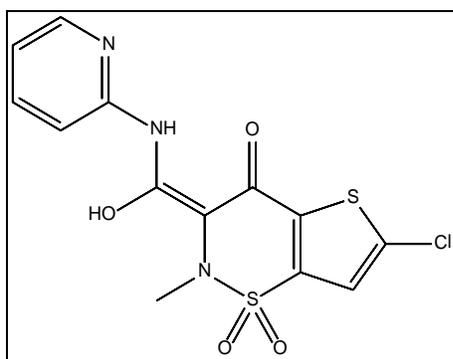


Determination of lornoxicam by UV spectroscopic method in bulk and combined dosage form**Shoab Mohammad Syed^{1*}, V G Rajurkar², R P Marathe³**¹ Assistant Professor, Dr Vedprakash Patil Pharmacy College, Aurangabad, Maharashtra, India² Principal, Dr. Vedprakash Patil Pharmacy College Aurnagabad, Maharashtra, India³ Professor and Principal, I/c Govt College of Pharmacy Ratnagiri, Maharashtra, IndiaDOI: <https://doi.org/10.33545/26647222.2021.v3.i1a.18>**Abstract****Objective:** In the present study a suitable UV Spectroscopic method was developed and validated for Lornoxicam determination in bulk and combined dosage form.**Methods:** UV Spectrophotometric method was performed at 376nm and samples were prepared with a solution of 0.1N HCl, validation parameters like accuracy, precision, LOD, LOQ, recovery study and range were determined.**Results:** The linearity demonstrated a correlation coefficient of 0.9992 various validation parameters like accuracy, precision, LOD, LOQ, recovery study and range were found to be within the specified range.**Conclusion:** The proposed method was simple, rapid, precise, accurate and sensitive and can be used for routine analysis of Lornoxicam in bulk and combined dosage form.**Keywords:** lornoxicam, UV method and validation**Introduction**

Lornoxicam is a: (3E)-6-chloro-3-[hydroxyl (pyridin-2-ylamino) methylene]-2-methyl-2, 3-dihydro-4H-thieno-[2, 3] [1, 2] thiazin-4-one1, 1-dioxide. Lornoxicam (chlortenoxicam) is a non-steroidal anti-inflammatory drug (NSAID) of the oxycam class with analgesic, anti-inflammatory and antipyretic properties. It is available in oral and parenteral formulations [1-2].

**Fig 1:** Structure of Fluconazole

The UV spectrophotometric method is very simple, rapid, economical, and it allows the determination of pharmaceuticals with enough reliability. For the UV spectrophotometric method, the survey of literature revealed very complex methods, using bands of the visible range using complexometry, derivative or chemometric assistance and interpolation on the calibration curve [2-3]. The aim of this work was the development and validation of a new UV spectrophotometric method, which can be more economical and simpler than the official methods and with other methods published. The UV spectrophotometric method is simpler than the others studied because it does not need derivative

and chemometric assistance. Moreover, this method can be used in dissolution studies because it uses its own dissolution medium as diluent [2-3].

Analysis is the most important aspect of any drug development whether in bulk or in combination, a suitable method must be developed so as to ensure that any drug either in dosage form or bulk form can be pointed out. The method development ensures that the amount of a particular drug can be easily determined. The validation parameters confirm that the developed method is precise, accurate and reproducible and can be used for routine evaluation of Lornoxicam in bulk and combined dosage form [2-4].

Materials and Methods**Instrumentation**

A UV-Visible Spectrophotometer (UV-1800 SHIMADZU) with 10mm matched quartz cells was used for Spectrophotometric method. All weighing were done on electronic balance (Model Shimadzu AUW-220D).

Reagents and Chemicals

Lornoxicam was received as gift sample from Piramal Healthcare, Mumbai. Tablet formulation manufactured by Pfizer limited was purchased from local market Lornoxi (Brand name of Lornoxicam) Manufactured by Hetero Healthcare Ltd. containing Lornoxicam 8mg per tablet.

Preparation of Standard Stock Solution

Standard drug solution of Lornoxicam was prepared by accurately weighing 10 mg of the drug, and dissolved in phosphate buffer pH 6.8 and the volume was made up to 100ml to obtain stock solution (100 µg/ml) [5-6].

Determination of Analytical Wavelength

From the standard stock solution 1ml was pipette out into 10ml volumetric flask. The volume was made up to 10ml with phosphate buffer pH 7.4. The resulting solution containing 10 μ g/ml was scanned between 200-400 nm [5-6].

