

Research Article

Development of Ultraviolet Spectrophotometric Method for Analysis of Lornoxicam in Solid Dosage Forms

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Abstract

Purpose: An ultraviolet spectrophotometric system was developed and validated for the quantitative determination of lornoxicam in solid dosage forms.

Methods: Lornoxicam was dissolved in 0.01M NaOH and analysed using ultraviolet (UV) spectrophotometry. Various analytical parameters such as linearity, precision, accuracy, limit of detection (LOD) and limit of quantification (LOQ) were determined according to International Conference on Harmonization (ICH) guidelines.

Results: Absorbance maximum in 0.01M NaOH was 377 nm. Beer's law was obeyed over the concentration range of 2 - 20 µg/ml with a correlation coefficient (r^2) value of 0.999. Percent range of error was 0.344 and 0.261 at 0.05 and 0.01 confidence limits, respectively. Intra- and inter-day precision (% RSD) at different concentration levels were < 2 %, indicating that the proposed derivative spectrophotometric method is highly reproducible during one run and between different runs; LOD and LOQ were 0.105 and 0.318 µg/ml, respectively signifying that it can be adopted for routine quality testing. Mean recovery was 100.82 % for tablets. Low values of % RSD indicate the reliability of the proposed method

Conclusion: The proposed method is highly sensitive, precise, accurate, cost-effective, reliable and rapid for the estimation of lornoxicam in solid dosage forms.

Keywords: Lornoxicam, UV Spectrophotometry, Quantitative determination, Solid dosage forms.

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INTRODUCTION

Lornoxicam (6-chloro-4-hydroxy-2-methyl-N-2-pyridyl-2H-thieno-[2,3-e]-1,2-thiazine-3-carboxamide-1,1-dioxide) a non-steroidal anti-inflammatory drug (NSAID) with potent analgesic and anti-inflammatory activity. It belongs to the class of oxicams and is approximately ten times more potent than oxicams such as piroxicam and tenoxicam [1]. It is used for the treatment of rheumatoid arthritis and other rheumatic diseases and is available in the market as tablet and injection.

A literature survey showed that the analysis of lornoxicam in pharmaceutical preparations is usually by high performance liquid chromatography (HPLC), voltammetry as well as zero- and first-order derivative UV spectrophotometric methods [2]. However, to the best of our knowledge, no method has been established and validated by linear regression equation method which is a promising straightforward method that has gained extensive recognition.

The objective of the present study was to develop a simple, precise and reliable spectrophotometric method, using 0.01M NaOH as solvent, as a rapid, direct and cost-effective alternative to the determination of lornoxicam in pharmaceutical formulations.

EXPERIMENTAL

Materials

A commercial brand of lornoxicam tablet, Zion[®], (8 mg lornoxicam, Unichem, India) was obtained from a local pharmacy. Lornoxicam powder (purity, 99.9 %), received *gratis* from Hetero Drugs, Hyderabad, India, was used as such without further purification. All other chemicals used were of analytical grade. All the solutions for analysis were freshly prepared and analyzed. Spectrophotometric analysis was performed with Systronic 2101 and Elico SL 159 UV-Vis spectrophotometer. Addir Dutt electronic

balance (AD-50B, Kolkata, India) was used for the assessment of tablet weight variation.

Preparation of working standard solutions

A working standard solution of lornoxicam was prepared by dissolving 50 mg of the standard (pure) lornoxicam in an appropriate volume of 0.01M NaOH to obtain a solution of 100 µg/ml concentration. Aliquots (1 - 10 ml) of the working standard solution were transferred into a series of 50 ml volumetric flasks to obtain final drug concentrations of 2 - 20 µg/ml.

Analysis of the drug and validation of the proposed method

Twenty tablets of lornoxicam were crushed in a mortar and pestle to a fine powder and an amount equivalent to 8 mg (accurately weighed) was extracted at room temperature in a shaker with 100 ml 0.01M NaOH for 45 min. The mixture was filtered through Whatman filter paper no. 1. and suitably diluted with 0.01M NaOH to obtain a concentration of 10 µg/ml of lornoxicam [3]. The absorbance of these solutions was determined at 377 nm against a reagent blank (0.01M NaOH solution). The repeatability of the method was established by carrying out analysis (n = 8) of the analyte (10 µg/ml) using the proposed method [4].

Recovery studies

The accuracy of the developed assay method was evaluated by evaluating the recovery of lornoxicam by standard addition method at concentrations of 80, 100 and 120 % of the target level in tablets [5]. The percent analytical recovery was calculated by comparing the concentration obtained from spiked samples with actual added concentration and the values are listed in Table 1.

