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U.V. Analytical Method Development and Validation of Piroxicam Bulk API

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Abstract—The study was aimed to developed a sensitive ultraviolet (UV) spectrophotometric method for the determination of piroxicam bulk API. The samples and standards were prepared in 0.1M methanolic HCL and the drug was quantified at a lambda max of 333nm using a double beam UV spectrophotometer. The range of linearity for the method was found to be between 2-14 µg/ml, which followed Beer-Lambert's law and the regression coefficient near 1. The validity of the method was extensively tested using statistical parameters such as linearity, range accuracy, precision, LOD and LOQ. The proposed method performed well in terms of mean values and standard deviations. The method was found to be accurate, precise, specific, and fast, making it a reliable way to analyze Piroxicam.

Keywords— *Accuracy; Linearity; Method development and Validation; Piroxicam; Precision; UV Spectrophotometric method.*

I. INTRODUCTION

Piroxicam, a potent anti-inflammatory drug from the oxicam group of NSAIDs, is commonly used to alleviate symptoms of arthritis, gout, and other inflammatory conditions, both orally and topically. It is chemically known as 2H, 1,2-Benzothiazine-3-carboxamide-4-Hydroxy-2-methyl-N-2-pyridinyl-1,1-dioxide and exhibits an odourless appearance ranging from off-white to light brown to light yellow. With a molecular weight of 331.35 and a melting point of 198-200ºC (1)(2).

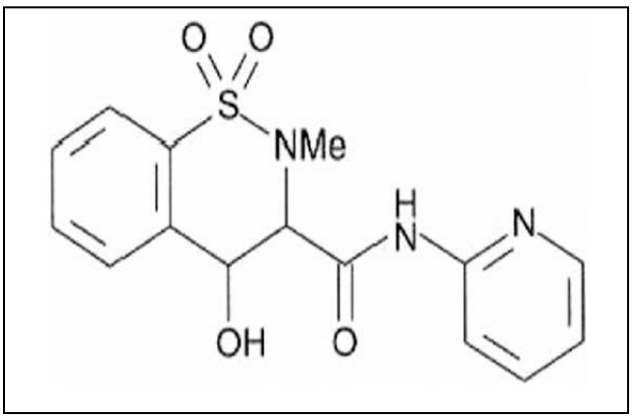

Fig. 1. Structure of Piroxicam

Piroxicam works by inhibiting the production of prostaglandins, which are hormones that cause pain and inflammation in the body. It is commonly used to treat osteoarthritis and rheumatoid arthritis pain, swelling, and stiffness. Piroxicam's anti-inflammatory effects are caused by its reversible inhibition of cyclooxygenase, an enzyme responsible for producing prostaglandins. Moreover, piroxicam suppresses platelet production of the aggregating chemical thromboxane A2 and leucocyte migration into areas of inflammation. It has 99% protein binding and belongs to BCS class II. Piroxicam has a 0.14 l/kg volume of distribution, and the recommended dosage is 20 mg orally or 0.5% w/w topically. (3)(4)(5)(6)

Piroxicam is an official drug listed in the Indian Pharmacopeia (7), British Pharmacopeia (8), European Pharmacopeia (9), and United States Pharmacopeia (10). All the pharmacopeias recommend an HPLC-based method for analyzing piroxicam. This study aims to develop and validate a simple, precise, accurate, specific, and fast UV-based method for analyzing piroxicam bulk API.(11)(12)

II. MATERIALS AND METHODS

Chemicals and Reagents:

Piroxicam was gifted by Sovereign Pharma Pvt Ltd, and all the other reagents are analytical grade and freshly prepared for the study. Analysis was conducted using a double-beam UV spectrophotometer (Shimadzu 1800).

Analytical Method Development:

A. Determination of Absorption maxima (max) of piroxicam:

Accurately 50mg of piroxicam was weighed and added to a 50ml volumetric flask. The volume was made up to volume with 0.1M methanolic HCL (1000µg/ml). From this, 1ml of the solution was withdrawn and diluted to 10ml, and 1ml was then withdrawn from this solution and further diluted to 10ml (10µg/ml). The resultant solution was scanned in a UV-Visible double-beam spectrophotometer in the range of 200 400 nm for lambda max, and the process was repeated in triplicate.

