Research Article

UV Spectrophotometric Stability Indicating Method Development and Validation for the Determination of Tenoxicamin Bulk and Dosage Form

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ABSTRACT

A simple, specific and economic UV spectrophotometric method has been developed using as diluents 0.1N NaOH to determine the tenoxicam content in bulk and pharmaceutical dosage formulations. The quantitative determination of the drug has been carried out at a predetermined λ_{max} of 370nm, it was proved linier in the range 2-12 µg/mL and exhibited good correlation coefficient (R^2=0.999) and excellent mean recovery (98-99%). LOQ and LOD was found to be 0.881, 2.67 respectively. The method was validated statically and by recovery studies for linearity, precision, repeatability and reproducibility as per ICH guideline. The obtained results proved that the method can be employed for the routine analysis of tenoxicam in bulk as well as in the commercial formulations.

Keywords: Tenoxicam, UV Spectroscopy, Method Validation.

INTRODUCTION

Analytical method development

Analytical methods are intended to establish the identity, purity, physical characteristics and potency of the drugs and to support drug testing against specifications during manufacturing and quality release operations as well as during long term stability studies.

Method validation

Validation of an analytical method is the process by which it is established, by laboratory Studies, that the performance characteristics of the method meet the requirements for the Intended analytical applications.

Tenoxicam chemically 4-hydroxy-2methyl-n-(pyridinyl-2-yl)-2h-thieno [2, 3-e]-1, 2thiazine-3-carboxamide 1, 1-dioxide, is a Non-steroidal anti-inflammatory drug[1]. It is used to relieve inflammation, swelling, stiffness, and pain associated with rheumatoid arthritis, osteoarthritis, ankylosing spondylitis. It is official in BP[2].

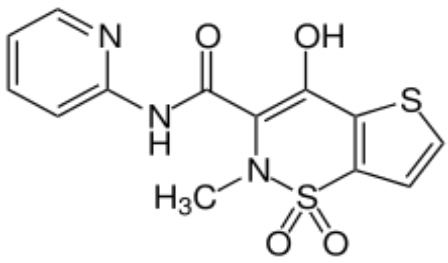

Figure 1: Structure of Tenoxicam

Literature survey reveals that LC-MS study has been reported for the quantification of tenoxicam in human plasma and HPLC method development for the determination of tenoxicam. No UV spectrophotometric method has been reported for the estimation of tenoxicam. In this study, efforts were made to develop a simple, easy and economic UV spectrophotometric method using a diluents 0.1N NaOH for the determination of tenoxicam in raw material as well as in the marketed dosage formulations. The developed method was optimized and validated as per the guidelines of International Council on Hormonisation (ICH) and demonstrated excellent specificity, linearity, precision and accuracy for tenoxicam.

MATERIALS AND METHODS

Instruments

A Shimadzu UV–visible spectrophotometer (UV1800, Shimadzu Corporation, Kyoto, Japan) was used for all absorbance measurements with matched quartz cells.

Materials

All chemicals and reagents were of analytical grade. Tenoxicam in the form of powder with certificate of analysis was provided by Ramdev Chemicals Pvt Ltd, Mumbai. Pharmaceutical grade excipients were obtained from Pharmaceutical Technology Lab. of Maharashtra.

Determination of wavelength of maximum absorption

A standard stock solution of Tenoxicam (100 µg/mL) was prepared using diluents to further obtain 10 µg/mL.An UV spectroscopic scanning (200-400 nm) was carried out with final diluted solution to determine λmax for the detection of Tenoxicam using diluents as a blank.

Linearity and Range

For linearity study, six solutions at different concentrations (2, 4, 6, 8, 10 and 12 µg/mL) were prepared using six different aliquots of stock solution, and the obtained data were used for the linearity calibration

plot. Limit of detection (LOD) and limit of quantification (LOQ) for the assay were also calculated.

Intra-day precision (repeatability) and inter-day precision study (intermediate precision)

Tenoxicam 100 µg/mL was prepared following the same dilution pattern of stock solution. Three different aliquots of stock solution were then diluted to 10 mL to obtain the concentrations of 4, 8 and 12µg/mL. This procedure was repeated in the different days.

Stability study

Samples prepared for repeatability study were preserved for 24 h at room temperature and analyzed on the following day to test for short-term stability.

Accuracy/recovery study

This study was carried out using tablet of tenoxicam. Calculation was done from the label claim and the average weight of the final product. Previously used dilution pattern was followed for the tablet to obtain three concentrations—80%, 100% and 120% of reference solution.

Specificity in the presence of excipients

The test for the specificity was carried out using only excipients. Spectra for placebo tablet, blank, and sample were compared. Secondly the specificity was determined by subjecting the sample solution to accelerated degradation by heat (60 °C) for 48 h in order to verify that none of the degradation products interfered with the quantification of the drug.

Assay of content of Tenoxicam in selected marketed brands

Market brands of tenoxicam tablet from different manufacturers were randomly selected and analyzed using the newly developed and validated method. Sample solutions of brand (10 mg/mL) were also prepared and assayed for content of tenoxicam against the standard. The content of tenoxicam in the marketed brands was determined using standard calculations.

