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New first derivative UV spectrophotometric determination method for lornoxicam in pharmaceutical formulations

Santosh R. Karajgi*, Shahid M. Gaffar and Ramaling B. Kotnal

Dept. of Quality Assurance, BLDEA's College of Pharmacy, Vijaypur 586103, Karnataka, India

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ABSTRACT

A simple, novel, sensitive, and specific First derivative uv spectrophotometric method was developed and validated for the determination of Lornoxicam in bulk and its dosage form. The drug was estimated by using 0.01 N NaOH as solvent for this study, which is determined by spectrophotometrically at 377 nm absorption maximum. Beer's law obeyed in the concentration range of 10-50µg/ml. The recovery studies ascertained the accuracy of the proposed method and results were validated as per ICH guidelines. The results were found satisfactory and reproducible.

Key words: Lornoxicam, First derivative, Spectrophotometric method, UV determination

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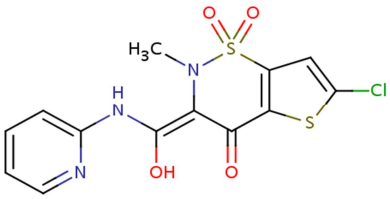
INTRODUCTION



Lornoxicam

Lornoxicam is a NSAID drug that belongs to the oxicam class. As with other NSAIDS, It is an effective inhibitor of the cyclooxgenase enzymes, which are responsible for catalyzing the formation of prostaglandins (in the process of inflammation act as messenger molecules) and thromboxane from arachidonic acid. It is indicated for the treatment of acute mild to moderate pain, as well as pain and inflammation of the joints caused by certain types of rheumatic diseases. Chemically it is (3E)-6-chloro-3-[hydroxyl (pyridin-2-ylamino)methylene]-2-methyl-2,3-dihydro-4H-thieno[2,3-e][1,2] thiazin-4-one 1,1-dioxide.[1] Literature review reveals that fourteen methods have been reported so far for the determination of Lornoxicam in its single component formulations. The reported methods include four HPLC methods [2,3,4,5], four zero order uv spectrophotometric methods [6,7,8,9], one first derivative spectrophotometric method by peak to peak height measurement [9], two area under curve uv spectrophotometric

methods [10], four visible spectrophotometric (Colorimetric) methods [8,11] and one stability indicating uv method in presence of its degradation products [12]. No method is reported by first order derivative absorbance maximum measurement method and there was a scope to develop a new rapid and simple first derivative uv spectrophotometric method for the routine determination of Lornoxicam in its bulk and single component dosage forms which has the advantage over the reported methods in its simplicity and application.

MATERIALS AND METHODS

MATERIALS:

Shimadzu 1800 spectronic model UV Spectrophotometer with a pair of matched 1cm quartz cells was used to data collection and analysis. 0.01 M Sodium Hydroxide solution was employed as a solvent for the drug substance. Commerical brands for the assay were collected from local market.

METHODOLOGY

Preparation of standard stock solution:

The standard stock solution was prepared by transferring 25 mg Lornoxicam in to a 25 ml volumetric flask. 10 ml 0.01 M NaOH was transferred in to this volumetric flask and dissolved. The volume was made up to the mark with 0.01 M NaOH to give a solution containing 1000 µg/ml Lornoxicam. From this solution 2.5 ml was transfer to 25 ml volumetric flask and the volume was adjusted to the mark with the 0.01 M NaOH to give a solution containing 100 µg/ml of Lornoxicam.

Determination of λ max:

Appropriate volume 2.5 ml of standard stock solution of Lornoxicam was transferred to 25 ml volumetric flask and the volume were adjusted to the mark with same solvent to obtain the solution of concentration 10 µg/ml. The solution was scanned in the UV range 200-400 nm and derivatized in first derivative mode of the instrument at N=5. The λ max was found to be 377nm.

