144 | 0.141 |
| 0.3 | 0.142 | 0.143 |
| 0.3 | 0.144 | 0.142 |
| | %RSD = 0.68 | %RSD = 0.52 |

**Table 5: Validation parameters**

| Parameters | Result |
|---|---|
| Absorption maxima (λmax) | 233 nm |
| Linearity (μg/ml) | 0.1–0.5 |
| Linear equation | y = 0.418x + 0.018 |
| Correlation coefficient ($r^2$) | 0.997 |
| Limit of detection (LOD) (μg/ml) | 0.029 |
| Limit of quantification (LOQ) (μg/ml) | 0.095 |

**Table 6: Stability study**

| Drug concentration (μg/ml) | Absorbance at 50°C | Absorbance at 60°C | Remark |
|---|---|---|---|
| 0.3 | 0.142 | 0.134 | Etoricoxib is stable at 50°C temperature, but increase of temperature to 60°C causes degradation |
| 0.3 | 0.143 | 0.110 | |
| 0.3 | 0.140 | 0.102 | |

## CONCLUSION

The developed method was found to be simple, rapid, cost-effective, and reproducible, with high accuracy and precision

**Table 7: Determination of active ingredients (%)**

| Sample | Labeled claim/tablet | Amount found per tablet | % Active ingredient of labeled claim |
|---|---|---|---|
| Brand 1 | 90 | 89.3 | 99.33 ± 0.44 |
| Brand 2 | 90 | 88.6 | 98.5 ± 0.56 |

value. The parameters were validated as per ICH guidelines. The satisfactory findings of the work suggest that the method may be applied for quantitative estimation of ETX from bulk and pharmaceutical dosage formulations. This method may also be used in routine quality-control aspects.

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