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Research Article

DEVELOPMENT AND VALIDATION OF LORNOXICAM BY SECOND ORDER DERIVATIVE SPECTROSCOPY

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ABSTRACT

Simple, precise and cost effective second order derivative spectrophotometric method has been developed for the estimation of Lornoxicam in bulk and its pharmaceutical formulations. Lornoxicam shows λ_{max} at 257.2 nm in second order derivative spectrum. The drug follows Beer-Lambert's law in the concentration range of 7.5 – 25.0 µg/ml with correlation coefficient of 0.999. The method was validated by following analytical performance parameters suggested by the International Conference on Harmonization. All validation parameters were within the acceptable range. The developed method was successfully applied to estimate the amount of Lornoxicam in bulk and pharmaceutical dosage form.

Keywords: Lornoxicam, Derivative spectroscopy, UV Spectrophotometry

INTRODUCTION

Lornoxicam is chemically, 6-chloro-4-hydroxy-2-methyl-N-2-pyridinyl-2H-thieno-[2,3-e]-1,2-thiazine-3-carboxamide 1,1-dioxide. It is a novel non-steroidal anti-inflammatory drug (NSAID) with marked analgesic activity. It works by blocking the action of cyclooxygenase, an enzyme involved in the production of prostaglandins from Arachidonic acid in the body[1].

Lornoxicam is an official drug in Merck Index. On detailed literature survey, it was found that Lornoxicam can be estimated by spectrophotometry[2], Polarographic[3], HPLC[4-5], Liquid Chromatography[6] methods individually or in combination with other drugs. The aim of the present work is to develop and validate second order derivative spectrophotometric method for the estimation of Lornoxicam in bulk and pharmaceutical formulations.

MATERIAL AND METHODS

Chemicals and reagents

Lornoxicam working standard was kindly provided by Glenmark Generics Ltd., (Pune) and was used as received. A commercial tablet formulation was purchased from the local market. Sodium hydroxide (0.1N) of analytical grade solution was prepared in double distilled water.

Instrument

A double beam UV-VIS spectrophotometer (UV CE7400, Cecil, UK) connected to computer loaded with spectra manager software UV probe was employed with **s**pectral bandwidth of 1 nm and wavelength accuracy of ±0.5 nm with a pair of 10 mm matched quartz cells. All weights were taken on electronic balance (XB120A, Precisa, Switzerland).

Preparation of standard stock solution

The standard solution of Lornoxicam was prepared by dissolving accurately weighed 10 mg of the drug in 0.1N sodium hydroxide and diluted to 100 ml with 0.1N sodium hydroxide to obtain a final concentration of 100 µg/ml. This stock solution was used to prepare further dilutions of standard solutions.

Determination of wavelength of maximum amplitude (D^2 value) of Lornoxicam

10 ml of above solution was diluted to 100 ml with the same solvent to get the concentration of 10 µg/ml. The UV spectrum of final solution obtained was scanned in the range of 200 to 400 nm against 0.1N sodium hydroxide as a blank. The λ_{max} was found 257.2 nm. The UV spectrum of lornoxicam is shown in Fig 1.

Preparation of calibration curve for lornoxicam

0.75 ml, 1.0 ml, 1.25 ml, 1.5 ml, 1.75 ml, 2.0 ml, 2.25 ml and 2.5 ml solutions were pipetted out individually from the Standard stock solution in a series of eight, 10 ml volumetric flasks. The volume in each flask was made up to 10 ml with 0.1N sodium hydroxide to yield final solution in the concentration range of 7.5 to 25 µg/ml. Then the amplitude (D^2 value) of all the solutions was measured at λ_{max} of drug, i.e. 257.2 nm, against 0.1N sodium hydroxide as a blank. The results of calibration curve data for Lornoxicam are shown in Table 1 and the calibration curve is depicted in Fig 2.

Estimation of Lornoxicam in tablets

Twenty tablets of Lornoxicam were weighed and finely powdered. A quantity of powder equivalent to 8 mg of the drug was transferred to a 100 ml volumetric flask and dissolved in 40 ml 0.1N sodium hydroxide by keeping on an ultrasonic water bath for 20 minutes. The solution was diluted to volume and filtered through Whatman filter paper no. 40. After suitable dilution, the spectrum of the final sample corresponding to 8.0 µg/ml was recorded against 0.1N sodium hydroxide as blank. The results are shown in **Table 2.**

Method validation

Linearity: A calibration curve was constructed at optimum experimental condition using D^2 (Second order derivative) values versus concentration in the range of 7.5-25 µg/ml. It has shown linear relationship with the regression equation y= 0.00007c + 0.00003, where 'y' is amplitude of the peak at 257.2 nm of second derivative spectra and 'c' is the concentration of the sample in µg/ml. High value of correlation coefficient (0.999) indicates good linearity and adherence of the method to Beer's law.

Precision: The intraday and interday precisions of developed method were determined by estimating the corresponding response three times on the same day and on three different days over a period of week for three different concentrations of Lornoxicam (13.0 µg/ml, 16.25 µg/ml, 19.5 µg/ml) and the results are reported in terms of relative standard deviation in Table 3.

Accuracy: This parameter was evaluated by the percent recovery studies at concentration levels of 80, 100 and 120%, which consisted of adding known amounts of Lornoxicam reference materials to a prequantified sample solution. Aliquots of sample solutions containing Lornoxicam at 10.0 µg/ml were transferred to three 10 ml volumetric flasks containing, respectively, 0.8, 1.0, and 1.2 ml Lornoxicam reference solution (100 µg/ml). The contents were mixed and diluted to volume in order to obtain final concentrations of 18.0, 20.0, and 22.0 µg/ml respectively. The recovery was verified by estimation of drugs in triplicate preparations at each specified

concentration level. The spectrums were recorded in the UV range and then analyzed. The results are reported in terms of % recovery in Table 3.

