IJPPR
INTERNATIONAL JOURNAL OF PHARMACY & PHARMACEUTICAL RESEARCH
An official Publication of Human Journals
ISSN 2349-7203
HUMAN

Human Journals
Research Article
June 2023 Vol.:27, Issue:3

UV Spectrophotometric Method Development and Validation for Estimation of Celecoxib

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ISSN 2349-7203
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Submitted: 27 May 2023
Accepted: 03 June 2023
Published: 30 June 2023

Keywords: Validation, celecoxib, UV Spectrophotometer.

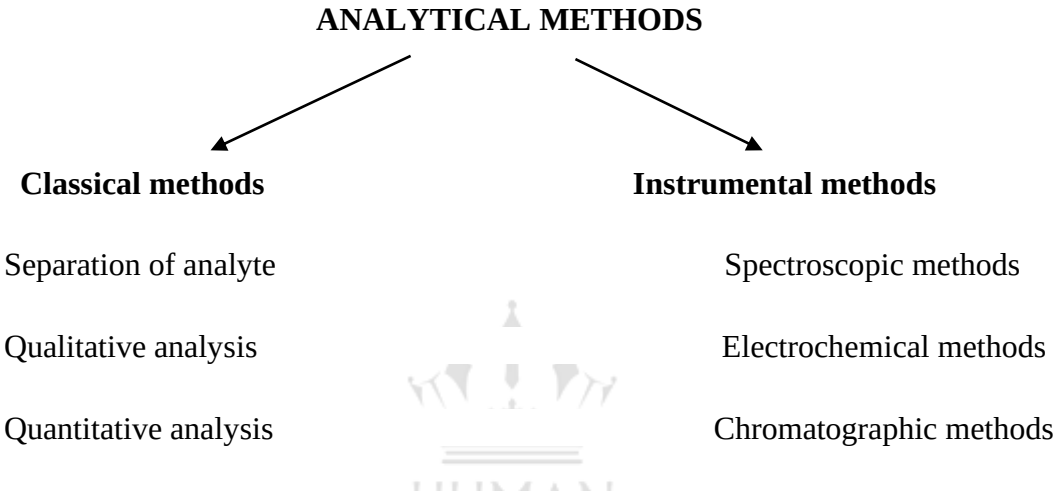
ABSTRACT

A Simple, specific, rapid, precise and accurate UV Spectrophotometric method have been developed and validated for the determination of Celecoxib drug. Celecoxib showed the absorption maxima at 254 nm and was linear in the range of 0.2 µg/ml-1 µg/ml. The validation of the above-proposed method was done by carrying out precision and accuracy studies. The analytical method showed good Intra-day precision (Repeatability) with relative standard deviation 0.410% and Inter-day precision with relative standard deviation is 0.328% which is less than 2. The proposed method was validated for the parameter Specificity, Precision, Linearity and range, Robustness, Accuracy and recovery. Hence proposed analytical method can be applied for routine quality control analysis of celecoxib drug by using a UV spectrophotometer.

INTRODUCTION:

Analytical method:[1]

Analytical method includes the use of a specified technique and detailed-stepwise instructions which are used in qualitative, quantitative or structural analysis of a sample for one or more analytes. Analytical methods are mainly classified into two types: Classical methods and Instrumental methods in which the signal is proportional to the absolute amount of analyte is called classical method. A method in which the signal is proportional to the analyte concentration is called the instrumental method.



Celecoxib is chemically designated as 4-[5-(4-methylphenyl)- 3-(trifluoromethyl)-1H-pyrazol-1-yl] benzenesulfonamide and is a diaryl-substituted pyrazole. The mechanism of action of celecoxib is due to the selective inhibition of cyclooxygenase-2 (COX-2), which is responsible for prostaglandin synthesis, an integral part of the pain and inflammation pathway. This pharmacologic activity gives celecoxib its analgesic, anti-inflammatory, and antipyretic effects. Celecoxib weakly inhibits COX-1 and, therefore, may affect platelet function less than aspirin. Celecoxib having the molecular formula C17H14F3N3O2S. The molecular weight of celecoxib is 381.373g/mol. In the present work, a simple, accurate and sensitive method for determining Celecoxib content in drug substance pure form was introduced. No simple and rapid work has been reported for the estimation of Celecoxib formulation drug. Hence it was felt necessary to build up a simple, rapid, economical and precise Spectrophotometric method for the direct estimation of Celecoxib formulation drug. The current research work deals with the development of UV Spectrophotometric method and its validation as per the International Conference on Harmonization (ICH) guideline. The developed method was found to be simple, specific, stable, rapid, accurate, precise, reliable,