Stress degradation studies

1) **Photolytic Degradation**

Specific amount of drug tenoxicam was weighed accurately & putted into the UV chamber for three days. After three days 10mg drug was weighed and made stock solution (100µg/mL) with diluents. Then an appropriate concentration (10 µg/mL) was prepared & absorbance was measured in UV spectrophotometer.

2) **Thermal Degradation**

Drug was taken in a Petri dish which was previously cleaned & dried then was put it into the oven for 48 hrs then it was taken out & weighed 10mg drug was weighed and made stock solution (100 µg/mL) with diluents. Then an appropriate concentration (10 µg/mL) was prepared & absorbance was measured in UV spectrophotometer.

3) **Acid Degradation**

0.01N HCl was taken in a 10 ml volumetric flask then accurately weighed 10mg drug tenoxicam was dissolved in it. Then the solution was refluxed for 4 hrs then from this solution an appropriate concentration (10 µg/mL) was prepared using diluents & absorbance was measured in UV spectrophotometer.

4) **Alkali Degradation**

0.01N NaOH was taken in a 10 ml volumetric flask then accurately weighed 10mg drug tenoxicam was dissolved in it. Then the solution was refluxed for 4 hrs then from this solution an appropriate concentration (10 µg/mL) was prepared using diluents & absorbance was measured in UV spectrophotometer.

5) **Oxidation with H_2O_2**

3% H_2O_2 solution was taken in a 10 ml volumetric flask then accurately weighed 10mg drug tenoxicam was dissolved in it. Then the solution was kept in dark for 4 hrs then from this solution an appropriate concentration (10µg/mL) was prepared using diluents & absorbance was measured in UV spectrophotometer.

RESULTS AND DISCUSSION

Method development and optimization

An accurately weighed quantity of tenoxicam (10 mg) transferred into a 100 ml volumetric flasks, dissolved well and diluted to the mark with0.1N NaOH to obtain standard solution having concentration of (100 µg/ml). A 1 ml of solution transferred into a 10 ml volumetric flasks and diluted to the mark with 0.1N NaOH to obtain the solutions having the concentrations of 10µg/ml for tenoxicam. The pre-determined wavelength of maximum absorption (λmax) was 370 nm. (Fig. 1)

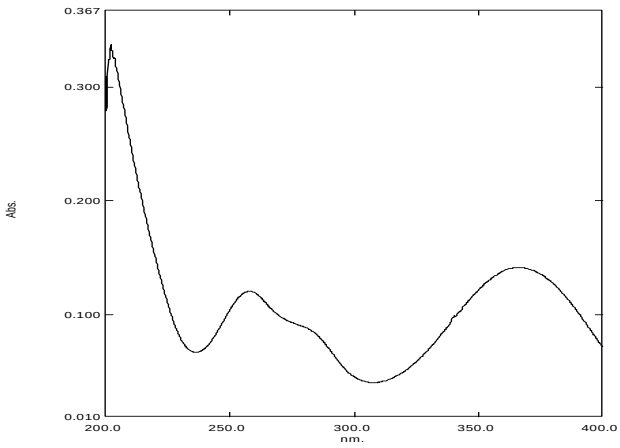

Figure 2: UV Spectrum of Tenoxicam

Method validation

Linearity and range

The calibration curve obtained was evaluated by its correlation coefficient. The absorbance of the samples in the range of 2.0–12.0 mg/mL was linear with a correlation coefficient (R^2) greater than 0.999 (Table 1). The LOD and LOQ were calculated as 0.881 mg/mL and 2.67 mg/mL respectively.

Table 1: Linearity data

Concentration µg/ml	Absorbance
2	0.150
4	0.284
6	0.420
8	0.563
10	0.695
12	0.841

Intra-day and inter-day precision

The intra-day and inter-day precision study (Table 2) of the developed method confirmed adequate sample stability and method reliability where all the RSDs were less than 2%.

Table 2: Intra-day and inter-day precision determined for three different concentrations of Tenoxicam (n=3).

Conc.µg/mL	Intra-day precision		Inter-day precision	
	Absorbance measured	RSD (%)	Absorbance measured	RSD (%)
4	0.281	0.523	0.294	0.515
8	0.563	0.244	0.581	0.235
12	0.841	0.116	0.560	0.200

Stability

Stability study's results were within the acceptance range (Table 3) and indicated the samples stability over 24 h (short-term).

Table 3: Short term stability determined by the proposed method (n=3).

Conc declared µg/mL	Concentration found µg/mL	RSD (%)	Average potency (%)
4	0.279	0.146	99.21
8	0.560	0.145	98.68
12	0.836	0.100	98.98

Accuracy/Recovery

Results within the range of 98.00–100.97% ensure an accurate method (Table 4) as well as indicate non-interference with the excipients of formulation.