Preparation of Calibration Curve

Aliquots of 0.2, 0.4, 0.6, 0.8, 1, 1.2 & 1.4 ml portions of stock solutions were transferred to a series of 10ml volumetric flasks, and volume made up to the mark with phosphate buffer pH 7.4. The serial dilutions in the range of 2, 4, 6, 8, 10, 12 and 14 μ g/ml were prepared. The absorbance was measured at λ_{max} 376nm [5-9].

Linearity and Range

The linearity of the response of the drug was verified at 2-14 μ g/ml concentrations. The calibration curve was obtained by plotting the absorbance versus the concentration data and was treated by linear regression analysis. The equation of the calibration curve for Lornoxicam was obtained [5-9].

Precision

The accuracy of the method was determined by recovery experiments. Each solution was repeated in triplicate and the percentage recovery was calculated. The precision of the method was demonstrated by intra-day and inter-day variation studies [5-9].

Limit of Detection (LOD) and Limit of Quantification (LOQ)

LOD and LOQ were calculated by the equations;

$$\text{LOD} = 3.3\sigma/S \text{ and } \text{LOQ} = 10\sigma/S$$

Where S is the slope of the calibration curve and σ is the residual standard deviation.

Recovery Study

Accuracy of the method was studied by recovery experiments. The recovery was performed at three levels, 80, 100, and 120% of Lornoxicam standard concentration. The recovery samples were prepared as above mentioned procedure for each recovery level. The solutions were then analyzed and the percentage recoveries were calculated from the calibration curve [7-11].

Results and Discussion

Analytical Wavelength

The maximum absorption was found to be at the wavelength of 376nm hence the wavelength for Lornoxicam was found to be 376 nm as shown in figure: 02

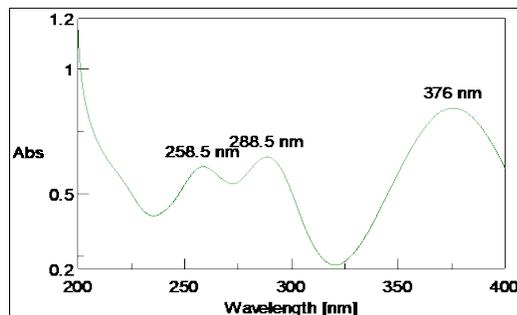


Fig 2: A typical UV Spectrum of Lornoxicam at 376 nm

Calibration Curve

The results of absorbance for all the prepared concentrations were plotted i.e. Concentration vs. Absorbance the method was found to be linear over the prepared concentration range with the standard equation $y=0.0429x+0.0036$ and Regression value was found to be 0.9992 as shown in figure: 03. From the calibration data obtained it was found that the regression coefficient was less than 1 which is within the limits of Beer lamberts' law.

Table 1: Calibration Curve data

Sr. No.	Concentration (μ g/ml)	Absorbance (nm) (Avg \pm SD) (n=3)
1.	2	0.089 \pm 0.0061
2.	4	0.169 \pm 0.0025
3.	6	0.267 \pm 0.0015
4.	8	0.358 \pm 0.0046
5.	10	0.429 \pm 0.0021
6.	12	0.515 \pm 0.0032
7.	14	0.601 \pm 0.0060

n=3

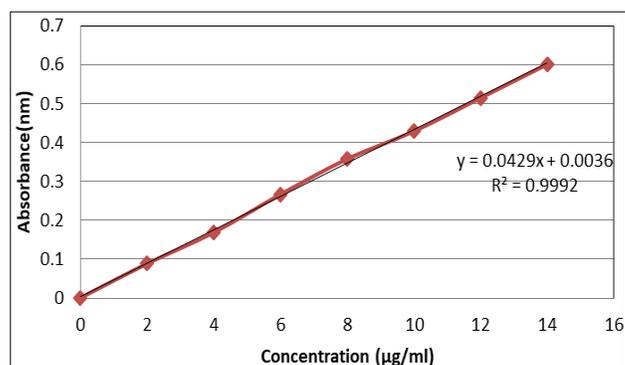


Fig 3: Calibration Curve of Lornoxicam

Precision

Precision of the method was evaluated for Lornoxicam. The reproducibility (inter-day precision) of the method and repeatability (intra-day precision) was evaluated in the same laboratory. The values obtained were 0.9841 and 0.4842 respectively (Table. 02). From the data obtained in Table 02 the method was found to be precise in respect of reproducibility as well as repeatability.

