

Simple Spectrophotometric Methods for determination of Piroxicam in Pharmaceutical Formulation

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Abstract: Simple sensitive and accurate spectrophotometric methods have been developed for the estimation of piroxicam in pharmaceutical dosage form. Methods I and II were based on reactions of para-aminophenol and metol with piroxicam in presence of sodium carbonate with maximum absorbance at 400 nm and 395 nm respectively. Method III was based on reaction of piroxicam with sodium nitroprusside in presence of sodium hydroxide with maximum absorbance at 395 nm. The proposed methods were validated statistically. Recoveries of methods were carried out by standard addition method. The linearity was found to be 10-60 µg/ml, 5-50 µg/ml and 10-70 µg/ml for methods I, II and III respectively. The low values of standard deviation and percentage RSD indicate high precision of methods. Hence these methods are useful for routine estimation of piroxicam in capsules.

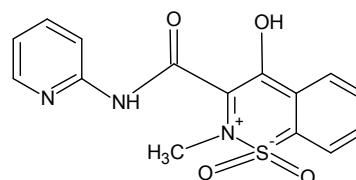
Keywords : Piroxicam, Para-aminophenol, Metol Sodium nitroprusside.

Introduction

Piroxicam is 4 hydroxy-2-methyl-N (2-pyridyl) 2 H-1,2-benzothiazine-3-carboxamide-1,1-dioxide, is a potent anti-inflammatory agent and differs radically in chemical structure from all commonly used non-steroidal anti-inflammatory drugs (NSAID). It is acidic, highly potent. It is used in variety of anti-inflammatory conditions such as rheumatoid arthritis, Osteo-arthritis and gout. Piroxicam has relative low toxicity and longer elimination half life in man compared to other NSAID.

Quantitative determination of the drug is very important in pharmaceutical control and assurance. The drug is been officially reported in IP^[1] and USP^[2] described the assay by high performance liquid chromatographic method. In BP^[3] the assay of related substances of piroxicam capsules were described by thin layer chromatography method. The literature survey revealed HPTLC^[4], GLC^[5], HPLC^[6-8], Spectrophotometric^[9-20] and Polarography^[21-24] methods for determination of piroxicam. In the proposed methods, an attempt has been made to

develop simple and suitable spectrophotometric methods for quantitative determination of piroxicam. The developed spectrophotometric methods were successfully validated.



Material and Methods :

A SHIMADZU UV-160 A double beam UV-VISIBLE recording spectrophotometer with pair of 10 mm matched quartz cells was used to measure absorbance of the solution. A Sartorius analytical balance with 0.01 mg was used. Para amino phenol, metol, sodium nirtroprusside, sodium carbonate, sodium hydroxide and methanol of A.R grade were used in the study.

Preparation of Standard solution

Stock solution of Piroxicam (200 µg/ml) was prepared in methanol. From this stock solution working standard for method I (10-60 µg/ml), method II (5-50 µg/ml) and method III (10-70 µg/ml) were prepared by appropriate dilutions. A 0.05 % w/v para-aminophenol, 0.05 % w/v metol, 0.05 % w/v sodium nitroprusside, 5 % w/v sodium hydroxide, 5% and 6% w/v sodium carbonate were prepared in distilled water.

Experimental**Method I** (with para-amino phenol)

Into a series of 10 ml graduated flask, varying amount of drug solutions were pipetted out. Then to each flask 0.5 ml of 0.05 % w/v para-aminophenol and 3 ml of 6% w/v sodium carbonate solutions were added. Solutions were allowed to stand for 30 minutes and volume was adjusted with methanol. Absorbance of the resulting solutions were measured at 400 nm.

Method II (with metol)

Into a series of 10 ml graduated flask, varying amount of drug solutions were pipetted out. Then to each flask 1.5 ml of 0.05 % w/v metol and 1.5 ml of 5 % w/v of sodium carbonate solution were added. Solutions were allowed to stand for 45 minutes and volume was adjusted with methanol. Absorbance of the resulting solutions were measured at 395 nm.

