FTIR Spectroscopic Method for Quantitative Analysis of Etoricoxib in Tablets


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ABSTRACT

A rapid FTIR spectroscopic method has been proposed for the estimation of Etoricoxib in bulk drug and pharmaceutical dosage form. The method involves the measurement of the area of the infrared band corresponding to the S=O stretching centered at 1049 cm\(^{-1}\). The excipients in the commercial tablet preparation did not interfere with the assay. The linearity range was found to be 100-350 µg/ml. The technique is reliable and useful for quality control for monitoring the adulteration of pure drug. The proposed method is statistically validated and found to be useful for the routine determination of etoricoxib in tablets.

Keywords: Etoricoxib, FTIR, Tablets, Validation.

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INTRODUCTION

Etoricoxib (ETX) is a specific type of an anti-inflammatory drug most commonly used for the relief of pain and swelling suffered by individuals [1,2]. Chemically it is 5-chloro-3-(4-methanesulfonyl phenyl)-2-(6-methyl pyridine-3-yl) pyridine [3]. Literature review revealed very few analytical methods including HPLC[4], HPTLC[5], LC-MS[6], Capillary Zone Electrophoresis[7] and Ultra Performance Liquid Chromatography[8] for quantification of ETX in pharmaceutical dosage forms. In the present work, a rapid FTIR spectroscopic method [9] has been developed for the estimation of ETX in bulk drug and pharmaceutical dosage form. To develop the quantitative analysis method, a standard solution of known concentration is prepared and spectra are collected from aliquots of the standard. Specified absorption band is identified and the peak area is calculated.

MATERIALS AND METHODS

Experimental

Instrument

All spectral and absorbance measurements were made on FTIR-Model ABB MB 3000.

Standard solution of ETX

A 1mg/ml stock solution of ETX was prepared by dissolving 100mg of drug in 100ml of ethanol.

Sample preparation

Twenty tablets were weighed. A quantity equivalent to 100mg of ETX was weighed accurately, transferred to a beaker, dissolved in ethanol, filtered through whatmann filter paper No.1 into a 100ml volumetric flask and made up to volume with ethanol to get a concentration of 1mg /ml.

Method

The stock solution was diluted suitably with ethanol to give a series of concentration ranging from 100-350 μg/ml of ETX. The IR spectrum was recorded for the various concentrations. The absorbance of the band due to S=O stretching at 1049 cm\(^{-1}\) was measured. All the determinations were conducted in triplicate. The IR spectra for ETX is shown in fig-1. The calibration curve of ETX was obtained by plotting the peak area (S=O stretching centered at 1050 cm\(^{-1}\)) versus concentration.
Sample analysis

Pharmaceutical formulation of ETX was successfully analysed by the proposed method. Appropriate aliquots were subjected to the above method and the amount of ETX was determined.

RESULTS AND DISCUSSION

The proposed method is statistically validated and found to be useful for the routine determination of etoricoxib in tablets. The regression characteristics like slope \((m)\), intercept\((c)\), correlation coefficient\((r)\), percent relative standard deviation \((\% \text{ RSD})\) and standard error \((\text{SE})\) were calculated and the results are summarized in Table-1. All validation parameters were found to be highly satisfactory. The results of sample analysis are furnished in Table-2. The result of sample analysis showed that the drug determined by the proposed method was in good agreement with the label claim proving the accuracy of the proposed method.

To study the accuracy and reproducibility of the proposed method, recovery experiments were carried out by adding a known amount of drug to pre-analysed sample and the percentage recovery calculated. The results are furnished in Table-2. The results indicate that there is no interference of other ingredient presents in the formulations. Thus the proposed method is rapid, sensitive, accurate and reproducible and useful for the routine determination of ETX in bulk drug and its pharmaceutical dosage forms.

Fig 1: IR spectrum of ETX
Table 1: Statistical parameters

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linearity range (µg/ml)</td>
<td>100 -350</td>
</tr>
<tr>
<td>Correlation coefficient(r)</td>
<td>0.9959</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>0.0802</td>
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<tr>
<td>Standard error</td>
<td>0.0327</td>
</tr>
<tr>
<td>%RSD</td>
<td>0.001336</td>
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<tr>
<td>Regression equation</td>
<td>y=mx+c</td>
</tr>
<tr>
<td>Intercept (c)</td>
<td>-0.1793</td>
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<tr>
<td>Slope(m)</td>
<td>0.3624</td>
</tr>
<tr>
<td>LOD (µg/ml)</td>
<td>50</td>
</tr>
<tr>
<td>LOQ(µg /ml)</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 2: Assay and recovery of ETX in dosage form

<table>
<thead>
<tr>
<th>Drug</th>
<th>Labelled amount (mg)</th>
<th>Amount obtained (mg)*</th>
<th>Percentage recovery*</th>
</tr>
</thead>
<tbody>
<tr>
<td>ETX</td>
<td>60</td>
<td>59.99</td>
<td>100.03</td>
</tr>
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</table>

*Average of 6 determinations

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**REFERENCES**