The precision of the method was demonstrated by inter-day and intra-day variation studies using three different concen-

Table 1: Recovery data for lornoxicam tablets by proposed method

Sample ID	Concentration of pure drug taken ($\mu\text{g/ml}$)	Concentration of tablet formulation ($\mu\text{g/ml}$)	% Recovery of pure drug (n=3)	% RSD
C : 80%	8	10	99.91 \pm 0.830	0.831
C : 100%	10	10	100.38 \pm 0.880	0.877
C : 120%	12	10	102.18 \pm 0.322	0.316

Note: RSD = relative standard deviation

Table 2: Precision data (n = 3)

Amount taken ($\mu\text{g/ml}$)	Inter-day		Intra-day	
	Content ($\mu\text{g/ml}$)	% RSD	Content ($\mu\text{g/ml}$)	% RSD
8	7.940 \pm 0.054	0.690	7.98 \pm 0.013	0.168
10	9.940 \pm 0.077	0.775	9.98 \pm 0.014	0.134
12	11.960 \pm 0.044	0.372	11.97 \pm 0.026	0.225

Note: RSD = relative standard deviation

trations of drug (in addition to calibration standards) covering the entire linearity range [6]. In intra-day studies, the drug was analyzed on the same day while inter-day precision was determined by analyzing the drug for three consecutive days. The results are presented as standard deviation and % relative standard deviation (R.S.D) in Table 2.

Reliability

The reliability of the proposed method was determined at 12 $\mu\text{g/ml}$ by using different instruments that is by Systronic 2101 and Elico SL 159 UV-Vis spectrophotometer under similar setting [7].

Limits of detection (LOD) and quantification (LOQ)

LOD and LOQ were calculated as 3.3 σ/S and 10 σ/S , respectively, where σ is the standard deviation of the standard concentration and S is the slope of the standard curve. The limit of detection (LOD) is the lowest concentration of an analyte that an analytical process can reliably differentiate from background levels. The limit of quantification (LOQ) is the lowest concentration of the standard curve that can be measured with acceptable accuracy, precision and variability [8].

Statistical analysis

The data obtained were expressed as mean \pm standard deviation (SD), and statistical analysis of the data was performed using one-way ANOVA (Graph Pad Prism software, version 5). Differences between formulations were considered to be significant at $p \leq 0.05$.

RESULTS

The optical characteristics were as follows: Beer's law limit, 2 to 20 $\mu\text{g/ml}$; absorbance maximum, 377 nm; molar absorptivity, 465.065 $\text{L M}^{-1} \text{cm}^{-1}$; Sandell's sensitivity, 0.023 $\mu\text{g cm}^{-1}/0.001$ absorbance unit; correlation coefficient (r^2), > 0.999; regression equation, $y = 0.043X + 0.016$; slope, 0.043; intercept, 0.016; % RSD, 0.834; and % range of error, 0.344 and 0.261 at 0.05 and 0.01 confidence limits, respectively. Molar absorptivity and Sandell's sensitivity data show that the method is perceptive. The assay value for the commercially marketed tablet was 99.91 % with a % R.S.D. 0.831.

Mean recovery was 100.82% for the tablets by the proposed method while LOD and LOQ were 0.105 and 0.318 $\mu\text{g/ml}$, respectively. The intra- and inter-day precision (% RSD) at different concentration levels were < 2 % (Table 2), indicating that the proposed derivative spectrophotometric method is

Table 3: Results of reliability studies

UV spectrophotometer	Amount taken ($\mu\text{g/ml}$)	Amount found ($\mu\text{g/ml}$)	% RSD
Systronics-2101	12	11.98 \pm 0.01	0.083
Elico SL-159	12	11.95 \pm 0.02	0.174

Note: RSD = relative standard deviation

highly reproducible during one run and between different runs [9]. Low values of % RSD for reliability studies (Table 3) at $p \leq 0.05$ indicate that there was no statistically significant difference between the two different UV spectrophotometric instruments used in a similar setting.

DISCUSSION

The present study is an attempt to develop a simple, accurate and sensitive UV spectrophotometric method for the determination of lornoxicam in solid dosage forms, specifically tablets. The linearity of lornoxicam solution in the concentration range 2 to 20 $\mu\text{g/ml}$ was satisfactory with absorbance maximum at 377 nm. The low values of standard error established the precision of the proposed method. The low values of LOQ and LOD of the proposed method signify that it can be adopted for routine quality testing and dissolution studies. Sample recovery for tablets was in good agreement with its label claim. The within-day and between-day precision (% RSD) were satisfactory and established the repeatability and reproducibility of the proposed method.

CONCLUSION

The proposed UV method for the analysis of lornoxicam is simple, fast and reliable, providing satisfactory accuracy and precision. Moreover, the short duration of analysis of lornoxicam make the developed method suitable for the routine quantitative analysis of lornoxicam in solid dosage formulations.

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Sahoo et al

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