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RESEARCH ARTICLE

DIFFERENTIAL SPECTRO-PHOTOMETRIC METHOD FOR DETERMINATION OF PIROXICAM IN PAREN-TRAL DOSAGE FORMS.

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Abstract: A difference spectrophotometric method for the estimation of piroxicam in pharmaceutical formulation was developed. The method is based on the nullification of UV absorbance at the wavelength corresponding to the point of intersection of drug spectra in acidic and basic media. The amplitude (λmax - λmin) of difference spectra followed Beer-Lambert's law in 2-10 µg/ml concentration range. The method was validated by statistically and recovery studies and was found to be accurate and precise.

Key words: Rosuvastatin calcium; Normal phase HPLC; Enantiomer; Impurities; Chiral method.

INTRODUCTION:

Piroxicam has anti-inflammatory, analgesic, and antipyretic properties in animals. Edema, tissue proliferation, fever, and pain can all be inhibited in laboratory animals by the administration of piroxicam. It is effective regardless of the etiology of the inflammation. The mode of action of piroxicam is not fully established at this time. However, a common mechanism for the above effects may exist in the ability of piroxicam to inhibit the biosynthesis of prostaglandins, known mediators of inflammation.

Several methods have been described for piroxicam determination in biological fluids and in pharmaceuticals by chromatographic[1-3], spectroscopic[4-6], and electrochemical[7-8] and capillary zone electrophoresis[9] techniques.

In this paper, we describe a sensitive, difference spectrophotometric method for the estimation of piroxicam in parentral dosage form. The proposed method is advantageous over other method because interference of additives is nullified as can be proved by no change in isobestic point.

MATERIALS AND METHODS

Helios α-double beam recording spectrophotometer with 10 mm matched quartz cells was used for the determination of absorbance and recording the spectrum. The standard drug piroxicam was procured from Pfizer Limited, Mumbai and formulations were obtained from drug store in market. Methanol AR grade, glacial acetic acid AR grade, sodium hydroxide AR grade from Ranbaxy Limited, Delhi, were used.

Solution Preparation

The standard drug piroxicam, 50 mg was dissolved in methanolic-hydrochloric acid and volume was made up to 50 ml using the same solvent. 10 ml of this solution was pipetted out into 100 ml volumetric flask and its volume was made up with the same solvent to get a working standard stock solution of 100 µg/ml.

0.1 N methanolic-hydrochloric acid solution (8.5 ml of concentrated hydrochloric acid was dissolved in 1000 ml of distilled methanol), 0.1 N glacial acetic acid solution (5.7 ml of glacial acetic acid was dissolved in 1000 ml of distilled water) and 0.1 N sodium hydroxide solution (4.0 g of sodium hydroxide pellets were dissolved in 1000 ml of distilled water) were prepared.

Different aliquots were taken from the stock solution (100 µg/ml) in to a series of 10 ml volumetric flasks to prepare different concentration of dilutions (2, 4, 6, 8 and 10 µg/ml) and graph was plotted against wavelength versus absorbance, are shown in fig. - 1, and also different aliquots were taken from the stock solution (100 µg/ml) in to a series of 10ml volumetric flasks to prepare different dilutions with acidic and alkaline solutions against the respective blank solution.

Acidic solution of known strength was kept in sample cell and alkaline solution of the same strength was kept in reference cell to record different spectra. The three spectra A (acidic), B (basic) and D (difference), are shown in fig. - 2. The amplitude of difference spectra obtained for various concentrations were plotted against the concentration to obtain a standard curve, are shown in fig. - 3.