less expensive and time-saving by UV Spectrophotometric method for the estimation of Celecoxib content in drug substance.[2]

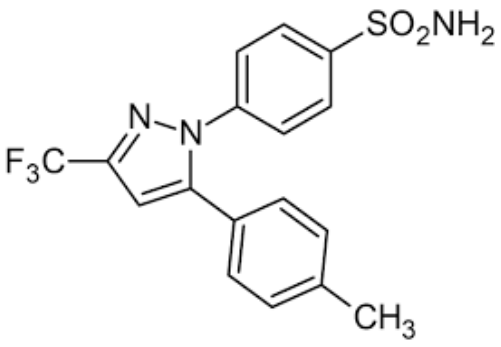


Fig 1: Chemical structure of Celecoxib

MATERIALS AND METHODS:

Instrumentation and Materials:

U.V. visible double beam spectrophotometer SL 210 Elico with Spectra treat software having path length 1cm U.V. matched quartz cells were used. Celecoxib Sample and Standard gifted from Aarati Labs Mumbai. All chemicals, solvents and reagents (Ethanol, Water and Methanol) used were of analytical grade.

Method Development

- **Preparation of Standard Solution:**

Weighed accurately about 10 mg of celecoxib and transferred it to 10 ml volumetric flask. Dissolved in 10 ml ethanol, then take 0.1ml of sample from that in 10ml volumetric flask and make up volume 10ml with ethanol. And concentration becomes 10µg/ml.

- **Selection of wavelength for analysis of Celecoxib:**

The standard solution having a concentration of 10µg/ml was scanned at 200 nm to 400nm with the blank to detect maximum wavelength.

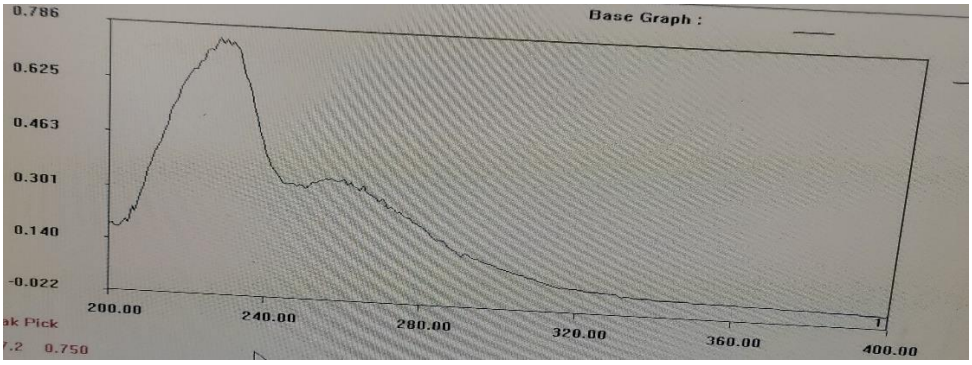


Fig 2: Estimation of Maxima of Celecoxib.

From the above (Figure-2) spectra of Celecoxib wavelength maxima identified for quantification was 254.0 nm (λmax).

- **Validation of proposed Analytical Method:**

The proposed method was validated according to International Conference on Harmonization (ICH) guidelines for the validation of analytical procedures. Analysis of variance was used to ensure the validity and performance effectiveness of the proposed analytical methods.