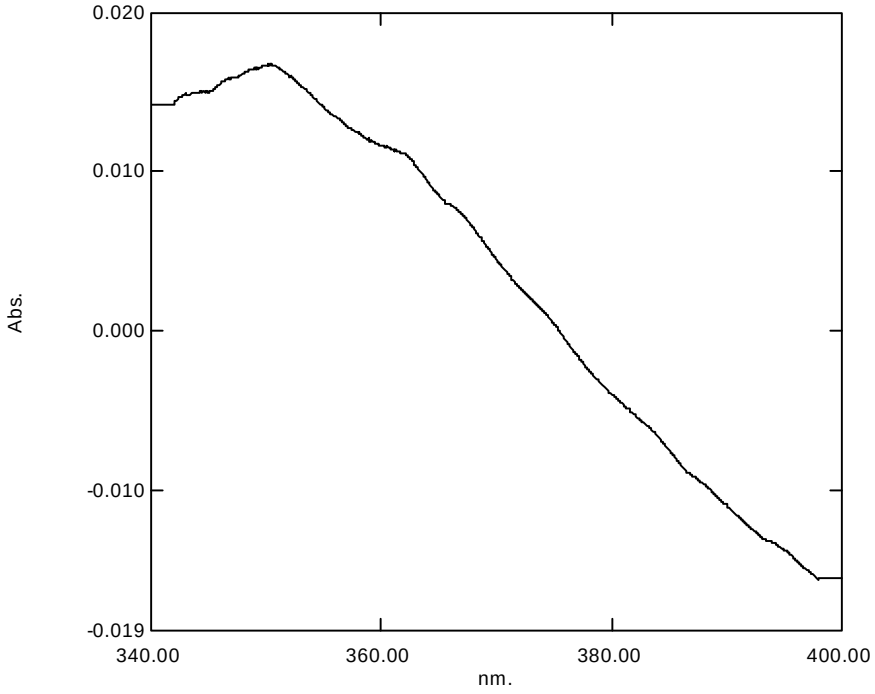


Fig 5.17: Spectrum of λ max of Lornoxicam

Stability of Drug in a Selected Solvent:
The stability of the drugs in the selected solvent was determined by measuring the absorbance of the drug solutions (20µg/ml) at different time interval. The absorbance was measured after every 15 min. The stability data is given in Table 1.

Table 1: Stability Data for Lornoxicam

Sr. No.	Time (min)	Absorbance
1	0	0.614
2	15	0.618
3	30	0.613
4	45	0.616

Study of Beer-Lambert's Law:
From the standard stock solution of Lornoxicam different volumes 2.5, 5, 7.5, 10, 12.5 ml were transferred to five separate 25 ml volumetric flask and volume were made up to the mark with 0.01 M Sodium Hydroxide to obtain concentrations 10, 20, 30, 40, 50µg/ml and calibration plot was constructed. The data is given in Table 2 and Figures 2 to 7.

Table 2: Standard Calibration data for Lornoxicam

Sr. No.	Concentration of Lornoxicam (µg/ml)	Absorbance at 377 nm
1	10	0.353
2	20	0.709
3	30	1.059
4	40	1.426
5	50	1.814