B. Development of calibration plot of piroxicam in 0.1M methanolic HCL:

50mg of the drug was added to a 50ml volumetric flask and make up volume to the solution with 0.1M methanolic HCL. From this solution, 2ml was taken and diluted to 100ml (20µg/ml). Then, 1ml, 2ml, 3ml, 4ml, 5ml, 6ml, and 7ml were withdrawn from this solution and diluted up to 10ml, resulting in concentrations of 2, 4, 6, 8, 10, 12, and 14 µg/ml,

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respectively. The process was repeated in triplicate.

Method Validation:

The validation of the developed method was carried out according to ICH Q2 (R2).

C. Linearity:

Linearity was calculated using the regression method for the calibration curve prepared in triplicate for concentrations of 2, 4, 6, 8, 10, 12, and 14µg/ml.

D. Range:

The range of the method is between the lowest and highest concentration for which absorbance readings are linear.

E. Limit Of Detection (LOD) and Limit of Quantification (LOQ):

The LOD and LOQ were determined using the following formula: LOD= 3.3σ/S and LOQ=10σ/S. Here, σ is the standard deviation of the y-intercept of the equation of the line, and S is the slope of the calibration curve.

F. Accuracy:

The accuracy of the method was calculated by conducting recovery studies. A concentration of 5 µg/ml was selected and considered as 100%. To this concentration, the drug was spiked at 80%, 100%, and 120% levels, and the recovery of the drug was calculated.

G. Precision:

The precision of the method was evaluated by taking a single concentration absorbance at three different times in a day that is intraday for three consecutive days that is interday.

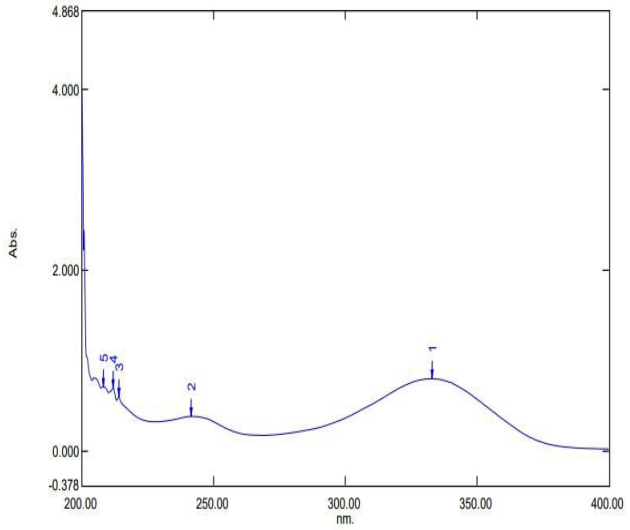

Fig. 2. Scan of piroxicam between 200-400nm.

Method validation of piroxicam in 0.1M methanolic HCL

III. Result and Discussion

A. Determination of Absorption maxima (max) of piroxicam:

The lambda max of Piroxicam in 0.1M methanolic HCl was found to be 333nm when scanned between 200nm to 400nm. (Shown in figure 2).

B. Linearity:

A UV spectrophotometric method for Piroxicam has been developed, and the Beer-Lambert law was followed between the concentration range of 2–14µg/ml, with a regression coefficient (R2) near to 1, i.e., 0.9997. This indicates that the method is highly linear and can accurately determine the concentration of Piroxicam in a sample over this concentration range. (Shown in figure 3).

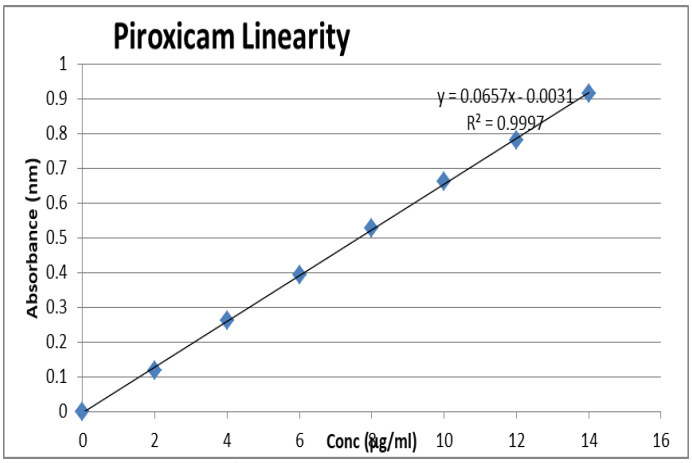

Fig. 3. Standard plot of piroxicam in 0.1M methanolic HCL at 333nm.