- **Specificity:**

Specificity is the ability to assess unequivocally the analyte in the presence of components that may be expected to be present. Typically, these might include impurities, degradants, matrix, etc. Specificity was done by scanning of blank solution and Standard solution of celecoxib having concentrations 10 µg/ml in Spectrophotometric range from 200 nm to 400 nm to check specific absorption maxima at predefined wavelength i.e. 254.0 nm and solution stability study performed to evaluate the solution stability at different time interval up to 26 hrs.

- **Instrument Precision**:

Instrument precision was performed to check the suitability of the developed analytical method with respect to the ability of instrument consistency to provide the precise wavelength maxima when scanned the Standard solution of Celecoxib having concentrations 10 µg/ml in the UV range from 200 nm to 400 nm. To check specific absorption maxima at a predefined wavelength 254.0 nm with reproducible absorption detection. Six separate standard preparations were scanned / analysed according to the proposed method of analysis.

The % RSD due to celecoxib concentration for the six standards was found 0.350%. Results of precision are depicted in table 1.

Table 1: Instrument precision

concentration	absorbance
0.6	0.175
0.6	0.171
0.6	0.173
0.6	0.174
0.6	0.171
0.6	0.176
mean	0.17333
SD	0.00207
%RSD	1.19169

- **Linearity and Range**

The linearity of an assay method is its ability to elicit test results, which are directly proportional to the concentrations of drugs in samples in a given range. Linearity justifies the use of single-point calibrations. The correlation coefficient of the Regression line for was found that 0.9993. Five levels of five different concentrations Standard solution of Celecoxib having concentrations 0.2 µg/ml, 0.4 µg/ml, 0.6 µg/ml, 0.8 µg/ml and 1 µg/ml, in the range relative to the working concentrations, were prepared and read according to the method of analysis. A linear regression curve was constructed, the correlation coefficient (R2) and assessment value calculated. The correlation coefficient (R2) for celecoxib obtained is 0.9993. The plot is a straight line and the results are tabulated in Table 2 and Curve is shown in Figure 2.

Table 2 Linearity and Range.

Concentration (µg/ml)	absorbance
0.2	0.129
0.4	0.228
0.6	0.331
0.8	0.421
1	0.531

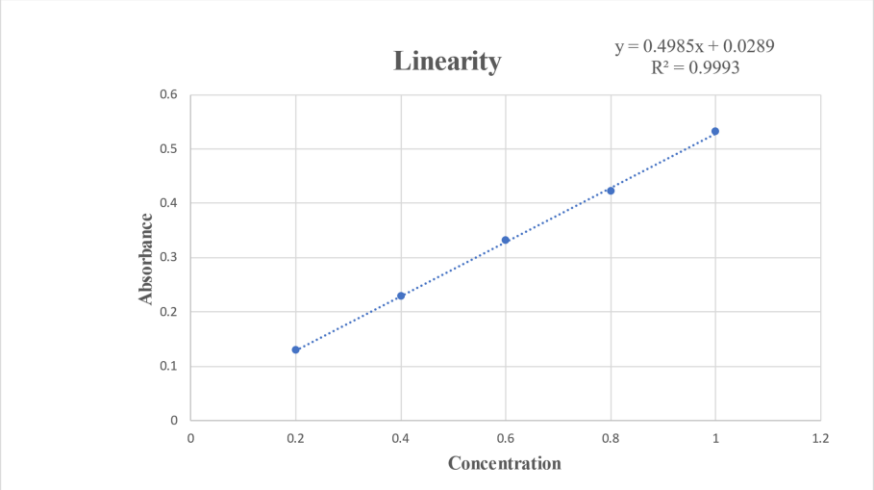


Fig 3: Calibration curve of Celecoxib in ethanol

Analytical Method Precision

The precision of an analytical procedure expresses the degree of agreement among individual test results when the method is applied to multiple sampling of a homogenous sample.

Procedure for analysis of Sample:

Weighed accurately about 10 mg of celecoxib and transferred it to 10 ml volumetric flask. Dissolved in 10 ml ethanol, then take 0.1ml of sample from that in 10ml volumetric flask and makeup volume 10ml with ethanol. And concentration becomes 10µg/ml.