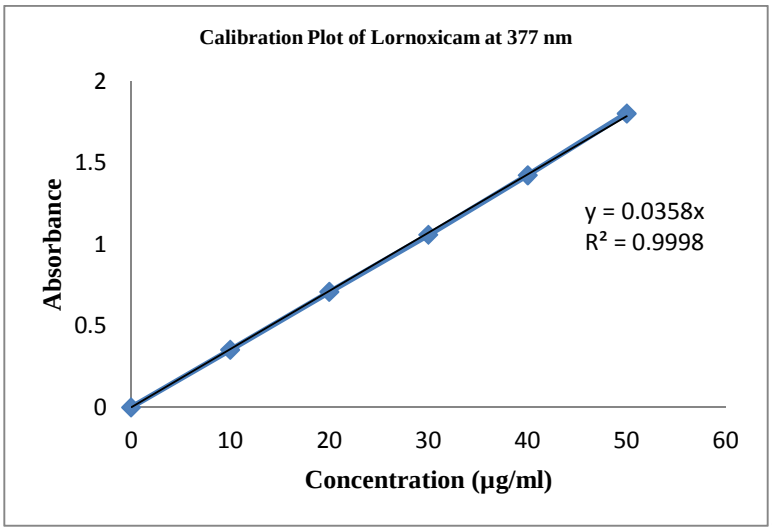


Figure 2: Calibration Plot of Lornoxicam

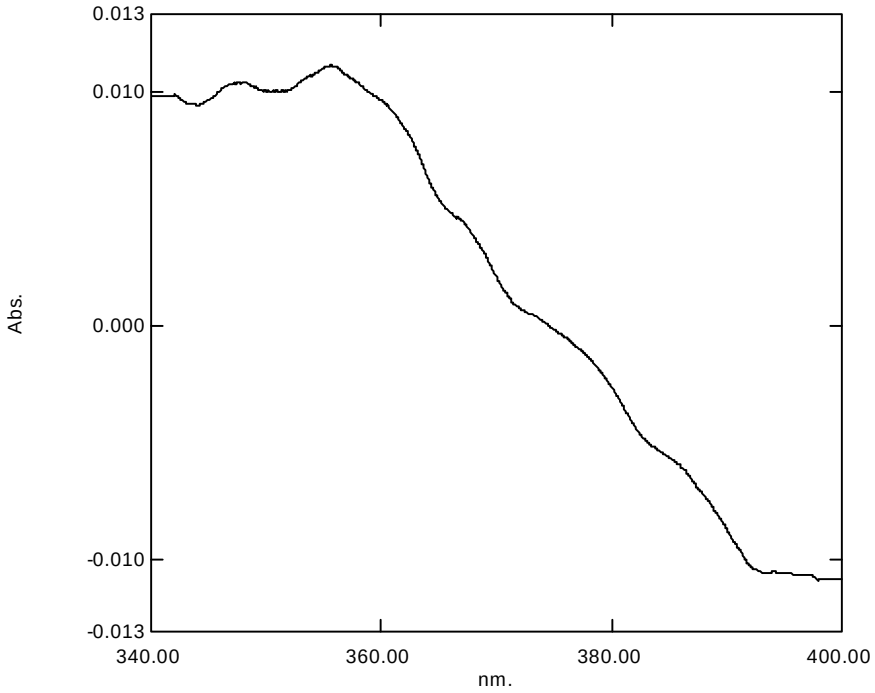


Figure 3: First derivative spectrum of Lornoxicam 10 µg/ml

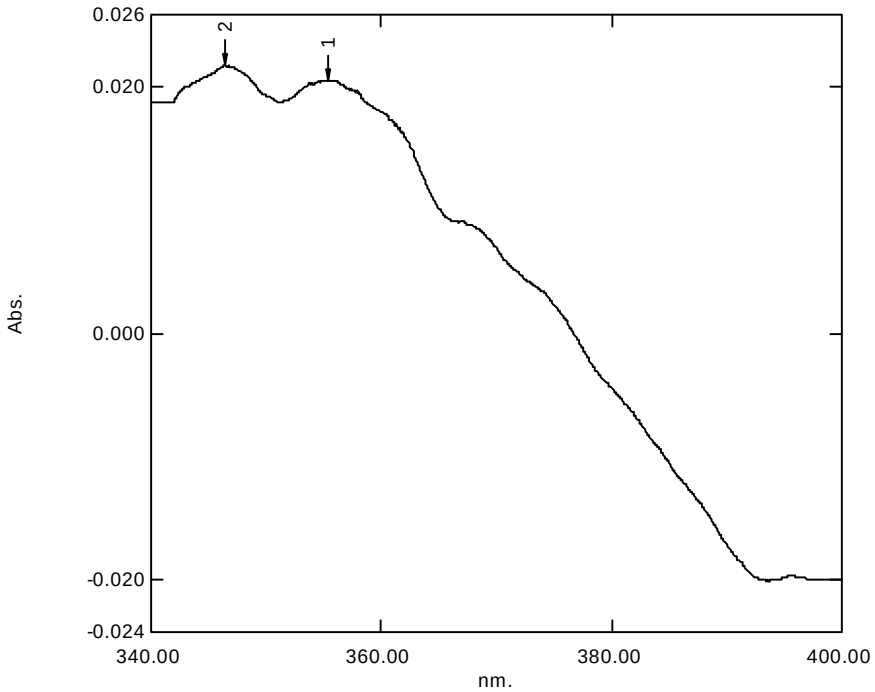


Figure 4: First derivative spectrum of Lornoxicam 20 µg/ml

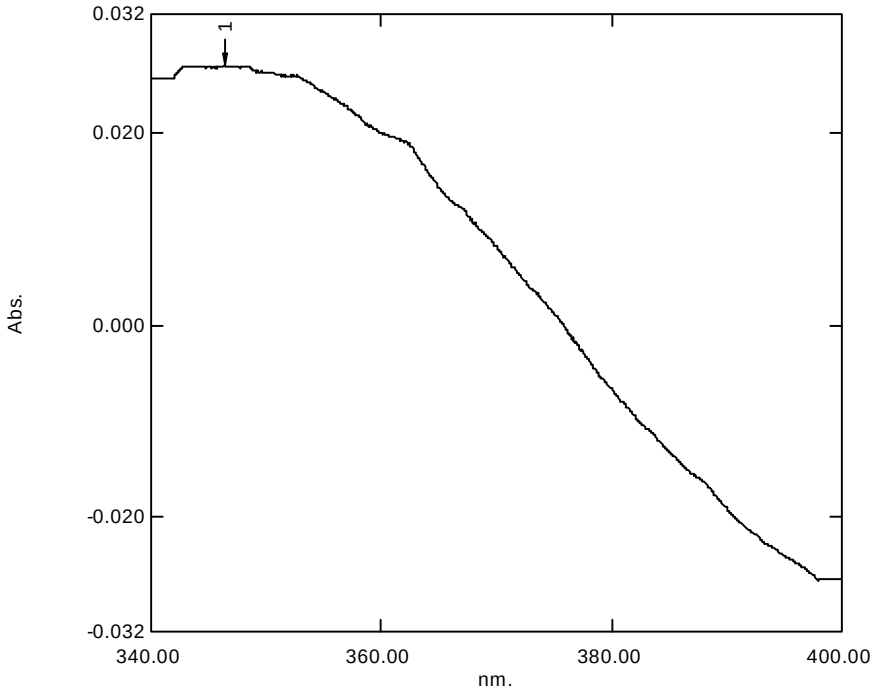


Figure 5: First derivative spectrum of Lornoxicam 30 µg/ml

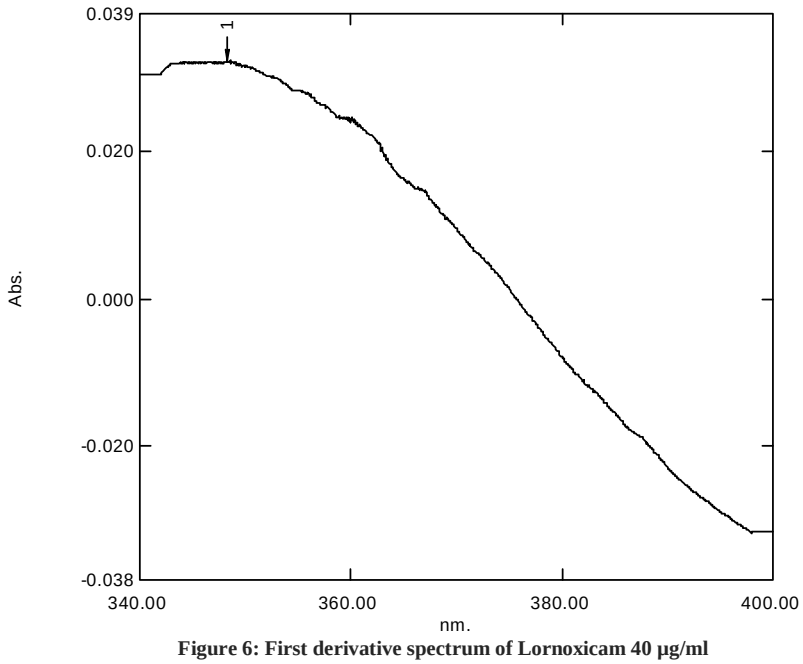


Figure 6: First derivative spectrum of Lornoxicam 40 µg/ml

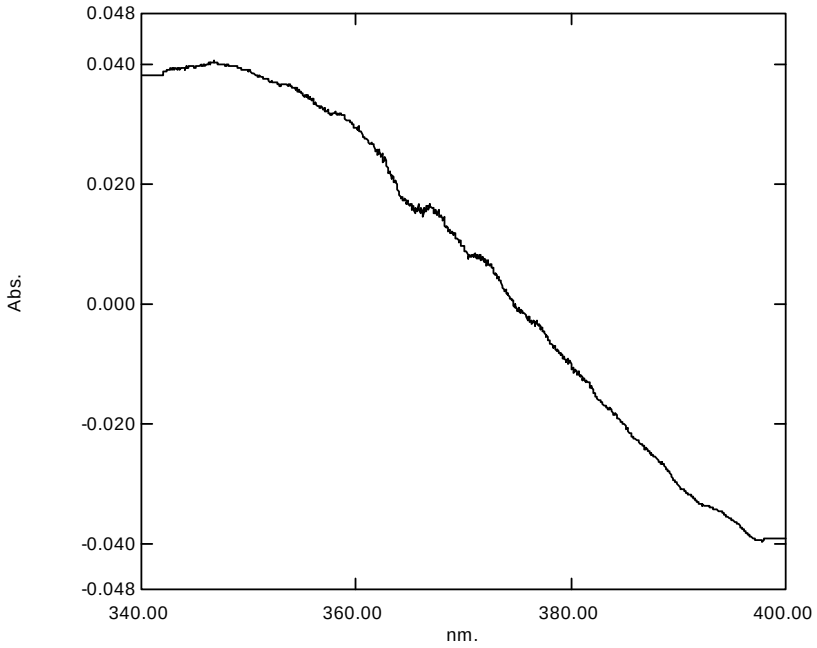


Figure 7: First derivative spectrum of Lornoxicam 50 µg/ml

Optimum Parameters for the Calibration curve.

The optical Parameters of the calibration curves are given in Table 3

Table 3: Optical Parameters for the Calibration Plot

Parameters	Lornoxicam
Linearity range (µg/ml)	10-50
Slope	0.035
Intercept	0
Regression coefficient(r^2)	0.999

Determination of Lornoxicam in bulk:

In order to see the feasibility of proposed method for estimation of lornoxicam in marketed pharmaceutical formulations, the method was first tried for estimation of drugs in standard bulk sample.

Accurately weighed 25 mg of Lornoxicam was transferred to 25 ml volumetric flask, dissolved in 0.01 M Sodium Hydroxide by vigorous shaking and volume was adjusted to mark by the same solvent. Appropriate aliquot 2.5 ml was transferred to 25 ml volumetric flask and volume was adjusted to mark with the same solvent to obtain the concentration 