The relative standard deviation (RSD) is low, which confirms the reliability and validity of the method. (See table 1 for result). The absorbance range was found to be linear between 0.119 - 0.913, indicating that the method is sensitive enough to detect Piroxicam at low concentrations and can accurately quantify Piroxicam in a sample within this range.

TABLE 1. Absorbance of piroxicam in 0.1M Methanolic HCL.

Sr No	Conc (µg/ml)	Absorbance (n=3)	SD	RSD
1	0	0	0	0
2	2	0.119	0.00216	1.815334
3	4	0.26	0.003742	1.439099
4	6	0.393	0.004497	1.145224
5	8	0.527	0.005715	1.084531
6	10	0.662	0.007587	1.147158
7	12	0.78	0.008654	1.109941
8	14	0.913	0.012392	1.357752

TABLE 2. Optimal characteristics for Piroxicam in 0.1M methanolic HCL.

Parameters	Values
Lambda max	333nm
Linearity range	2-14 µg/ml
Slope	0.0657
Intercept	-0.0031
Correlation coefficient	0.9997
LOD	0.281425
LOQ	0.852802

C. LOD & LOQ:

LOD and LOQ of the method was found to be 0.281425 and 0.852802 µg/ml (as given in Table 2), these demonstrate the method is very sensitive and can detect even small quantity of drug.

D. Accuracy

Accuracy of method was assessed by recovery study (results are shown in Table 3) which shows good

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reproducibility. %RSD is below 2 % which confirm the corrections in reading. The percentage recovery is between 98.01-101.42%. This proves that method is accurate.

TABLE 3. Results of Accuracy of Piroxicam

SR NO	Test Concentration	Amount Spiked	% Recovery	% Mean Recovery	SD	RSD
1		4 ppm	97.8916	98.01	1.32	1.34
	5ppm	4 ppm	96.762			
		4 ppm	99.3976			
2		5ppm	101.254	101.04	1.26	1.25
	5ppm	5ppm	99.6865			
		5ppm	102.194			
3		6ppm	101.427	101.42	0.509	0.50
	5ppm	6ppm	100.917			
		6ppm	101.937			

E. Precision:

The result of intra and inter day was given in the table 4 and 5, there is negligible variation intra and inter day which can be confirmed from low %RSD value. Percentage RSD of interday precision was between 0.45-0.52, intraday precision was between 0.89-1.2. The % RSD was found lesser than 2 which promise the precision of the developed method.

TABLE 4. Result Intraday (same day) Precision

Sr no	Time	Conc	Abs	Mean	SD	RSD
		5ppm	0.334	0.338	0.0036	1.06
1	Morning	5ppm	0.339			
		5ppm	0.341			
		5ppm	0.344	0.340333	0.0040	1.18
2	Afternoon	5ppm	0.341			
		5ppm	0.336			
		5ppm	0.339	0.339	0.003	0.88
3	Evening	5ppm	0.342			
		5ppm	0.336			

TABLE 5. Result Interday (different day) Precision

Sr no	Time	Conc	Abs	Mean	SD	RSD
		5ppm	0.34	0.341667	0.001528	0.44
1	Day 1	5ppm	0.342			
		5ppm	0.343			
		5ppm	0.336	0.337	0.001732	0.51
2	Day 2	5ppm	0.339			
		5ppm	0.336			
		5ppm	0.339	0.340667	0.001528	0.44
3	Day 3	5ppm	0.342			
		5ppm	0.341			

IV. Conclusion

From the above result it can be conclude that this research successfully developed and validated a UV-based method for analyzing Piroxicam bulk API. The method's adherence to Beer-Lambert's law, coupled with its linear concentration range of 2-14 µg/ml and a regression coefficient near 1, proved highly accurate, precise, specific, and rapid. The lambda max of 333nm, determined using a double beam UV spectrophotometer, provides a dependable means of analyzing Piroxicam. Overall, this method represents a valuable contribution to the field of pharmaceutical analysis and can serve as a reliable technique for the assessment of Piroxicam bulk API.

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