Table 2: Precision

Sample no.	Intra-Day Precision*	Inter-Day Precision*
1	98.80	98.80
2	98.33	97.61
3	97.85	96.90
Mean \pm SD	98.33 \pm 0.47	97.77 \pm 0.96
%RSD	0.4842	0.9841

n=3

Accuracy (Recovery Study)

Accuracy of the method was studied by recovery experiments. The recovery was performed at three levels 80, 100 and 120% of Lornoxicam standard concentration. Three samples were prepared for each recovery level. The solutions were then analyzed and the percentage recoveries were calculated from the calibration curve. The recovery value for Lornoxicam was 101.12 \pm 1.616 and RSD was 1.598.

Table 3: Recovery Study

Pure Drug	Amount $\mu\text{g/ml}$	Level of Addition* (%)	Amount added* ($\mu\text{g/ml}$)	Drug found* ($\mu\text{g/ml}$)	% Recovery	Average % recovery	RSD
Lornoxicam (Lornoxi)**	8	80	6.45	6.404	99.29	101.12 \pm 1.616	
	8	100	8	8.190	102.38		1.598
	8	120	9.6	9.761	101.68		

n=3

Limit of Detection (LOD) and Limit of Quantification (LOQ)

Limit of detection is the lowest amount of analyte which can be detected but not necessarily quantified, and limit of quantification is the lowest possible concentration that can be quantified. LOD and LOQ were found to be 0.29719 $\mu\text{g/ml}$ & 0.9004 $\mu\text{g/ml}$ respectively.

Specificity

Specificity is the ability of the method to accurately measure the analyte response in the presence of all potential sample components (excipients). The results were compared with the

analysis of a standard Fluconazole and tablet formulations. Excipients of the solid dosage form did not interfere with the analyte, which shows that the method has good specificity.

Validation Parameters

All the validation parameters as reported in table 04 were found to be within the desired range which depicts that the method was found to be reproducible with respect to all the validation parameters and can be a useful tool for routine evaluation of eletriptan in bulk and combined dosage form.

Table 4: Validation parameters

Sr. No	Parameter	Result
1.	Absorption maxima(nm)	376
2.	Linearity range ($\mu\text{g/ml}$)z	2-14
3.	Standard regression equation(y)	$y = 0.042x + 0.003$
4.	Correlation coefficient (r^2)	0.999
6.	Slope (m)	0.042
7.	Y-Intercept(c)	0.003
6.	A (1%, 1cm)	435.34
7.	Accuracy (% recovery \pm SD)	101.12 \pm 1.616
8.	Precision	98.33% \pm 0.4761 (Intra-day) 97.77% \pm 0.9622 (Inter-day)
9.	Specificity	A 10 $\mu\text{g/ml}$ sample of std at absorbance 376 nm, 99.28% \pm 0.24
10.	LOD ($\mu\text{g/ml}$)	0.2971
11.	LOQ ($\mu\text{g/ml}$)	0.9004

Discussion

A suitable UV spectroscopic method was developed and validated for Lornoxicam. The Absorbance the method was found to be linear over the prepared concentration range with the standard equation $y=0.0429xx+0.0036$ and Regression value was found to be 0.9992. From the calibration data obtained it was found that the regression coefficient was less than 1 which is within the limits of Beer lamberts' law. The reproducibility (inter-day precision) of the method and repeatability (intra-day precision) was evaluated in the same laboratory. The values obtained were 0.9841 and 0.4842 respectively, the recovery value for Lornoxicam was 101.12 \pm 1.616 and RSD was 1.598 which is less than 2, which shows that the method has good reproducibility, LOD and LOQ were found to be 0.29719 $\mu\text{g/ml}$ & 0.9004 $\mu\text{g/ml}$ respectively.

Conclusion

A suitable UV Spectrophotometric method was developed and validated as per ICH guidelines for the determination of Lornoxicam in dosage formulations. It was shown above that the proposed method was linear, accurate, reproducible, repeatable, precise, selective, specific and cost-effective proving the reliability of the method. More over single solvent was used throughout the experimental work, and it was found that no interference from any excipients was observed in the method.

Hence, the proposed method was successfully applied to routine analysis of Lornoxicam in bulk and combined formulations.

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Authors Contribution

Shoab Mohammad Syed developed the method and performed validation and R P Marathe made the results and conclusion.

Conflict of Interest

None

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