100 µg/ml. From that appropriate aliquot 5 ml was transferred to 25 ml volumetric flask and volume adjusted to mark with the same solvent to obtain the concentration 20 µg/ml. The absorbance of the solution was recorded at 377nm with N=5 against blank and results are reported.

Validation of proposed method:

Application of proposed method for analysis of tablet formulation:

For analysis of commercial formulation; three different brands were uses for the assay. The tablet powder equivalent to 100mg of Lornoxicam was used prepare the 100 µg/ml stock solution. Then the stock solution was diluted to get 20µg/ml solution and the absorbance was taken at 377 nm with N=5 mode. Results are shown in Table 4, 5 and 6.

Table 4: Assay of Lornoxicam in Tablet formulation (Brand A)

Amount Taken (mg/tab)	Amount found (mg/tab)	Amount found (%)
8	7.98	99.85
8	8.04	100.55
8	8.02	100.35
8	7.95	99.38
8	8.06	100.75
	Mean	**100.17**
	SD	**0.5566**
	CV	**0.3098**

Table 5: Assay of Lornoxicam in Tablet formulation (Brand B)

Amount Taken (mg/tab)	Amount found (mg/tab)	Amount found (%)
8	8.02	100.36
8	8.07	100.99
8	7.97	99.74
8	7.96	99.52
8	8.00	100.10
	Mean	**100.14**
	SD	**0.5737**
	CV	**0.3292**

Table 6: Assay of Lornoxicam in Tablet formulation (Brand C)

Amount Taken (mg/tab)	Amount found (mg/tab)	Amount found (%)
8	7.98	99.81
8	7.95	99.39
8	8.06	100.82
8	7.98	99.77
8	8.02	100.31
	Mean	**100.02**
	SD	**0.5539**
	CV	**0.3069**

Accuracy (Recovery Test):