Specificity in the presence of recipients

The specificity of the analytical method was proved by comparing the spectra of placebo and degradation product of sample solution with that of accuracy sample (Fig. 3).

Table 4: Accuracy/Recovery for three different concentrations of tenoxicam by the proposed method.

Brand	Label claim (mg/tab)	Amount added (%)	Percentage Recovery
1	200	80	98.40
		100	99.60
		120	98.40

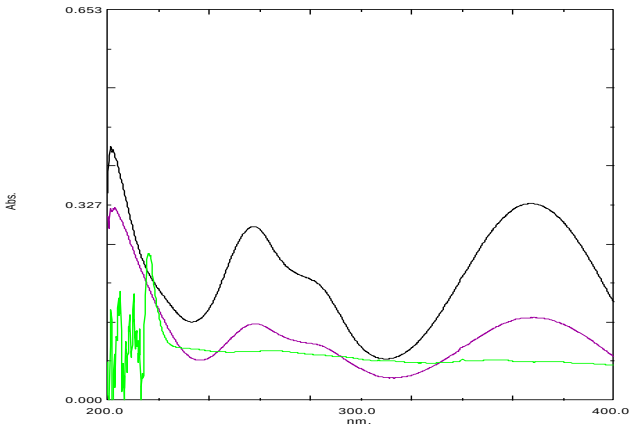

Figure 3: Specificity of the method determined by comparing the spectra of accuracy sample, placebo and degradation products

CONCLUSION

The results and the statistical parameters demonstrate that the proposed UV spectrophotometric method is simple, rapid, specific, accurate and precise. Therefore, this method can be used for the determination of tenoxicam either in bulk or in the dosage formulations without interference with commonly used excipients and related substances.

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REFERENCES

1. Merck Index, 13th edn. Merck and com. Inc, White house station, 2001, 1632.
2. British Pharmacopoeia, Published by the deptt. Of health and stationary office under the license from the controller of her majesty; stationary office for the dept. of Health Minister, UK, 2002, 2477.
3. Ji HY, Lee, HW, Kim YH. Simultaneous Determination of Piroxicam, Meloxicam and Tenoxicam in Human Plasma by Liquid Chromatophy. J. Chromatog., 2005, 826-214.

4. Troconiz JI, Lopez B. HPLC Analysis of Piroxicam&TenoxicamIn Plasma, Blood,Buffer Solution. J. Arzneimittelforschung. 43, 1993, 679.
5. Doliwa A, Santoyo S. Sensitive LC Determination of Piroxicam after invitro Transdermal Permeation Studies. J. Pharm. Biomed. Anal. 26, 2001, 531-537.
6. Taha EA, Salama NN. Stability-Indicating Chromatographic Methods For The Determination of Some Oxicams. J. AOAC. Int. 87,2004, 366-373.
7. Sora I, Galaon T. Fast RPLC-UV method on short sub-two microns particles packed column for the assay of tenoxicam in plasma samples. J. Pharm. Biomed. Anal. 43, 2007, 1437-1443.
8. Dasandi B, Saroj H. LC Determination and Pharmacokinetics of Meloxicam. J. Pharm. Biomed. Anal., 28, 2002, 999-1004.
9. Raghavan CV, Abimon VD. Intransal Delivery of Tenoxicam in rat", J. Int. Pharm. 221, 2001, 227-229.
10. Walily AF, Blaih SM. Simultaneous determination of tenoxicam and 2aminopyridine using derivative spectrophotometry and high-performance liquid chromatography. J. Pharm. Biomed. Anal., 15, 1997, 1923-1928.
11. El-ries MA. Spectrophotometric Determination of Piroxicam and Tenoxicam in Pharmaceutical Preparations Using Uranyl Acetate as a Chromogenic Agent. J. Anal. Lett. 31, 1998, 793-807.
12. Momani AL. Indirect flow injection Spectrophotometric Determination of Meloxicam, Tenoxicam and Piroxicam in pharmaceutical formulations. Analytical Science. 22, 2006, 1611.
13. Cardoso SM, Rolim CM. Quantitative determination of tenoxicam in tablet by UV spectrophotometry. J. Actafarmaceuticabonaerense 25, 2006; 262-266.
14. Taha EA, Salama NN. Spectrofluorimetric and spectrophotometric stability-indicating methods for determination of some oxicams using 7-chloro-4-nitrobenz-2-oxa-1,3-diazole. J. Chem. Pharm. Bull. 54, 2006, 653-658.
15. El-Ries MA. Spectrophotometric and potentiometric determination of piroxicam &tenoxicam in pharmaceutical preparations. J. Chem. Pharm. Bull. 51, 2003, 6-10.
16. Amin, a.s. Spectrophotometric determination of piroxicam and tenoxicam in pharmaceutical formulations using alizarin. J. Pharm. Biomed. Anal., v.29, n.4, 2002, p.729-736.
17. Atkopar, z.; tunçel, M. The polarographic determination of tenoxicam in the pharmaceutical preparations. Anal. Lett. v.29, n.13, 1996, p.2383-2397.

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