Available on line www.jocpr.com

Journal of Chemical and Pharmaceutical Research

ISSN No: 0975-7384
CODEN(USA): JCPRC5

J. Chem. Pharm. Res., 2011, 3(2):258-263

Analytical method development and validation protocol for Lornoxicam in tablet dosage form

Atul R. Bendale*[1], Jigneshkumar J Makwana[2], Sushil P Narkhede[1], Sachin B. Narkhede[2], Anil G. Jadhav[2], G. Vidyasagar[3]

[1]Suresh Gyan Vihar Universe, Jaipur, Rajsthan
[2]Smt. B. N. B. Swaminarayan Pharmacy College, Salvav (Vapi), Gujarat
[3]Pharmacy Department, Kutch University, Bhuj, Gujarat

ABSTRACT

Lornoxicam (chlortenoxicam) is a non steroidal anti- inflammatory drug (NSAID) of the oxicam class with analgesic, anti-inflammatory and antipyretic properties. It is available in oral and parental dosage formulation. Lornoxicam is a yellow or slightly yellow powder. It is slightly soluble in water, soluble in sodium hydroxide, slightly soluble in methanol. A simple spectrophotometric method was developed for the determination of lornoxicam in pharmaceutical tablet dosage form. Lornoxicam exhibiting λ max at 258 nm in mobile phase (0.05 M NaOH) and obeyed linearity in the concentration range of 5-30mcg. The proposed method was statistically validated.

Keywords: Lornoxicam, Analytical method development, Validation protocol.

INTRODUCTION

The scope of developing and validating analytical method is to ensure a suitable method for a particular analyte more specific, accurate and precise the main objective for that is to improve the condition and parameter, which should be followed in the development and validation[1,2]. Lornoxicam (chlortenoxicam) is a non steroidal anti- inflammatory drug (NSAID) of the oxicam class with analgesic, anti-inflammatory, and antipyretic properties.

Tablet formulation containing 4mg and 8mg lornoxicam in film-coated and dispersible form are available in the market. Literature survey revealed that various analytical methods such as TLC, HPLC, liquid chromatography, polarographic method, LC-ESI-MS (liquid chromatography-Electrospray ionization- tandem- mass spectrometric) are used for estimation of lornoxicam [4, 5]. No simplest UV-Spectrophotometric method has been reported for estimation of lornoxicam

in tablet dosage form. Hence, an attempt has been made to develop new spectrophotometry method for its estimation in pharmaceutical tablet dosage form with good accuracy, simplicity, and precision. In UV-Spectrophotometric method UV spectrum is set in 258 nm and blank is placed in the cuvettes, after setting zero transmittance the sample is placed in another cuvette for measuring the absorbance the sample.

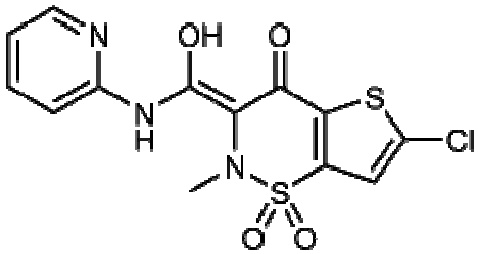

FIG.1: LORNOXICAM: (3e) - 6- Chloro- 3- [Hydroxy (Pyridin- 2- Ylamino) Methylene] - 2- Methyl-2, 3- Dihydro- 4h- Thieno [2, 3-E] [1, 2] Thiazin- 4- One 1, 1-Dioxide [8,9]

EXPERIMENTAL SECTION

Absorbance measurements were made on LABINDIA, UV 3000^{+}Spectrophotometer. ConTECH-CA 123balance was used for weighing the sample. Commercially available tablets of the lornoxicam were procured from the local market and estimated.

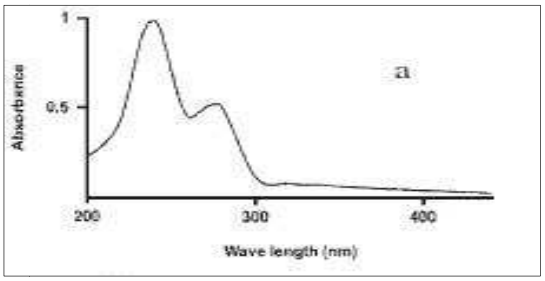

FIG 2: Determination of maximum wavelength of Lornoxicam

Preparation of mobile phase (100ml): Dissolve 0.2 gm of NaOH in 100 ml of distilled water to make 0.05M NaOH solution.