Accuracy of the method was studied by recovery experiments. The recovery experiments were performed by adding known amounts of standard solutions to solutions of tablets. The recovery was performed at three levels, 80, 100, 120 % of Lornoxicam standard concentration. The recovery samples were prepared in afore mentioned procedure. Three samples were prepared for every recovery level. The solutions were analyzed, and the percentage recoveries were calculated by using formula

$$\% \text{ Recovery} = \frac{\text{Observed amount of compound in sample}}{\text{Amount of all compound present in sample}} \times 100$$

The data is given in Table 8.

Table 8: Results of accuracy parameter of Lornoxicam

Level of % Recovery	Amount present (µg/ml)	Amount of standard added (µg/ml)	Total amount recovered (µg/ml)	% Recovery	% mean Recovery	SD	CV
80	8	6.4	14.42	100.140	99.708	0.5226	0.2731
80	8	6.4	14.27	99.127			
80	8	6.4	14.37	99.857			
100	8	8	16.05	100.350	99.898	0.6548	0.4288
100	8	8	16.03	100.197			
100	8	8	15.86	99.147			
120	8	9.6	17.57	99.853	99.615	0.4290	0.1841
120	8	9.6	17.44	99.120			
120	8	9.6	17.57	99.873			

Precision:
Assay of method precision was evaluated by carrying out three independent assays of test sample of Lornoxicam. The intermediate precision of the method was also evaluated using four different analysts, systems in the same laboratory. The Assay values obtained by four analysts were summarized in Table 9.