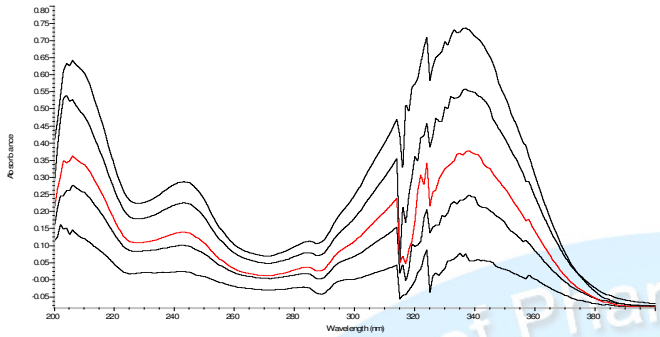
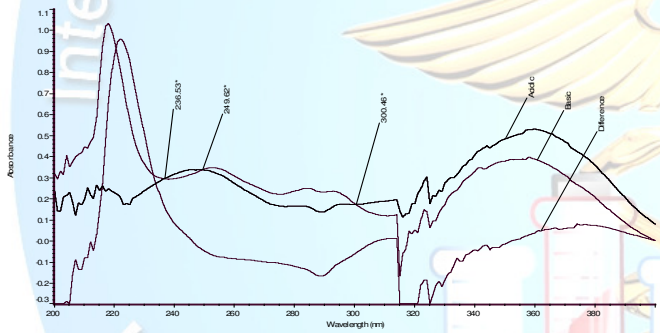


Figure 1: UV spectra of piroxicam on different concentration

Five ampoules were procured from Pfizer Limited, Mumbai. Each ampoule (2 ml) is equivalent to 20 mg/ml was taken and dissolved in methanolic-hydrochloric acid solution and made up the volume to 20 ml. Then 10 ml of this solution was diluted to 100 ml with respective solution to get 100 µg/ml concentration. Then concentrations were obtained on dilutions (2, 4, 6, 8 and 10 µg/ml). Similar procedure was followed for piroxicam obtained from Cipla Limited, Goa.



* = Isobestic Point

Figure 2: Overlain acidic, alkaline and difference spectra of piroxicam

The difference spectra of appropriately diluted solutions, acidic and alkaline medium were recorded and are reported in Table 1. The amount of drug present was calculated from the standard curve equation, are shown in Table 2.

To study the accuracy, reproducibility and precision of the proposed method recovery experiments were carried out. A fixed amount of the pre-analyzed sample was taken and standard drug was added at three different levels. Each level was repeated at least 5 times. The summaries of the recovery studies are reported in Table 3. The λmax in acidic and alkaline solutions were found to be 342 and 357 nm respectively. The chosen λmax and λmin of difference spectra were at 338 and 242 nm. The isobestic points were at 236.53, 249.62 and 300.46.

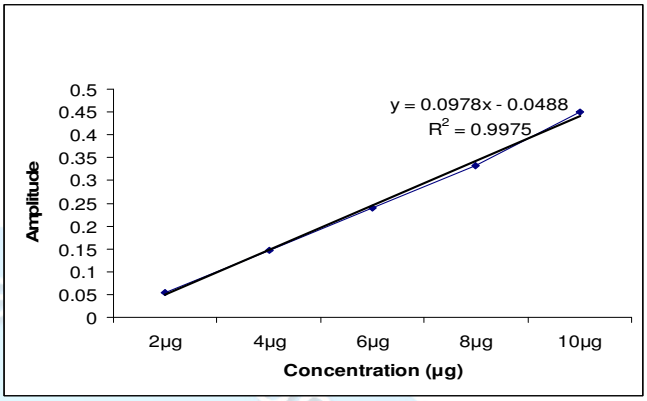


Figure 3: Difference standard curve of piroxicam

Table 1: Difference Standard Curve of Piroxicam

Sl. No.	Concentration (µg/ml)	Absorbance		Amplitude
		λmax	λmin	
1.	2	0.076	0.022	0.054
2.	4	0.247	0.100	0.147
3.	6	0.376	0.137	0.239
4.	8	0.556	0.223	0.333
5.	10	0.734	0.284	0.450

Table 2: Analysis of Formulation Of Piroxicam

Sl. No.	Formulation	Labeled amount (ml/ampoule)	Amount found (ml/ampoule)
1	T_1*	2	2.001
2	T_2*	5	4.998

* = Different formulation of piroxicam

Table 3: Recovery Studies Of Piroxicam

Sl. No.	Amount added (ml)	% Recovery	% Recovery (Mean±SD)
1	2	100.1	99.5 ± 0.53
2	6	99.1	
3	8	99.3	

An excellent correlation was found between amplitude and concentration with value r = 0.9975 and intercept 0.0488. Slope was found to be 0.0488. The linearly regressed equation of standard curve y = 0.0978x + 0.0488 followed the Beer - Lambert's law in the concentration range of 2-10 µg/ml. The % recovery and standard deviation showed that the proposed method was found to be reproducible, accurate and precise.

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