Preparation of standard stock solution: Standard stock solution was prepared by dissolving 100 mg of lornoxicam in 100 ml of mobile phase to get concentration of 1 mg/ ml.

Preparation of working standard solution and construction of calibration curve: The prepared stock solution was further diluted with mobile phase to get working standard solution of 5, 10, 15, 20, 25, and 30 mcg of lornoxicam to construct Beer's law plot for the pure drug, the absorbance was measured at λ max at 380 nm, against mobile phase as blank .The standard graph was plotted by taking concentration of drug on X-axis and absorbance on Y-axis in the concentration range of 5-30 mcg.

Preparation of sample stock solution and working sample solution: Ten tablets were accurately weighed and average was calculated. The tablet were then crushed to obtain fine powder equivalent to about 4 mg of lornoxicam was transferred to 100 ml volumetric flask ,

added mobile phase and shaken for a while. The volume was made up to the mark with mobile phase and required dilutions were made from the sample stock solution.[6]

Validation [7]

Assay: The assay of the proposed method was ascertained by performing assay of the standard drug with reference to the sample drug and finding out the absorbance. From the absorbance percentage purity was calculated. The readings are shown in **table 1**.

Table 1: Assay of Lornoxicam Tablet

LORNICAM* 4	Claim of tablet (mg/tablet)	Drug found (mg/tablet)	%Purity
	4	4.04	101.00%

RESULTS AND DISCUSSION

Linearity: To establish linearity of the proposed methods, six separate series of solutions of lornoxicam (5-30 mcg) in mobile phase (0.05 M NaOH) were prepared from the stock solutions and analyzed. Least square regression analysis was performed on the obtained data. Linearity data are shown in **table 2 & 3** as follows:

Table 2: Linearity Table of Lornoxicam in Working Standard

Concentration (mcg)	Absorbance
5	0.311
10	0.441
15	0.604
20	0.746
25	0.892
30	1.046

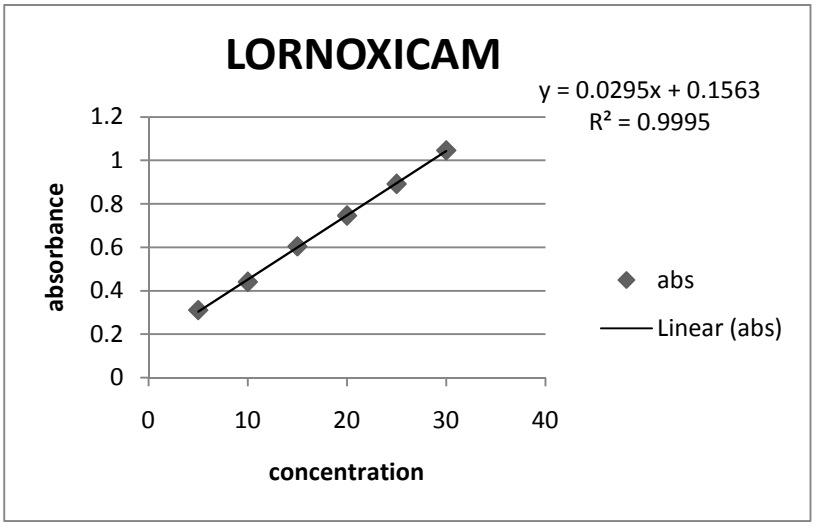

Fig. 3: linearity curve of Lornoxicam in working standard

Table 3: Linearity Curve Data

Beer's law limit (mcg)	5-30
Correlation coefficient (R^2)	0.999
Regression equation (y^*)	y = 0.029x + 0.156
Slope (m)	0.029
Y-Intercept(c)	0.156

Precision

Repeatability

Percentage R.S.D. was found between 0.07745 - 0.19186 %.
Percentage R.S.D. is less than 1 %, it proves that UV-Visible spectrophotometer gives precise results.

Table 4 : Repeatability data of Lornoxicam

Concentration (µg/ml)	% R.S.D.(n = 3)
15	0.191860
15	
15	
20	0.07745
20	
20	
25	0.129484
25	
25	

Intraday Precision

Percentage R.S.D for intraday precision was found between 0.15 - 0.36 %. Percentage R.S.D. is less than3 %, it prooves that method is precise.

Interday Precision

Percentage R.S.D. for interday precision was found between 0.35 – 0.92%. Percentage R.S.D. is less than 5 %, it proves that method is precise.