Method III (with sodium nitroprusside)

Into a series of 10 ml graduated flask, varying amount of drug solutions were pipetted out. Then to each flask 0.5 ml of 0.05 % w/v of sodium nitroprusside and 0.5 ml of 5 % w/v of sodium hydroxide solution were added. Solutions were allowed to stand for 20 minutes and volume was adjusted with methanol. Absorbance of the resulting solutions were measured at 395 nm.

Estimation from capsules.

Twenty capsules of labeled claim 10 mg of piroxicam were weighed accurately and powder from each capsule was collected carefully and it was mixed thoroughly. A portion equivalent to 20 mg was weighed accurately. It was transferred into a beaker and 25 ml methanol was added to dissolve the drug. It was filtered through whatmann filter paper no. 41. The filtrate and washing were collected in a 100 ml volumetric flask. Filtrate and washing were diluted up to the mark with methanol to obtained final concentration 200 µg/ml. This solution was used for method I, II and III respectively. Appropriate aliquots of drug solution were taken and the individual assay procedures were followed for the estimation of drug contents in capsules. The concentration of the drug in capsules was calculated using calibration curve. The recovery experiment was carried out by standard addition method. Results of analysis are given in Table 1.

Table1: Optical and regression of drug in different methods

Parameter	Methods		
	I	II	III
λ max (nm)	400	395	395
Beer Law Limits (µg/ml)	10-60	5-50	10-70
Molar absorptivity(l/mol.cm)	2.02125×10^3	3.44604×10^3	2.5514×10^3
Sandell's sensitivity	6.1×10^{-3}	1.04×10^{-2}	7.8×10^{-3}
Correlation coefficient	0.9992	0.9991	0.9995
Regression equation (y=b+ac)			
Slope (a)	0.0061	0.0104	0.0077
Intercept	0.001	0.001	0.001

Table 2 : Results of recovery of drug with different reagents

Reagent	Amount Label claim ($\mu\text{g/ml}$)	Amount of standard added ($\mu\text{g/ml}$)	Total amount recovered	Percent recovery (%)	Standard deviation	Percentage of relative standard deviation (C.O.V.)
Para amino phenol	10	0	9.994	99.94	0.13017	1.30169
	10	10	19.894	99.47	0.1222	0.61442
	10	20	29.999	99.99	0.1840	0.6135
	10	30	39.93	99.825	0.1102	0.27605
Metol	10	0	9.999	99.99	0.140806	1.840
	10	10	19.999	99.995	0.1480	1.840
	10	20	29.391	97.97	0.2611	0.8119
	10	30	40.183	100.457	0.2852	0.7096
Sodium Nitro-prusside	10	0	9.994	99.94	0.0474	0.4742
	10	10	20.0178	100.089	0.03047	0.15233
	10	20	30.0089	100.029	0.02362	0.0787
	10	30	39.866	100.336	0.237	0.1338

Result and Discussion

Para- amino phenol , metol and sodium nitroprusside react with piroxicam in basic medium to give coloured species. The working condition of these methods were established by varying one parameter at time and keeping the other parameter fixed by observing the effect produced on the absorbance of the colour species. The various parameters involved for maximum colour development for these methods were optimized. The proposed methods were validated statistically and by recovery studies .The molar absorptivity and Sandell's sensitivity values show the sensitivity of methods while the precision was confirmed by % RSD (relative standard deviation). Assay results of recovery studies are given in Table 2. Results are in good in agreement with labeled value. The percent recovery obtained indicates non interference from the common excipients used in the

formulation. The reproducibility, repeatability and accuracy of these methods were found to be good, which is evidenced by low standard deviation. Previous methods are tedious and time consuming, they require costly chemical hence they are not convenient for routine analysis. The proposed methods are simple, sensitive, accurate, precise and reproducible. They are directly applied to drug to form chromogen. Hence they can be successfully applied for the routine estimation of piroxicam in bulk and pharmaceutical dosage form even at very low concentration and determination of stability of drug in formulation such as capsules.

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