Intra-day Precision (Repeatability):

This parameter determines the repeatability of celecoxib assay results under the same operating conditions over a short period of time. The % RSD due to celecoxib concentration for the six samples was found to be 0.4104 Six separated sample preparations were analysed according to the proposed method of analysis. The % RSD due to celecoxib concentration for the assay meets the requirements. Results are tabulated in Table 3.

Table 3 Intra-day Precision (Repeatability) Results.

concentration(µg/ml)	Absorbance
0.6	0.421
0.6	0.421
0.6	0.422
0.6	0.421
0.6	0.425
0.6	0.421
mean	0.422
SD	0.001732051
%RSD	0.41043858

Inter-day Precision (Repeatability):

This parameter determines the Intermediate repeatability of celecoxib assay results under the same operating conditions test performed on a different day, using different makes of reagents and solvents. The %RSD due to celecoxib concentration for the six samples was found to be 0.328% Six separated sample preparations were analysed according to the proposed method of analysis. The % RSD due to celecoxib concentration for the assay meets the requirements. Results are tabulated in Table 4.

Table 4: Inter-day Precision (Repeatability) Results.

Concentration(µg/ml)	Absorbance
0.6	0.487
0.6	0.485
0.6	0.489
0.6	0.488
0.6	0.489
0.6	0.489
mean	0.487833
SD	0.001602
%RSD	0.328408

ACCURACY:

This parameter determines the accuracy of the assay results under the same operating conditions test.

A Celecoxib sample was constituted analysed for the accuracy with known quantity of samples of Celecoxib at 80%, 100%, 120% concentration levels and assayed as per the method stated under analytical Methods respectively. Three determinations were performed under each concentration levels respectively. Results are shown in Table 5. The % RSD due to recovery of Celecoxib at 80%, 100%, 120% concentration levels were found to be 98.7%, 99.2% and 97.6% respectively. Nine sample preparations were analysed according to the proposed method of analysis.

Table 5: Accuracy and Recovery Results in 80%, 100 %, 120% Concentration level.

Sr.No	%level	Amt spiked	Amt recovery	%recovery
1	80	8	7.9	98.7
2	100	10	9.92	99.2
3	120	12	11.72	97.6

Robustness:

Robustness, the UV method was obtained utilizing different percentages of ethanol in a co-solvent system. Ethanol percentage in a co-solvent system was purposefully changed 45 and 55%. Celecoxib (90µg/ml) was prepared utilizing above mentioned co-solvent system independently, sample was analysed at a max wavelength 254nm for celecoxib content. The result was determined in terms of %RSD.

Table 6: Robustness

Sr. No.	Concentration (µg/ml)	Absorbance	% RSD
1	90	0.1987	0.38
2	90	0.2003	0.32

RESULTS AND DISSCUSION:

The method discussed in the present work provides a simple, stable, rapid, accurate, precise, reliable less expensive, time-saving and convenient method for the analysis of Celecoxib using U.V. spectrophotometry. λ max selected for quantitation was 254 nm. In the developed analytical method, the linearity was observed 0.9993 in the concentration range 0.2µg/ml to 1µg/ml.

Accuracy of the proposed method was ascertained by recovery studies and the results were expressed as percent recovery and were found in the Range 97.6% to 99.2%.

Values of standard deviation and coefficient of variance were satisfactorily indicating the accuracy of both methods. Intra-day and Inter-day precision studies were carried out/by analysing the sample of Celecoxib different time interval on the same day and on different days respectively. Standard deviation and coefficient of variance for Intra-day and Inter-day precision studies was found to be less than 2 indicating precision of the proposed method.

Based on the outcome of analytical method development and analytical validation study test results, it was found that, the proposed analytical method for estimation of Celecoxib by UV Spectrophotometry is Accurate, Precise, Reproducible, Stable, Simple, Rapid and less expensive (Economical). The analytical method can be employed for routine quality control estimation of Celecoxib formulation drug in pharmaceutical analysis.

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