Specificity: Commonly used excipients present in selected tablet formulation were spiked into a preweighed quantity of drugs. The D^2 value was measured.

RESULTS AND DISCUSSION

According to the International Conference on Harmonization[7], the main objective of the validation of an analytical procedure is to demonstrate that it is suitable for its intended purpose, and the parameters that need to be selected are the responsibility of the analyst. the solubility of Lornoxicam in dilute sodium hydroxide, so it was used in this method. Lornoxicam in 0.1N NaOH shows absorption maxima at 257.2 nm in second order derivative spectrum. The response for Lornoxicam was found to be linear in the concentration range of 7.5–25.0 µg/ml. The optical characteristics of the method and regression analysis of the calibration curve are shown in Table 3.

The recovery of Lornoxicam was found to be satisfactory. Excipients used in the specificity study did not interfere with response of the drug at its analytical wavelength. Also, no significant change in response of Lornoxicam was observed after 24 hrs. Hence, the method is specific and robust for estimation of Lornoxicam. The developed spectrophotometric method was applied to the determination of Lornoxicam in its pharmaceutical formulations.

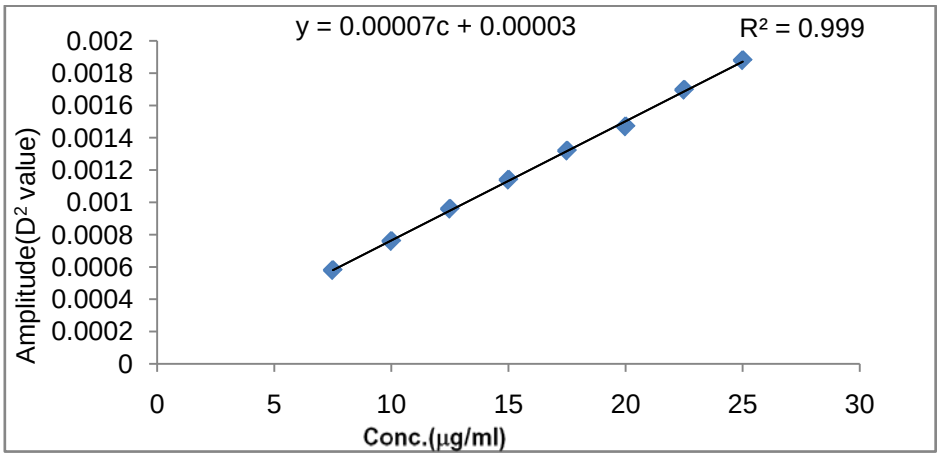

Fig. 1: It shows second derivative UV spectrum of Lornoxicam

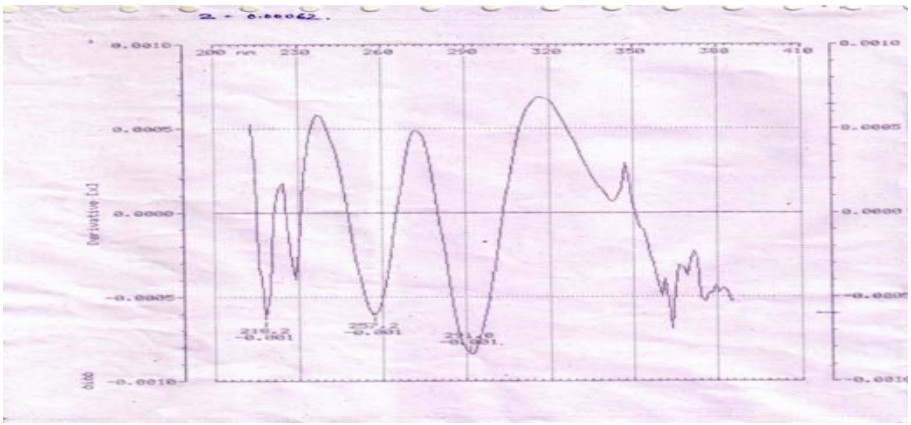

Fig. 2: It shows Calibration curve of lornoxicam at 257.2 nm

Table 1: shows calibration curve data for Lornoxicam

Conc. (µg/ml)	D^2 value (amplitude)
7.5	0.000580
10.0	0.000760
12.5	0.000960
15.0	0.001140
17.5	0.001320
20.0	0.001470
22.5	0.001697
25.0	0.001880

Table 2: Shows assay result of Lornoxicam in tablets

Label claim (mg/tab)	Amount found (mg/tab)	Standard deviation	% Mean recovery
8.0	7.976	1.755	99.702

Table 3: Shows optical characteristics and validation parameters of Lornoxicam

Parameter		Values
Beer's law limit (µg/ml)		7.5-25
λ_{max} (nm)		257.2
Molar absorptivity (mole^{-1} cm^{-1})		245.395
Regression equation (Y=a + bc)		Y=0.00007c +0.00003
Correlation coefficient (r^2)		0.999
Slope (b)		0.00007
Intercept (a)		0.00003
Limit of detection (µg/ml)		0.27086
Limit of quantitation (µg/ml)		0.90286
Precision (RSD, %)	Repeatability	0.96090
	Intraday	0.94630
	Interday	1.3200
Accuracy (% recovery)		100.5965

CONCLUSION

The method was validated and found to be simple, sensitive, accurate, and precise. Hence, the method can be used successfully for routine analysis of pharmaceutical dosage form of Lornoxicam.

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