Table 5: Intraday and Interday precision of Lornoxicam

Concentration (µg/ml)	INTRADAY (n =5) % R.S.D.	INTERDAY (n =5) %R.S.D.
5	0.36	0.92
10	0.30	0.46
15	0.15	0.54
20	0.18	0.35
25	0.19	0.44
30	0.17	0.61

ACCURACY: To determine the accuracy of the proposed method, recovery studies were carried out by adding different amounts of standard bulk sample of lornoxicam within the linearity range were taken and added to the pre-analyzed formulation of concentration 5 mcg and percentage recovery values are calculated (**Table 6**).

Mean percentage recovery was found between 101.00 – 101.53 %.
Mean percentage recovery is between 98 – 102 %, it proves that method is accurate.

Table 6: Accuracy data of Lornoxicam

Amount of Lornoxicam in sample (µg)	Amt of std Lornoxicam added (µg)	Total amount of Lornoxicam	Amount of Lornoxicam found	% Recovery (n = 3)	Mean % recovery
5	-	5	5.05	101.00	101.53
5	-	5	5.09	101.80	
5	-	5	5.09	101.80	
5	5	10	10.10	101.00	101.00
5	5	10	10.15	101.50	
5	5	10	10.05	100.50	
5	10	15	15.30	102.00	101.53
5	10	15	15.30	102.00	
5	10	15	15.09	100.60	
5	15	20	20.20	101.00	101.21
5	15	20	20.23	101.15	
5	15	20	20.30	101.50	

RUGGEDNESS

The data for ruggedness obtained from two different analysts is presented in Table 7. Percentage R.S.D. was found between 0.48 - 1.79 %.

Percentage R.S.D. is less than2 %, it proves that method is rugged.

Table 7: Ruggedness data of Lornoxicam

Concentration (µg/ml)	% R.S.D. (n = 2)
5	0.74
10	1.01
15	0.48
20	1.79
25	1.45
30	1.73

TABLE 8: Summary of validation parameter

PARAMETER	RESULTS OFLORNOXICAM
Linearity range (µg/ml)	5-30
Correlation coefficient, r	0.999
Precision (% R.S.D.) Repeatability Intraday (n=5) Interday (n=5)	0.07 -0.19 0.15 -0.36 0.35 -0.92
Ruggedness (% R.S.D.)	0.48 – 1.79
Mean % recovery	101.00 – 101.53 %.

CONCLUSION

The proposed method was simple and reliable with good precision, accuracy, linearity and ruggedness. The proposed method is specific while estimating the commercial formulations without interference of the excipients and other additives. Hence, this method can be used for the routine determination of lornoxicam in pure and pharmaceutical formulation.

REFERENCES

[1] Ewing G. W., **(1985),** Instrumental methods of chemical analysis, Fifth Edition., Mcgraw Hill International Edition., Chemistry Series, 1-4.
[2] Connor K. A., **(1999),** A textbook of pharmaceutical analysis, Third Edition, John Wiley and Sons Publication, 173-179.
[3] Skoog D.A., West D. M. and Holler F.T., **(2001),** Fundamentals of analytical chemistry, Seventh Edition, Harcourt College Publication, 710.
[4] Sharma. B. K., **(2002)**, Instrumental method of chemical analysis, Twenty First Edition, Goel Publishing Housing, Krishna Publication, 3.
[5] Backett A. H. and Stenlake J. B., **(2002)**, Practical pharmaceutical chemistry, Fourth Edition, Part 2, CBS Publishers and Distributors, New Delhi, 1-3.
[6] Sethi P.D., **(2001)**, HPLC Quantitative analysis of pharmaceutical formulation, First Edition, CBS Publishers and Distributors, 6,11,135.
[7] Jonathan B. and Crowther, **(2001),** Validation of pharmaceutical test methods, Handbook of modern pharmaceutical analysis, Third Edition, Academic Press, 418-419.
[8] Martindale, **(1999),** The complete drug reference, Thirty Second Edition, Pharmaceutical Press, London, 128, 523.
[9] Martindale, **(1999),** The complete drug reference, Thirty Third Edition, Pharmaceutical press, London, 232, 599.
[10] USP, NF 19, **(2000)**, The United States Pharmacopoeia and National Formulary, XXIV, U.S. Pharmacopoeial Convention, Inc., Rockville, 330-331.
[11] Budhavari S., Neil J., Smith A., Heckelman P. E. and Kinneary J. F., **(1997)**, The Merck Index, An Encyclopedia of Chemicals, Drugs and Biologicals, Thirteenth Edition, Merck and Co., 860.
[12] Kamble A. and Venkatachalam A., **(2005)**, *Indian Drugs*, 42(11), 723-725.