Table 9: Data for Precision of Lornoxicam

Sample Number	Assay of Lornoxicam as % of labeled amount			
	Analyst- 1	Analyst-2	Analyst-3	Analyst-4
1	99.72	100.99	99.63	100.37
2	99.42	99.93	100.73	100.97
3	100.17	99.48	99.31	99.55
4	99.12	99.73	99.79	99.03
5	99.39	100.14	99.64	99.29
6	99.89	99.87	100.39	99.91
Mean	**99.61**	**100.02**	**99.91**	**99.85**
S.D.	**0.3816**	**0.5218**	**0.5344**	**0.7217**
CV	**0.1456**	**0.2723**	**0.2856**	**0.5208**

RESULTS AND DISCUSSION

The standard solutions of Lornoxicam in 0.01 N NaOH (10µg/ml each) subjected to a scan 200 nm to 400 nm at first order and the derivative spectra were taken at N=5 using Shimadzu 1800 spectronic UV-Visible spectrophotometer. The λ max was found to be at 377 nm. The calibration curve of Lornoxicam was found to be linear at 377 nm. Beer's law obeyed in the concentration range of 10-50 µg/ml.

The newly developed method was validated as per the international guidelines and parameters. The novel method for the quantitative investigation of Lornoxicam was subjected to different validation parameters like selectivity and specificity in presence of formulation additives and excepients, studied for Linearity and range at different levels of concentrations and calibration standards where the determination range was optimized, accuracy was proved by recovery studies at different concentration levels, precision was established through different analyst studies.

With the intention of determining the practicability of the developed technique for the assessment of commercially available brands of medicinal formulations, the technique was initially attempted on bulk drugs and concentrations were estimated. Then the technique was subjected to the assay of tablets in marketed dosage forms and satisfactory results were attained within the acceptable limits as per the content of the label claim for Lornoxicam.

The technique was validated by principles of ICH guidelines for various parameters including specificity, linearity, accuracy, precision- repeatability and the results were found to be satisfactory with lower standard deviation and coefficient of variation values within the acceptable limits for Lornoxicam in their bulk and dosage forms i.e. marketed tablet formulations for their First derivatization UV-spectrophotometric estimation. The method showed specificity in presence of formulation additives, because there was no interfering from the tablets formulation additives. The method was linear, with low deviation values and the regression equation was calculated by the method of least squares. The method was also accurate, indicated by satisfactory recovery studies at different level of confidence. Intermediate precision studies were carried out by different analysts and the results were found to be satisfactory demonstrating that, the process was reproducible. The scheme was not susceptible to change in the method parameters, because the data obtained were reproducible in different temperature conditions applied at the time of determination of these drug substances with very negligible deviations under the conditions employed.

The described method offer precise and accurate results for the quantitization of Lornoxicam in their bulk drugs and tablets formulations and applied without any difficulty for the regular determinations. The method is also simple, rapid and economical method which gives reproducible results on the instrument used. The reported method is an economical method in which only 0.01 N Sodium Hydroxide is used as the solvent and does not require the use of costly reagents. This proposed method is competent of being used for the quantitization of Lornoxicam drugs in bulk and tablet dose forms devoid of the interfering of additives with a significant and comparative correctness and exactness with the reported methods. This newly developed method has the advantages over the previously reported methods because, present methods is economical.

The percentage standard deviation values show that the proposed method provides acceptable variation of Lornoxicam. The standard deviation percentages of proposed technique is within the acceptable limits for Lornoxicam shows the competence of the technique to stay unchanged by minute and purposeful changes in the system restraints and assures its consistency in regular routine application.

CONCLUSION

The results and the statistical parameters demonstrate that the proposed First derivative UV spectrophotometric method for Lornoxicam is simple, rapid, specific, accurate and precise. The most striking features of spectrophotometric method are their simplicity and rapidity. Result of validation parameters demonstrated that these analytical procedures are suitable for its intended purpose and meets the criteria defined in ICH Q2/B. This new method is useful for routine analysis of Lornoxicam in its single component dosage forms without interference of impurities.

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